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Shaw® Shaw Environmental & Infrastructure, Inc.

field support services, inc.

FINAL WORK PLAN
REMOVAL ACTIONS
SWMU 6, SWMU 7, AOC J, and AOC R
FORMER NAVAL AMMUNITIONS SUPPORT DETACHMENT
VIEQUES, PUERTO RICO

Prepared for:

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EXECUTIVE SUMMARY

This report presents a Work Plan for removal actions at Solid Waste Management Unit (SWMU) 6, SWMU 7, Area of Concern (AOC) J, and AOC R at the former Naval Ammunitions Support Detachment (NASD) in the western portion of Vieques Island, Puerto Rico. This Draft Work Plan has been prepared for the Department of the Navy, NAVFAC Atlantic, Norfolk, Virginia, under Contract No. N62470-03-D-4402, Task Order No. 0015 (TO #15).

Following previous investigations and an Engineering Evaluation and Cost Analysis, Field Support Services, Inc. (FSSI/Shaw) has been contracted to perform removal actions at SWMU 6, SWMU 7, and AOC J. Also presented in this document is a removal action at AOC R which is not included in the current scope of work but may be performed in the near future. The overall goal of these Removal Actions is to eliminate the uncertainty of the debris being a potential future source of contamination by removing the debris and associated contaminated soil, if present. The scope of work includes the removal of an estimated combined total of 11,050 tons of debris and soil from the four sites. The actual tonnage may be more or less, depending upon the site conditions encountered. Excavation will continue until visible debris is removed and acceptable residual risk values have been achieved. The excavated waste will be transported to an off-site permitted recycling or disposal facility. Additionally, FSSI/Shaw will provide unexploded ordnance (UXO) support for proper identification and disposal of any suspect items encountered during clearing and grubbing earth disturbing activities. The following provides a brief description for each of these sites:

SWMU 6

SWMU 6 is located approximately 100 feet south of the Vieques Passage and was used for disposal of solid wastes during the 1960's and 1970's. Waste discarded at the site includes rusty metal, car parts, rubber tires, broken glass, rubble, and empty containers formerly filled with lubricants, oil, solvents, and paints. Additionally, there are five large concrete filled inert bomb casings partially buried in the water and sediment on the site. The main area of debris is just north of Road 200 and runs along the side of the road. The debris pile is approximately 165 feet long and 10 feet wide by two feet deep. Total estimated waste present is 450 tons.

SWMU 7

SWMU 7 is approximately 1,100 feet south of the Vieques Passage. From the early 1960's to the late 1970's, an ephemeral stream at the site was used for disposal of solid waste materials. Discarded materials include old tires, sheet metal, empty containers (such as drums, cans, and bottles), old batteries, and construction rubble. Debris also includes two sea mine casings, six rocket launchers, a Volkswagen Beetle, and hundreds of tires. Disposal activities appear to have

been concentrated in a segment of the stream approximately 420 feet along the length of the dirt access road. The estimated total waste present is 9,000 tons.

AOC J

AOC J is located two miles west of the entrance to the former NASD and north of Highway 200. The area extends approximately 50 feet south of the Vieques Passage. AOC J was used as a solid waste disposal site from 1965 to 1973. The debris piles consist of metallic and non-metallic materials and one empty drum. Estimated total waste present is 600 tons.

AOC R

AOC R is located approximately 2 miles west of the entrance to the former NASD, and several hundred feet south of the Vieques Passage along Highway 200. A portion of the western boundary is adjacent to an ephemeral stream. This stream is the same ephemeral stream adjacent to AOC J, which is topographically down gradient of AOC R. AOC R was used as a public works operational area from 1965 to 1971. The debris piles consist of a practice bomb, ammunition cans, metal pipes, rocket launchers, wood power poles, tires, white corrugated roofing sheet material, and munitions items. Total estimated waste present is 1,000 tons.

Field Activities

The following provides a brief summary of the field activities for Task Order 15:

- Collect and analyze soil samples for the characterization of the waste at each site. This analysis will be required for the disposal facilities before the waste can be accepted and to ensure that the waste is not hazardous.
- Install erosion and sediment control devices at the sites to minimize off-site migration of sediment. These devices will include silt fence, diversion dikes, and construction entrances.
- Clear and grub the removal areas. This will consist of clearing grass, brush and necessary vegetation within the approximate areas of removal. FSSI/Shaw will chip/mulch as much of the vegetation as possible. All practicable efforts will be made to preserve endangered vegetation in the vicinity where the debris removal is planned.
- During the clearing, grubbing and excavating activities UXO technicians will monitor work and identify UXO items that may be encountered.
- Excavate and remove an estimated total of 11,050 tons of debris and soil from the four sites. The majority of the debris and soil is anticipated to be disposed of at the Vieques Municipal Landfill. The material (soil and debris) will be classified based upon the criteria listed in the QAPP Worksheet No. 17, and its final disposition (landfill cover, landfill placement, ect.) will be determined based upon those results. Recyclable materials and tires will be transported to the island of Puerto Rico for recycle/disposal.

- Collect and analyze confirmatory samples from SWMU 6, SWMU 7, AOC J, and AOC R to confirm that residual media concentrations are protective of human health and the environment (via a residual risk assessment).
- Prepare a report documenting all field activities. Included in the report will be copies of the waste disposal documentation and analytical reports as well as the residual risk assessment report.

Resumen Ejecutivo

Este resumen presenta un plan de trabajo para las trabajos de remoción en las Unidades de Manejo de Desperdicios Sólidos (SWMU) 6, SWMU 7, y el Área (AOC) J y AOCR en la antigua facilidad de Apoyo para Municiones Navales (NASD) in la parte oeste de la Isla de Vieques, Puerto Rico. Este borrador de trabajo ha sido preparado para el Departamento de la Marina de Estados Unidos, NAVFAC Atlantic, Norfolk, Virginia, bajo el contrato No. N62470-03-D-4402, Orden 15.

Siguiendo previas investigaciones y una Evaluación de Ingeniería y Análisis de Costo, "Field Support Services, Inc." (FSSI/Shaw) ha sido contratada para realizar las labores de remoción en SWMU 6, SWMU 7 y AOC J. Tambien se incluyen en este documento las acciones de remoción para el area AOCJ, esta aun no esta incluida en la presente orden de trabajo, pero los trabajos de la misma podrian realizarse en un futuro cercano. La meta general de estas labores de remoción es eliminar la posibilidad de que estos escombros tengan el potencial de convertirse en el futuro en una fuente de contaminación, para evitar esto se removerán los escombros y terreno que se determine estar contaminado si alguno. El alcance del trabajo incluye la remoción de un total combinado de 11,050 toneladas de escombros y tierra de las cuatro areas en cuestión. Las cantidades finales pueden ser mayores o menores a la cantidad mencionada dependiendo de las condiciones que se encuentren en las areas cuando se realicen los trabajos. Los trabajos de excavación y remoción continuaran mientras se encuentren escombros visibles y hasta tanto se obtengan los niveles aceptables de riesgo residual. Los escombros y tierra removidas serán transportados y dispuestos en facilidades de reciclaje y / o disposición y que cuenten con los debidos permisos de las agencias reguladoras para recibir desperdicios no-peligrosos. Además, FSSI/Shaw proveerá los servicios de técnicos especializados para la identificación y manejo de municiones sin explotar (UXO), para identificar y disponer de cualquier artefacto sospechosos que se encuentre durante el proceso de desmonte y de actividades donde se realicen.

SWMU 6

SWMU 6 se encuentra localizado a aproximadamente 100 pies al sur del pasaje de Vieques y fue usado para disponer desperdicios sólidos durante las décadas de 1960 y 1970. Los desperdicios depositados en esta área incluyen metales ferrosos, partes de automóviles, neumáticos de autos, vidrio, escombros y envases vacíos de lubricantes, aceite, solventes y pintura. También se encuentran en el lugar 5 caparazones de bombas (inertes) llenos de cemento y parcialmente sumergidas en el agua. El área principal de escombros esta al norte de la carretera # 200 y esta paralela a lo largo de la carretera. La pila de escombros es

aproximadamente 165 pies de largo y 10 pies de ancho por 2 pies de profundidad. Se estima una cantidad de 450 toneladas en el area.

SWMU 7

SWMU 7 se encuentra localizado a aproximadamente 1,100 pies al sur del pasaje de Vieques. Desde la primera parte de la década de 1960 hasta el final de la década de 1970 se usaba una quebrada intermitente para disponer los desperdicios sólidos. Los desperdicios depositados en el lugar incluyen planchas de metal, neumáticos, envases vacíos tales como barricas, latas, botellas, baterías viejas y escombros de construcción. También se encuentran en el lugar dos caparazones de minas (inertes) acuáticas, un caparazón de lanzacohetes, un auto volkswagen y cientos de gomas de autos y camiones. Las actividades de disposición aparentemente se concentraron en un trecho de aproximadamente 420 pies de largo, paralelo a el camino de tierra que da acceso al lugar. Se estima que en esta área se removerán aproximadamente 9,000 toneladas de escombros y terreno

AOC J

AOC J se encuentra a dos millas oeste de la entrada de la antigua NASD y al norte de la carretera # 200. El área se extiende aproximadamente 50 pies al sur del pasaje de Vieques. AOC J fue usada para disponer desperdicios sólidos generados por las actividades de construcción realizadas entre 1965 y 1973. Los desperdicios localizados en el área incluyen metales y escombros así como un barril vació. La mayoría de la basura sólida en AOC J está en las laderas de la zanja, con escombros adicionales situados hasta a 100 pies al oeste de la zanja. El total estimado de desperdicios en el lugar es de 600 toneladas.

AOC R

AOCR está localizada aproximadamente 2 millas al oeste de la entrada de lo que solía ser NASD, y varios cientos de pies al sur del pasaje de Vieques a lo largo de la autopista 200. Una porción de la extremo oeste del aea esta adyacente a una quebrada intermitente a AOC J, la cual está topográficamente mas abajo que AOC R. AOC R era usada como área de operaciones públicas de 1965 a 1971. Los desperdicios sólidos están compuestos por una bomba de práctica, latas de municiones, tubos de metal, lanzadores de cohetes, postes eléctricos de madera, llantas, material blanco de metal corrugado para el techo y objetos de municiones. El total estimado de los desperdicios es 1,000 toneladas.

Actividades de Campo

A continuación una descripción breve de los trabajos de campo para la orden de contrato # 15.

- Recolectar y analizar para caracterización muestras de suelo en cada área de trabajo. Estos análisis son requeridos por la facilidad de disposición para demostrar que los mismos no son clasificados como desperdicios peligrosos.

- Instalar controles de erosión y sedimentación en cada área de trabajo para minimizar la migración de sedimentos. Se usaran membranas de control de sedimentos, diques para desviar las escorrentías y controles de las entradas de las áreas de trabajo.

- Desmonte y preparación de áreas. Esta tarea consistirá en la remoción de arbustos y hiervas en las áreas donde se realizaran las remociones. FSSI/Shaw, triturara dentro de lo posible la vegetación que se remueva. Se realizaran esfuerzos para preservar vegetación que se considere protegida y que se encuentre dentro de las áreas donde se realizara la remoción de escombros.

- Durante el desmonte y preparación de área, técnicos especializados en UXO, o explosivos sin detonar estarán monitoreando los trabajos para identificar cualquier pieza de explosivos que se pueda encontrar.

- Excavar y remover un estimado de 11,050 toneladas de escombros y tierra de las cuatro áreas en cuestión. Se anticipa que la mayor parte de los escombros y tierra que se remueva se llevara al vertedero del Municipio de Vieques para disposición. Los materiales (escombros y tierra) se clasificaran basado en los criterios mencionados en la hoja de trabajo # 17 de el "QAPP" y su disposición final (material de cobertura, basura para colocar en el vertedero, etc.) se determinara basado en esos resultados. Materiales reciclables y llantas de automóviles se transportaran a la isla de Puerto Rico y se dispondrán en facilidades de reciclaje.

- Recolectar y analizar muestras de confirmación de las áreas SWMU 6, SWMU 7, AOC J y AOC R, para confirmar que la concentración residual no afecta la salud de humanos y el ambiente. (determinado por un estudio de riesgo)

- Preparar un reporte de todas las actividades de campo que se realicen. Se incluirán copias de la documentación de disposición de los reportes analíticos así como o del estudio de riesgo.

1.0 INTRODUCTION

1.1 BACKGROUND

Field Support Services, Inc. (FSSI/Shaw) has been retained by the Department of the Navy, NAVFAC Atlantic, Norfolk, Virginia, under Task Order No. 0015 (TO #15) to provide Removal Actions at former Naval Ammunitions Support Detachment (NASD), located in the western portion of Vieques Island, Puerto Rico (**Figure 1**). The Removal Action will include activities at Solid Waste Management Unit (SWMU) 6, SWMU 7, Area of Concern (AOC) J, and AOC R. SWMU 6, AOC J, and AOC R are located on the Department of Interior, United States Fish and Wildlife Service land and SWMU 7 is located on the Municipality of Vieques land; therefore, coordination with the landowners will be required. **Figure 2** shows the locations of the four sites.

The overall goal of the Removal Actions is to eliminate the uncertainty of the debris being a potential future source of contamination by removing the debris and associated contaminated soil, if present. The scope of work includes the removal of an estimated combined total of 11,050 tons of debris and soil from the four sites. The excavated waste will be transported to and disposed of at a permitted off-site facility. The sites and surrounding areas impacted by field activities will be restored in accordance with Section 2.7. **Figures 3, 4, 5, and 6** show the approximate areas of excavation and restoration for the four sites.

The scope of work for TO #15 is summarized as follows in six major steps (the details of each step are presented in Section 2):

- Waste Profiling Set Up
- Sampling
- Sample Analysis and Waste Characterization
- Federal, State, and Local Coordination
- Removal Action
- Contingency Planning for Unanticipated Discovery
- Site Restoration
- Project Schedule

1.2 SITE DESCRIPTION

This section provides descriptions of the four sites included in this Removal Action.

1.2.1 SWMU 6

SWMU 6 is located approximately 100 feet south of the Vieques Passage and was used for disposal of solid wastes generated by Navy operations within the former NASD during the 1960's and 1970's. The area of waste disposal is rectangular in shape and is approximately 260' wide and 180' long. Waste discarded at the site includes: rusty metal, car parts, rubber tires, broken glass, rubble, and empty containers formerly filled with lubricants, oil, solvents, and paints. Many items have deteriorated due to natural corrosion in the saltwater environment and are in the form of small pieces of metallic or glass debris. This Removal Action addresses only removal of the wastes, including debris and soil. Munitions related items such as inert concrete filled bombs, empty bomb dispensers, and empty shell casings have been identified during previous investigations at the site and shall be removed prior to confirmatory sampling. The main area of debris is just north of Road 200 and runs along the road. The main pile is approximately 200 feet long by 10 feet wide by 3 feet deep from visual observations. There are also additional smaller piles throughout the area. Additionally, there are five large concrete filled bombs partially buried in water and sediment on the site. The total estimated waste present at SWMU 6 is 450 tons.

1.2.2 SWMU 7

SWMU 7 is approximately 1,100 feet south of the Vieques Passage. From the early 1960's to the late 1970's, an ephemeral stream at the site was used for disposal of solid waste materials. Discarded materials disposed on SWMU 7 include old tires, sheet metal, empty containers such as drums, cans, and bottles, old batteries, and construction rubble. SWMU 7 is currently located on Municipality of Vieques property. Disposal activities appear to have been concentrated in a segment of the ravine approximately 420 feet along the length of the dirt access road where waste materials were pushed over the edge. Most of the waste material is confined to the steep slopes. Debris also includes: two sea mines, six rocket launchers, a Volkswagen Beetle, and hundreds of tires. Munitions related items shall be removed prior to confirmatory sampling. This removal action addresses only waste, including debris and soil containing debris, located at SWMU 7. The total estimated waste present is 9,000 tons.

1.2.3 AOC J

AOC J is located two miles west of the entrance to the former NASD and north of Road 200. The area extends approximately 50 feet south of the Vieques passage. AOC J was used as a solid waste disposal site from 1965 to 1973. The debris piles consist of metallic and non-metallic materials and one empty drum. A munitions and explosives of concern (MEC) avoidance survey

conducted in May 2000 found 105-mm shell casings and empty ammunition boxes. Munitions related items discovered shall be removed prior to confirmatory sampling. A geophysical survey was conducted at AOC J to delineate the extent of buried waste. Subsurface debris does not extend beyond the western bank of the stream. Total waste present at AOC J is approximately 600 tons.

1.2.4 AOC R

AOC R was used as a public works operational area from 1965 to 1971. A portion of the western boundary of AOC R is adjacent to an ephemeral stream. This stream is the same ephemeral stream adjacent to AOC J which is topographically down gradient of AOC R. Based on previous investigations and a visual inspection, there are several areas on site requiring waste removal. The first area contains a debris pile along the bank of the ditch. The waste appears to have been pushed onto the bank of the stream with some material buried approximately two feet deep. These waste materials appear to consist of a practice bomb, ammunition cans, metal pipes, and rocket launchers. Munitions related items that are discovered shall be removed prior to confirmation sampling. The second area, north of the concrete pad, consists of four 20 feet long, 18 inch diameter wood telephone poles with approximately 30 cars tires on the poles. Near the east end of the concrete pad, a third debris pile measuring approximately ten feet long by ten feet wide by six feet high consists of white corrugated roofing sheet material. The fourth waste area contains four munitions items. All munitions items shall be removed prior to the commencement of confirmatory sampling. The total estimated waste present at AOC R is approximately 1,000 tons.

2.0 Removal Action Activities

FSSI/Shaw has developed an operational approach for the removal actions at former NASD that is protective of the personnel and the environment. This approach is based on firsthand experience with similar projects and consultation with in-house specialists. Additionally, comments and review by the Vieques ERP Technical Subcommittee were incorporated into the operational approach for the removal action.

2.1 WASTE PROFILING SET-UP

- To assist in planning of the site activities, FSSI/Shaw will submit a request to the Municipality of Vieques Public Works for a utility mark-out for all sites in question. Additionally, FSSI/Shaw may contact a private utility locator to perform a subsurface electromagnetic scan of the area outside the proposed excavation areas to determine if underground utilities cross the site. The need for such actions shall be determined by the site superintendent based upon field observations. All efforts shall be made to ensure that there is no utility damage during the removal actions.
- FSSI/Shaw will schedule a mobilization date upon receiving a notice to proceed from the Navy Technical Representative. FSSI/Shaw personnel, equipment, and materials will be mobilized based upon nearest available resources. All equipment will be mobilized from vendors. At all times, FSSI/Shaw will comply with site security requirements; all personnel not associated with the project will be restricted from entering the work areas.
- After receiving approval, FFSI/Shaw will conduct vegetative clearance throughout the entirety of the known and suspected debris areas, utilizing UXO avoidance support. The clearing and grubbing will consist of the grass, brush, and necessary vegetation within the approximate areas of removal. Prior to clearing activities, FFSI/Shaw will walk these areas with a USFWS representative. Wetland areas will be marked to minimize equipment from impacting and disturbing these areas. Additionally, within AOC J, two species of *Stahlia monosperma* shall be protected from the removal actions. A protective barrier shall be installed prior to clearing activities. This procedure is further detailed within section 2.5.3. Once cleared, FSSI/Shaw will install silt fence around the perimeter of each proposed removal area to limit the impact of migrating soils. All practical efforts will be made to preserve any and all endangered vegetation in the vicinity of the debris removal areas. Two UXO technicians will be present during the clearing, grubbing, and removal actions to monitor the activities, and identify any UXO items that may be present. Following any identification, the UXO technicians will aid in the disposal of said items. **Appendix D** contains a copy of the Explosive Safety Submittal for the sites in question.

- Following vegetative clearance, the remedial investigation (RI) sample points will be flagged to identify where soil data already exists. Based upon the RI sample points, a waste characterization grid will be determined in accordance with the QAPP, attached within **Appendix C**. Using a different color pin flag, the waste profile sampling points will be flagged with bias towards any observed debris. Profiling shall be biased toward the areas suspected to best represent the highest levels of contamination (i.e., debris areas). Once all sample points are identified and marked, the ERP Technical Subcommittee will conduct a site visit to agree upon sample locations. This site visit will include the use of a metal detector to help confirm or adjust proposed waste profiling sample locations.
- The Navy and its subcontractors have performed surveys of the areas in question for threatened and endangered species, and for archeological significance. All areas of significance shall be avoided where necessary. **Appendix G** contains a copy of these studies.

2.2 SAMPLING

- Sampling shall be conducted in accordance with the Field Sampling and Analysis Plan and the QAPP worksheets, contained within **Appendix C.**

2.3 SAMPLE ANALYSIS AND WASTE CHARACTERIZATION

- Each of the waste profiling soil samples collected will be analyzed for the presence of the target compound list (TCL) of VOCs, pesticides, polychlorinated biphenyls (PCBs) and semi volatile organic compounds (SVOCs), explosives and target analyte list (TAL) metals. The samples will also be analyzed for full toxicity characteristic leaching procedure (TCLP); reactivity, corrosivity, and ignitability (RCI); and total petroleum hydrocarbons (TPH). All samples will be collected in accordance with the QAPP, Worksheet 19, located within **Appendix C.** All analytical data (except TCLP, RCI, and TPH) will be validated; During the Waste Characterization Sampling, TCLP, RCI, and TPH shall go through a data verification process instead. The Field Sampling and Analysis Plan (SAP), **Appendix C**, contains all validation process. Once soil sample analysis is complete, results will be characterized based upon pre-established criteria listed in the QAPP, Worksheet 17. Final disposition of the soil will be based upon this characterization.
- Where applicable the soil sample data will also be used to identify the areas where the soil requires removal due to a risk to the human health and the environmental receptors. Because residual human health and ecological risk assessments will be performed using the post-removal confirmatory data, Target Contaminant Concentrations shall be developed through the results of the pre-removal risk assessment and will be used

internally to establish intermediate guidelines for the removal action. These samples shall be collected by Shaw, to assist in determining when it is appropriate to cease the excavation and begin the confirmation process. These intermediate samples shall be for internal use only, and will not be included within the confirmation sampling package. Therefore, these Target Contaminant Concentrations are not subject to regulatory comment. However, the Target Contaminant Concentration information will be provided to the regulatory agencies for informational purposes

- Based upon soil analysis, the final disposition of the soil and debris will lie in either a hazardous permitted facility, a permitted recycling facility, in the Vieques Municipal Landfill as waste, used as daily cover for the Vieques Municipal Landfill, or remain onsite. The decision matrix for this protocol can be found within the QAPP, Worksheet 10**.** This information (soil analysis and associated risk assessment) will be provided to the regulatory agencies for concurrence with the proposed disposal protocol. Once concurred upon, the final disposal process will be documented as an addendum to the removal action work plan.
- Once the extent of the removal and disposal protocol is concurred upon, Shaw/FSSI will submit to the Puerto Rican EQB documentation for the modification of the Vieques Municipal Landfill permit. This will allow for the landfill to temporarily accept a larger daily volume of debris, if said soils are to be disposed of there.
- FSSI/Shaw will submit an Army Corps of Engineers (USACOE) form 4345 "Application for Department of the Army Permit" for the USACOE 404 Permit, to work within the wetlands at SWMU-6. Since this work is being performed as a CERCLA action as directed by the EPA, the project is exempt from the formal permit process. The submittal of this form is solely for notification to the Army Corps of Engineers.
- Based upon the analytical results, the number and locations of confirmatory samples will be proposed and provided to the regulatory agencies for review. The concurred-upon locations will be documented in an addendum to the removal action work plan.

2.4 FEDERAL, STATE, AND LOCAL COORDINATION

- FSSI/Shaw will consult with the Municipality of Vieques to determine the requirements for placing the Fire Department on Stand-By status during site activities. In addition, FSSI/Shaw will implement on-site fire protection measures.
- Due to limited sight distances caused by uneven terrain, FSSI/Shaw will establish defined haul routes for material handling and movement around the site. Where necessary, spotters will be positioned to guide heavy equipment operators and vehicles transporting materials in tight work areas. A general traffic pattern for the site will be established and will be communicated in advance to the field personnel.

- FSSI/Shaw will conform to Navy requirements with respect to controlling vehicle access into the site. Additionally, FSSI/Shaw will coordinate roadway and refuge access/logistics with the Navy Technical Representative, Municipality of Vieques, and the USFWS. FSSI/Shaw will maintain communication with the USFWS throughout the life of the project to ensure compliance with refuge access restrictions, and limit the impacts to the refuge.
- The T&D contractor will monitor the truck traffic conditions on the road. Off-site haul routes will be agreed upon prior to any movement. The trucks will depart the site at staggered intervals to minimize traffic congestion on the roads, since the landfill route is through the community. To limit truck impact, the approximate number of truck trips will be limited to 50 trips per day. During transit all loads will be covered.
- The schedule of activities within the site is listed in the project schedule, located in **Appendix E.**

2.5 REMOVAL ACTION

- Exclusion zones will be setup around each excavation area at each site. The exclusion zones will also encompass any excavated material staging areas and ingress/egress areas used to access the waste piles. FSSI/Shaw will install a temporary, high-visibility barricade fence (approximately three feet in height) at the limits of each exclusion zone to restrict unauthorized access by both humans and animals. These exclusion zones will be monitored and maintained by the site manager. All activities within the exclusion zone will conform to the Site Specific Health and Safety Plan. All control devices shall be removed at the completion of removal activities.
- The removal action is anticipated to consist of the removal of approximately 11,050 tons of debris and soil, based upon the initial sampling. It is possible that the removal actions will encounter debris not visible at the ground surface that may alter the actual tonnage of the removal actions. The removal action quantities are subject to change based upon the disposal protocol referenced in Worksheet #10 of the QAPP (Appendix C).
- Within the general disposal area, test excavations will be made at various locations where waste is not visible. This will confirm/refute the presence of metals beneath the land surface. Screening will also be conducted with a photo ionization detector (PID)
- The excavation process will be tailored to each individual site, as conditions warrant, however the general methods for removal will take place as follows:
    - All material (soil and debris) shall be excavated from the sites and placed into a daily staged pile for load-out. This daily pile shall be lined with 2 sheets of 8-mil plastic, to prevent contaminated material from migrating into other areas onsite. It is anticipated that all daily piles shall be located within

the areas determined to be part of the removal actions, this will aid in the prevention of contaminants migrating to unaffected areas. Any materials that cannot be staged within the excavation area shall be directly loaded into trucks for disposal.

- At all times the excavations shall be worked to eliminate any runoff. At the end of each day, the entire area shall be graded to prevent any migration of soils, debris, or storm water from the excavation area.
- Material loaded to the staging pile, shall be segregated by backhoe, excavator, and manual labor. Material shall be segregated into soils and trash and metal and large debris. Materials segregated shall be of 6" in diameter or greater. In areas where a temporary pile is not utilized, the materials shall be segregated by the same means, but segregation shall take place during the load out to the disposal trucks.
- Soils shall be further segregated to remove any trash optimizing the soils for possible use as daily cover. Note: Material used as daily cover does not count towards the daily disposal capacity of the landfill. The determination for use as daily cover is detailed within QAPP Worksheet 17, located within **Appendix C.**
- If the soils are deemed acceptable to be left onsite based upon intended land use and acceptable risk as outlined in the waste characterization risk analysis to be performed, the soils and debris shall be excavated as indicated in this work plan and associated QAPP, however the soils shall be re-distributed onto the site after debris removal, confirmation sampling, and associated risk assessment have been performed. The redistribution of soils shall mimic the original landscape to the extent practical. All efforts shall be made to avoid unnecessary disturbance of soils.
- In order to ensure the effectiveness of the plastic liner, a 6" layer of soil shall be left in place to protect the liner from mechanized equipment. This soil layer and the plastic liner shall be removed simultaneously to ensure all material is collected and disposed of accordingly.
- Soil and trash shall be loaded into trucks (separately) for disposal, if required. Disposal methods will be in accordance with the methods outlined in the QAPP, Worksheet 10.
- Metal and Large debris shall be loaded into roll-off containers for transport to the main island of Puerto Rico for recycling and disposal.

- A solid waste manifest will be completed for each load of debris and impacted soil transported and disposed of at the landfill.
- Large debris encountered during the removal, will be segregated from the soil, and where possible sent to an approved recycling facility.

- Confirmatory sampling shall be conducted to confirm the completion of the removal activities. Confirmation sampling protocol shall be established once the results of the waste characterization sampling are available, and shall be based upon the criteria detailed within the QAPP, Worksheet #17, located within Appendix C. All confirmation sampling locations shall be concurred upon by all the regulatory agencies prior to any confirmatory sampling events.
- A new risk assessment for each site will be conducted on the post-removal confirmatory soil/sediment/surface water data. Confirmatory sampling will be conducted across the entire area, with potential bias based upon removal action observations. This risk assessment, conducted in accordance with the HHRA and ERA protocols in the Master QAPP (CH2M HILL, February 2007), will be included in the removal action report submitted to the agencies for review.
- If additional impacted areas are presented, the areas will be marked in the field and additional excavations shall be performed according to *Section 2.6 Contingency Planning for Unanticipated Discovery*. The decision matrix on this protocol is displayed within the QAPP, Worksheet 10, **Appendix C.**

2.5.1 SWMU 6

In conjunction with the clearing and grubbing of the area, 250 linear feet of ten-foot high chain link fence will be removed. The fencing will be rolled up and donated to the municipality for future use. Following these activities, approximately 450 tons of debris and soil from the site will be removed in a manner consistent with the type of debris encountered at the site. The depth of the excavation will be dependent upon the debris requiring removal based on visual observations, PID readings, and characterization data. FSSI/Shaw will utilize small equipment during the removal action to minimize damage to the larger mangrove trees at this site. FSSI/Shaw will scrape the area with a backhoe or bobcat to remove the area to the desired depth. In the event the material is too saturated to scrape, the solids will be removed first. Some dewatering may be necessary, and the material will be staged to dry on plastic sheeting in the middle of the access road adjacent to the excavation area, and then loaded with the loader for transportation to the main access road (the highway). The staging area shall be constructed so that runoff from the excavated material shall flow back into the excavation area, to prevent the possible spread of contaminated soils.

2.5.2 SWMU 7

Approximately 9,000 tons of debris and soil from the site will be removed. The excavation depth will be based on visual observations, PID readings, and characterization

data. SWMU 7 contains areas of extreme slope and steep ravines. Caution shall be taken to minimize any additional erosion and runoff created by steep slopes. Excavation shall proceed from the top of slope downward to eliminate the creation of additional sloped areas and prevent soil migration from storm water run off. Extra precaution shall be taken to prevent any adverse effects to the surrounding areas from the excavation. Additionally, extra soil erosion measures such as hay bales and super silt fence may be warranted based upon weather and working conditions. During the removal, tires and large metal debris will be segregated from the remainder of the waste. The tires will be transported to the island of Puerto Rico for disposal and the large metal will be transported to a recycler on Puerto Rico. The remainder of the waste will be transported to the Vieques Municipal Landfill for disposal in accordance with the waste profiling protocol defined in the QAPP Worksheet No. 17.

2.5.3 AOC J

Approximately 600 tons of debris and soil from the site will be removed in a manner consistent with the type of debris encountered at the site. The depth of the excavation will be dependent upon the extent of the debris and based on visual observations, PID readings, and characterization data.

Two individuals of the endangered *Stahlia monosperma* have been identified in the vicinity of where the debris removal is planned. Conservation measures described by the Navy to the Department of Interior United States Fish and Wildlife Service will be implemented to protect the specimens during the removal actions. The conservation measures will consist of establishing a buffer zone around the *Stahlia monosperma* to prevent their accidental damage or destruction. The size of the buffer zone will be the radius of the approximate root area (estimated by the horizontal distance from the trunk of the tree to the tip of the branch farthest from the trunk) plus an additional 10 feet. Prior to any clearing/grubbing/investigation/removal activity in the vicinity of these *Stahlia monosperma* specimens, physical barriers (e.g., posts and barricade tape) will be set up. The barriers will be removed following the investigation/remediation activities.

2.5.4 AOC R

Approximately 1,000 tons of debris and soil from the site will be removed in a manner consistent with the type of debris encountered at the site. During the removal, tires and large metal debris will be segregated from the remainder of the waste. The tires will be transported to the island of Puerto Rico for disposal and the large metal will be transported to a recycler on Puerto Rico. The remainder of the waste will be transported to the Vieques Municipal Landfill for disposal. The depth of the excavation will be dependent

upon the extent of the debris and based on visual observations, PID readings, and characterization data.

2.6 CONTINGENCY PLANNING FOR UNANTICIPATED DISCOVERY

The possibility exists of encountering unexpected items of concern (i.e. drums, evidence of release, UXO items, and compressed gas cylinders) during the removal actions. If such an item is encountered, the following contingency plan will be placed into operation:

- The unexpected item or area of soil will be removed from the area, and placed within a previously staged roll off container. Each site will have a roll off container dedicated for such events. Each container will remain empty unless an unanticipated discovery is made.
- The soil immediately surrounding the item or area of concern shall be removed to an extent that visually indicates all affected material has been removed. This material will be placed within the roll-off container for further analysis. A PID shall also be used to confirm that all affected material is captured within the removal action.
- The materials within the roll-off container shall be sampled and characterized to evaluate disposal options. If a material is encountered that does not meet the original waste profile, the regulatory agencies shall be contacted to discuss and concur upon the appropriate disposal methods.
- If an unanticipated UXO item is discovered, the UXO technicians will evaluate and determine the disposal options. Notification to all agencies involved shall take place as soon as the area is determined to be safe by the UXO technicians. The soil immediately surrounding the item shall be treated as described in bullet #2 of this section.
- If it appears as though the unanticipated discovery is creating a point source discharge (i.e. leaking drum), a ground water sample well shall be installed, so that the groundwater immediately beneath the point source discharge is captured within this well. It is the intent of this installation to immediately capture the contaminants of concern that are entering the groundwater, that may or may not be present in the wells immediately down gradient of the excavation areas. Prior to the installation of this well, all agencies involved shall concur on such installation. Sampling of additional wells down gradient of the discovery shall be completed by the Title II services contractor, or their designated representative. This sampling shall be guided by the Master QAPP.

2.7 SITE RESTORATION

All site restoration will be limited to minimal re-grading and erosion reduction measures. No seeding or backfilling shall be performed. If the scenario presents itself such that the soils shall be onsite, the areas shall be re-graded to mimic the existing landscape and promote positive drainage to the extent possible. Specific restoration activities limited to SWMU 6 are as follows:

2.7.1 SWMU 6

Upon receipt of confirmatory analytical data, and if desired by USFWS, mangroves will be planted to stabilize the disturbed areas and to enhance the natural regeneration of the site.

Combinations of mangrove species are already present in the area and will be acquired locally. FSSI/Shaw will grade the soil to follow the original contour of the site and plant mangrove species according to their water level regime and salinity preferences. The USFWS will be consulted concerning the selection of the species of mangroves required. For areas constantly flooded along the canal, red mangrove will be planted because it is most suitable and more likely to survive. Black mangrove will be planted in areas that flood intermittently during tide fluctuations. Either white and/or button mangroves will be planted in drier areas along the road. Spacing will be variable but will follow the natural spacing of the already present mangroves and verified with the USFWS. A USFWS representative shall be present during all plantings to ensure proper installation.

2.8 PROJECT SCHEDULE

The proposed project schedule is presented in **Appendix E**.

3.0 SAMPLING AND ANALYSIS SUMMARY

All sample analytical methods, procedures for soil sample collection, and data analysis methods are discussed in the SAP, **Appendix C**, which incorporates all associated QAPP Worksheets with this Task Order.

4.0 REPORTING

4.1 DATA MANAGEMENT REPORTING

To ensure the integrity of sample analytical data from the time of collection in the field to the tabulation of results, data documentation protocols will be implemented. This will include providing sample labels, chain-of-custody records, and field information forms to document field information; and comparing laboratory analysis reports with tabular displays and graphic displays to evaluate the accuracy of data transfer. The data management process is detailed in the QAPP, Worksheet 29, located in **Appendix C.** The confirmatory data will, as applicable, be used to perform human health and ecological risk assessments in accordance with the HHRA and ERA protocols in the Master QAPP (CH2M HILL, February 2007). The Navy will meet with the regulatory agencies to discuss the results of the risk assessments and confirm that the removal action was sufficient.

A report will be prepared summarizing the data collection and the analytical results for each sampling event including the residual risk assessment. These reports will be submitted after each sampling event for inclusion within the risk analysis. The project manager will be responsible for assessing whether the data collected meets the project Data Quality Objectives (DQOs), as described in **Appendix C** (the SAP) and the project remedial goals as determined by the residual risk assessment. The residual risk assessment will be included as an appendix to the report.

4.2 CONSTRUCTION COMPLETION REPORT PREPARATION AND REVIEW

Upon completion of all field activities, Shaw will prepare a construction completion report for submittal to the Navy and Regulatory Agencies, documenting site activities and reporting all data. This report will include all daily notes from field activities, a description of each project task, any problems encountered along with corrective measures, project photographic documentation, as-build drawings, and all pertinent analytical data/reports. Additionally, this report will include the data collection and analytical results detailed in Section 4.1 of this report. Shaw will submit a draft closeout report to the Navy and Regulatory Agencies for review and comment. Upon receiving review comments, Shaw will incorporate all comments and will submit the report as the final version.

5.0 ENVIRONMENTAL PROTECTION PLAN

5.1 PURPOSE

This plan presents environmental protection measures to be employed during site activities for TO #15. FSSI/Shaw will implement procedures and supply materials necessary for environmental protection for activities associated with these Removal Actions. Principal concerns include small spills and erosion and sediment control. Additional information may be found in the Health and Safety Plan **(Appendix B)**.

5.2 EMERGENCY AND DECONTAMINATION EQUIPMENT

5.2.1 Small-Scale Emergency Equipment

Small scale emergency equipment will include dry chemical, ABC-rated fire extinguishers; spill control equipment; absorbent materials; decontamination equipment; air purifying respirators; radio and telephone equipment; and various hand tools. This equipment will be made accessible to all on-site workers.

5.2.2 Large-Scale Emergency Equipment

Large-scale emergency equipment will consist of an excavator, if equipment of such size and power is necessary. FSSI/Shaw will coordinate with the Municipality of Vieques Fire Department and Civil Defense prior to the mobilization of heavy equipment for the removal action, to determine what emergency equipment is available in the event of an emergency. A list of emergency equipment available in case of an emergency shall be prepared and kept at the site incase of an emergency.

5.2.3 Decontamination Equipment

Equipment necessary for decontamination activities will be provided, installed, and verified in working order if deemed necessary. A decontamination pad will be constructed at the exit to the site. As a minimum all equipment will be dry decontaminated before it departs. Loose oil and debris on the equipment will be removed using brooms. Equipment for the decontamination area includes the following items:

- Clean water supply
- Detergent solution
- Brushes
- Waste containers

5.3 PREVENTATIVE ACTIONS

5.3.1 Equipment Maintenance

Construction equipment will be properly maintained to ensure safe operation. The excavator will be regularly inspected and maintained (and documented in the inspection/maintenance log) in such manner as to minimize spillage or leakage which may occur during on-site transportation operations.

5.4 PROTECTION OF NATURAL RESOURCES

FSSI/Shaw will preserve the natural resources within the project boundaries and outside the limits of disturbance, except as is necessary to implement required work. This process is outlined in Section 2.1 of this document, entitled Clearing and Grubbing/UXO Support. Mangroves will be re-planted at SWMU 6. FSSI/Shaw will take photographs showing existing environmental conditions in and adjacent to the site. Shaw will also conform to contract requirements with respect to the protection of natural resources. All practicable efforts will be made to preserve endangered vegetation in the vicinity of the debris removal areas within AOC J. FSSI/Shaw will remove obvious traces of construction, such as stockpiles of excess waste material and barricades. Prior to the initialization of any activities, FSSI/Shaw will coordinate with USFWS to discuss the impacts to the refuge. Please refer to Section 2.4 of this document for further discussion.

5.5 WASTE HANDLING

Contaminated materials will be excavated and temporarily piled onsite to allow for adequate scheduling of transportation and disposal of the materials. Each day a temporary pile will be constructed. The area will first be protected utilizing a double layer of 8-mil plastic sheeting beneath the excavated material. This area will be used as a loading pile for the transportation and disposal contractor. In all cases this pile shall be staged within the excavation area to minimize the impacts to outside areas. This temporary pile will be removed and plastic sheeting will be disposed of properly each day to prevent the spread of contaminated materials offsite. The T & D activities will be performed in accordance with local, state, and federal regulations and requirements. T & D service will be procured prior to mobilizing to the site. The services will be scheduled based on the schedule of the site activities.

5.6 SEDIMENT AND EROSION CONTROL

FSSI/Shaw will limit the extent of operations to the areas required for excavation. All reasonable attempts will be made to minimize the disturbed area in order to prevent potential damage to the service area. However, if additional erosion protection is needed, silt fencing or other devices will be used. All staging areas shall be placed within the proposed excavations, and shall be constructed so that all storm water run off is directed towards the open excavation

areas. In the event that this construction is not possible, additional soil erosion measures will be installed to direct water towards the open excavations.

6.0 EROSION AND SEDIMENT CONTROL PLAN

This plan describes the erosion and sediment control measures that will be installed at former NASD to limit the migration of soil and sediment from distributed areas. Additional measures are provided for stabilization of the site including minor site grading. Measures to address Storm Water Pollution Prevention are discussed throughout this Erosion and Sediment Control Plan and the Environmental Protection Plan (**Section 5**).

Earth-disturbing activities are as follows:

- Grubbing of vegetation
- Removing surface debris
- Excavation of debris and contaminated soils

Proposed erosion and sediment control measures are as follows:

- Silt fence
- Temporary stone construction entrance
- Temporary diversion dike
- Dust control

6.1 SILT FENCE

Silt fence will be installed at the sites to minimize off-site migration of sediment. At a minimum, silt fence will be installed as part of the site preparation work and will be in place locally prior to any ground disturbance. The silt fence will be inspected during each workday and after every significant precipitation event. FSSI/Shaw will complete any needed repairs promptly following detection. The silt fence will be removed upon establishment of adequate vegetative cover and with the prior authorization of the NTR.

6.2 TEMPORARY STONE CONSTRUCTION ENTRANCE

FSSI/Shaw will construct temporary stone construction entrances, consisting of filter fabric overlain by coarse aggregate (2 to 3 inch diameter) to control migration of sediment onto adjacent roadways. The construction entrances will be removed upon completion of all earthmoving and prior to site restoration.

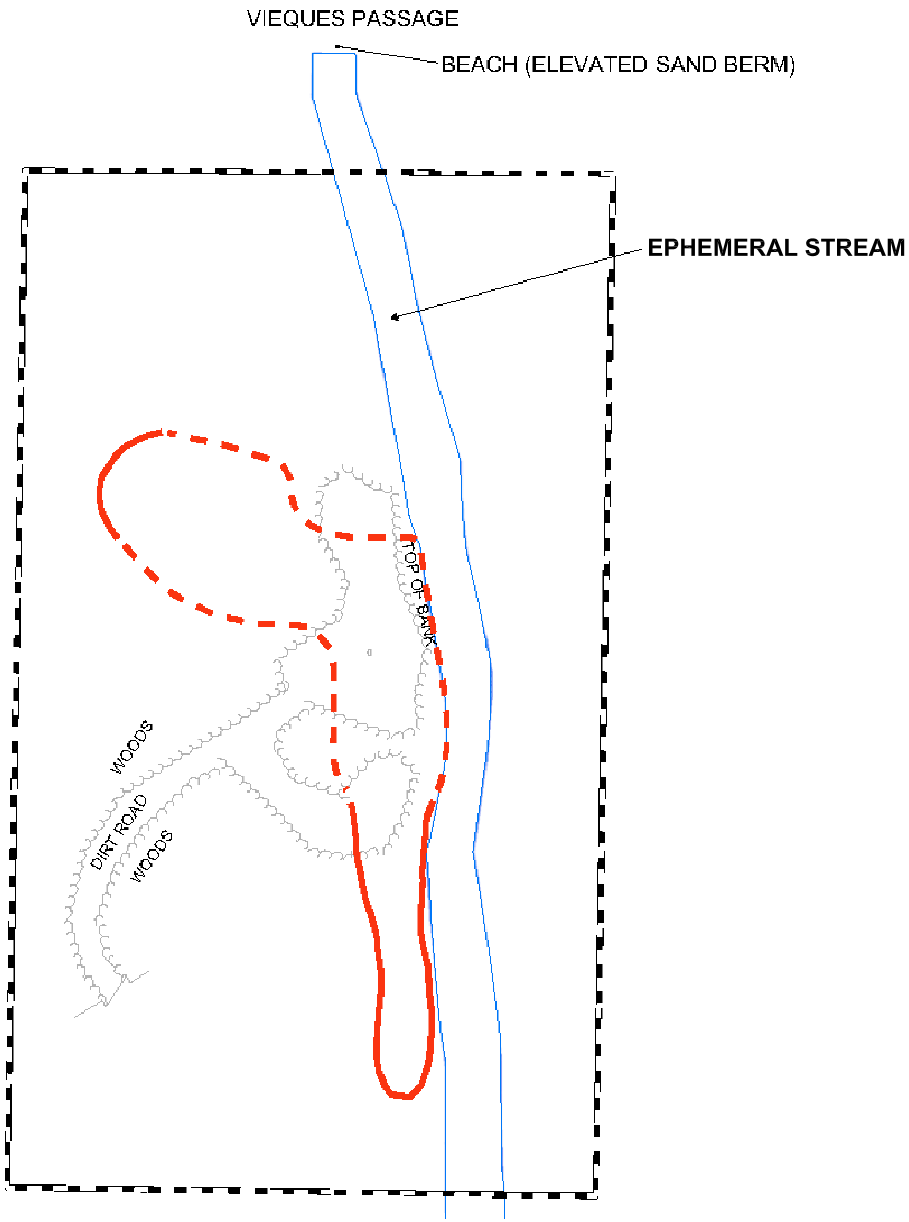
6.3 TEMPORARY DIVERSION DIKES

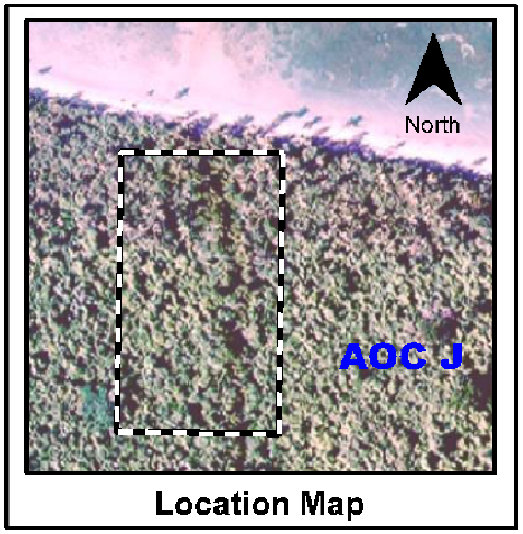
Temporary diversion dikes, consisting of compacted soil, will be used as necessary to divert storm water runoff away from open excavation during removal activities. All diversionary structures shall be installed up-gradient of all excavations.

6.4 DUST CONTROL

Dust control will be performed as necessary in all disturbed areas using a small centrifugal pump, polyethylene water tank, and water authorized for use by the Navy Technical Representative. Dust control will be performed in accordance with the federal, local, and contract regulations. Trucks will be covered to minimize dust dispersion.

Figures





Legend

- Approximate Site Boundary
- Interpreted Waste Boundary - Geophysics (Draft RI)
- Visual Waste Boundary (Draft RI)

0 25 50 100 150 200
Feet

N

SOURCE: **CH2MHILL**

DEPARTMENT OF THE NAVY — NAVAL FACILITIES ENGINEERING COMMAND

MID–ATLANTIC DIVISION

NAVAL STATION — NORFOLK, VIRGINIA

Former Naval Ammunitions Support Detachment — VIEQUES, PUERTO RICO

AOC J SITE MAP

CODE ID. NO.	A
SCALE:	NOT TO SCALE
EFD NO.	
TASK ORDER NO.	0015
SPEC. NO.	
CONSTR. CONTR. NO.	N62470–03–D–4402
NAVFAC DRAWING NO.	110572–A1.DWG
FIGURE:	5

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DRAWN BY	TFR	08/18/06	APPROVED BY	WLH	08/18/06

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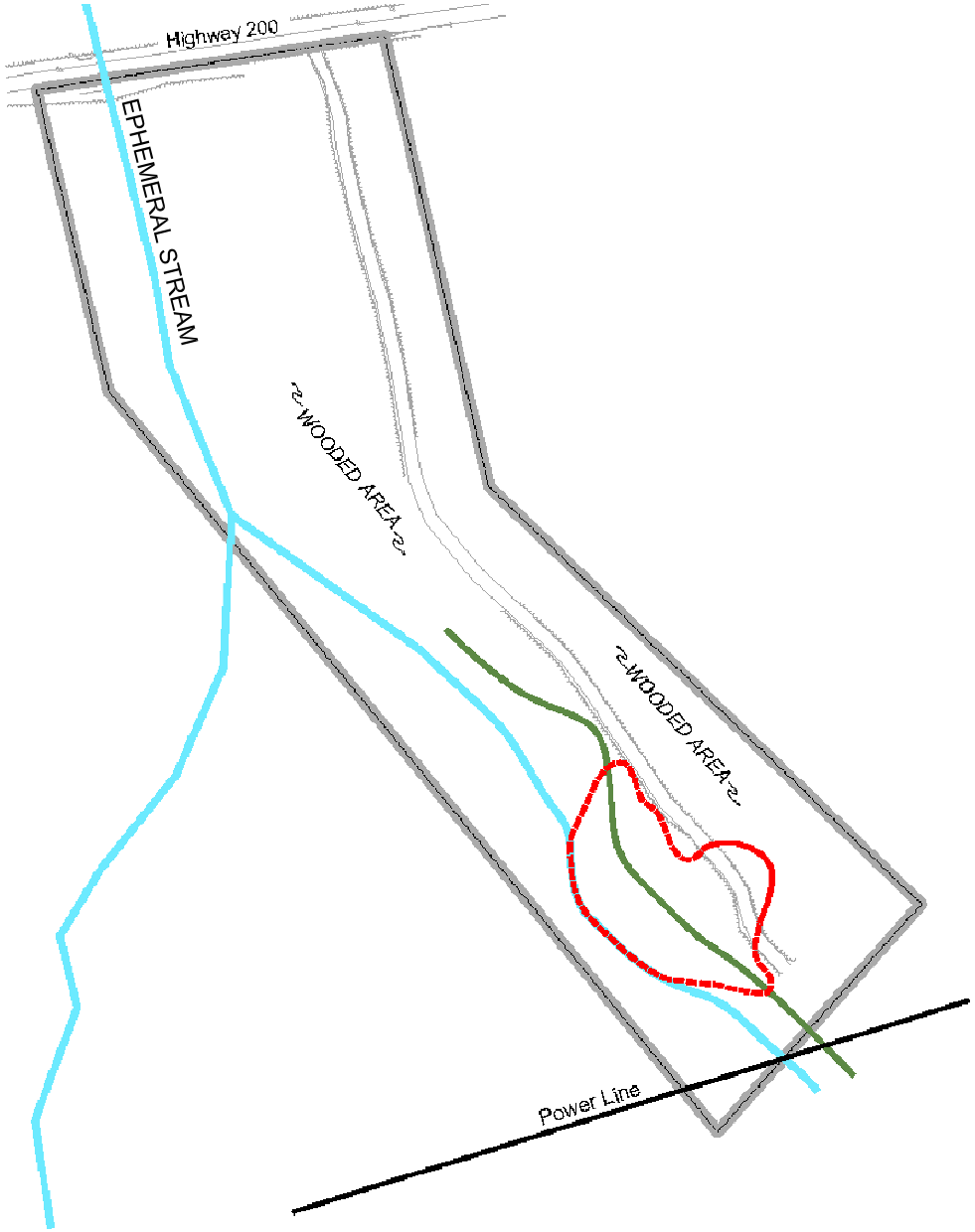

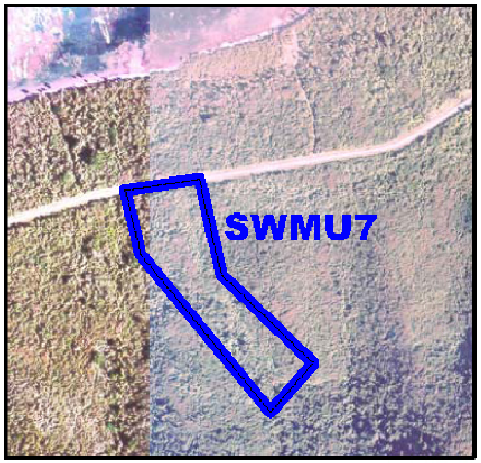

Location Map

Legend

- Approximate Site Boundary
- Interpreted Waste Boundary (Draft RI)
- Visual Waste Boundary (Draft RI)
- Top of Ephemeral Stream
- Ephemeral Stream

100 50 0 100 200 300
Feet

N

SOURCE: **CH2M**HILL

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FIGURE: 4	NAVFAC DRAWING NO. 110572-A1.DWG	CONTR. CONTR. NO. N62470-03-D-4402	SPEC. NO.
TASK ORDER NO. 0015	EFD NO.	SCALE: NOT TO SCALE	CODE ID. NO. A

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MID-ATLANTIC DIVISION

NAVAL STATION — NORFOLK, VIRGINIA

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SWMU 7 SITE MAP

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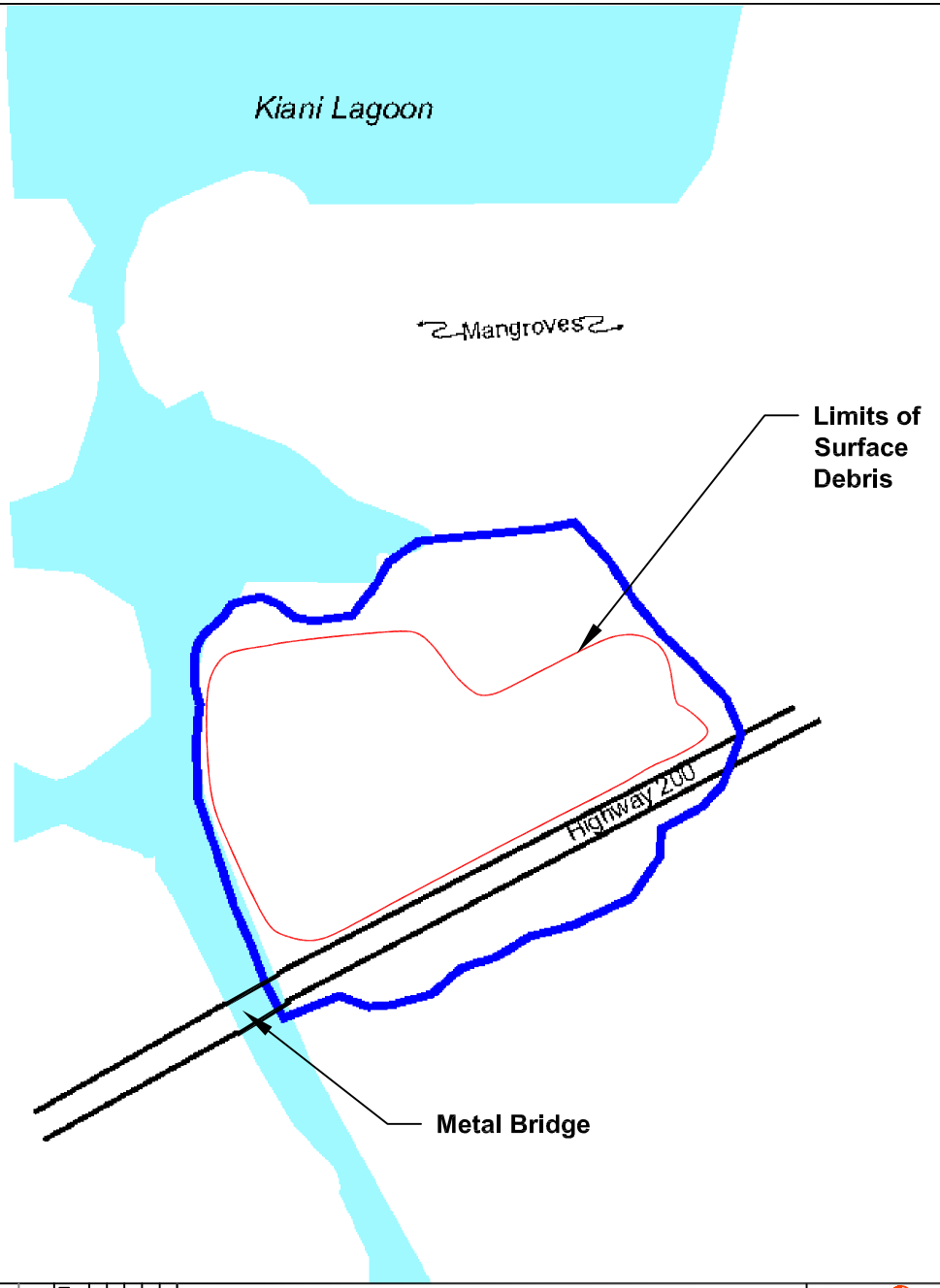

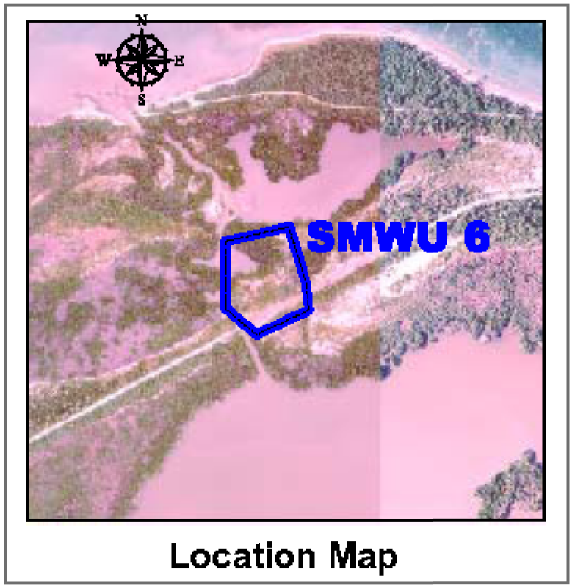

Legend

- Site Boundary
- Surface Debris Limits

N

0 90 180

Scale in Feet

SOURCE: **CH2MHILL**

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MID–ATLANTIC DIVISION

NAVAL STATION — NORFOLK, VIRGINIA

Former Naval Ammunitions Support Detachment — VIEQUES, PUERTO RICO

SWMU 6 SITE MAP

CODE ID. NO.	A
SCALE:	AS SHOWN
EFD NO.	
TASK ORDER NO.	0015
SPEC. NO.	
CONSTR. CONTR. NO.	N62470–03–D–4402
NAVFAC DRAWING NO.	116701–A3–r
FIGURE:	3

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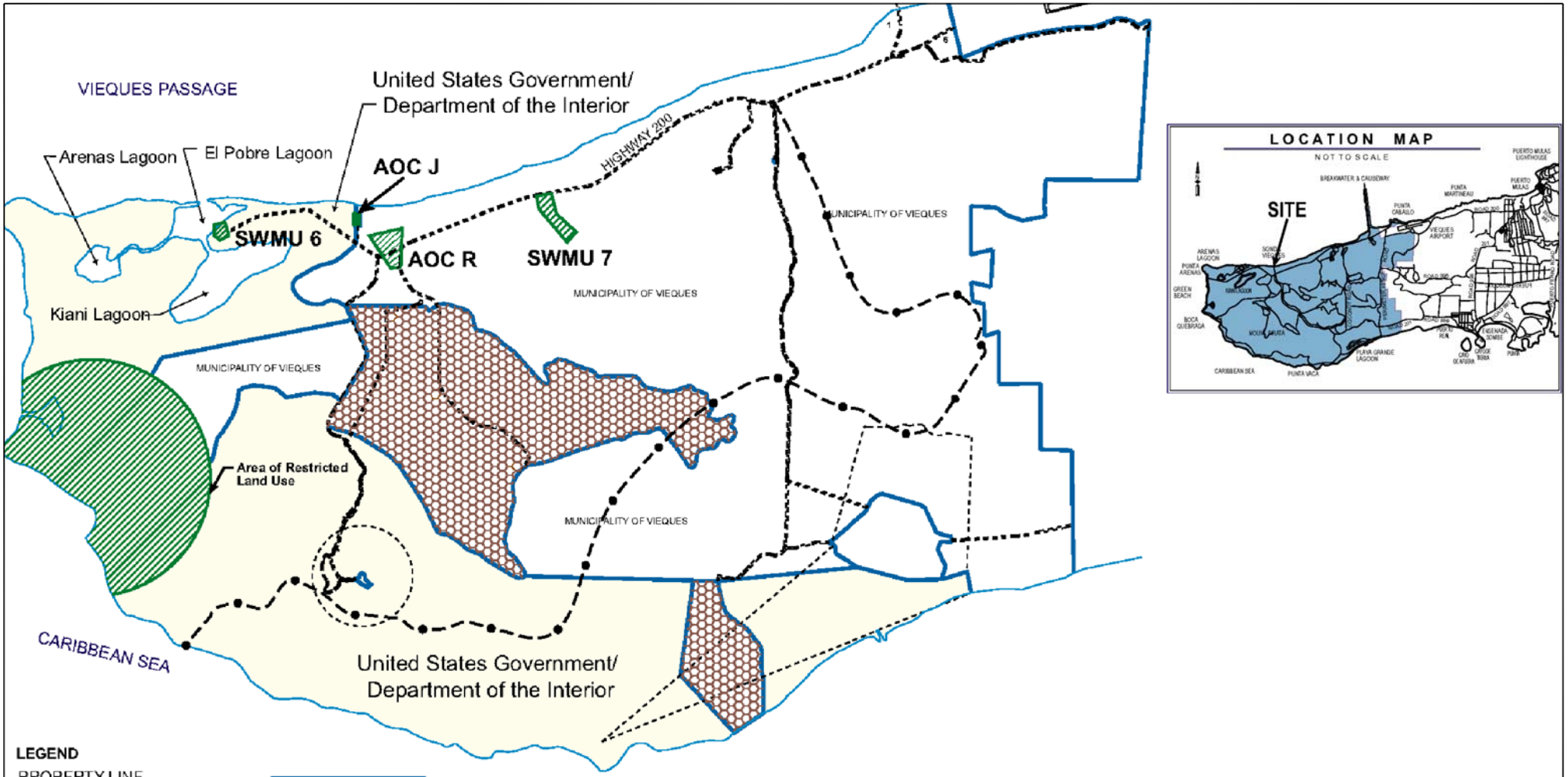

SOURCE: CH2MHILL

DEPARTMENT OF THE NAVY			NAVAL FACILITIES ENGINEERING COMMAND		
MID–ATLANTIC DIVISION					
NAVAL STATION			NORFOLK, VIRGINIA		
Former Naval Ammunitions Support Detachment			VIEQUES, PUERTO RICO		
SWMU 6, SWMU 7, AOC J LOCATION MAP					
DESIGNED BY	LM	08/18/06	CHECKED BY	WLH	08/18/06
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FIGURE: 2
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SPEC. NO.
TASK ORDER NO. 0015
EFD NO.
SCALE: NOT TO SCALE
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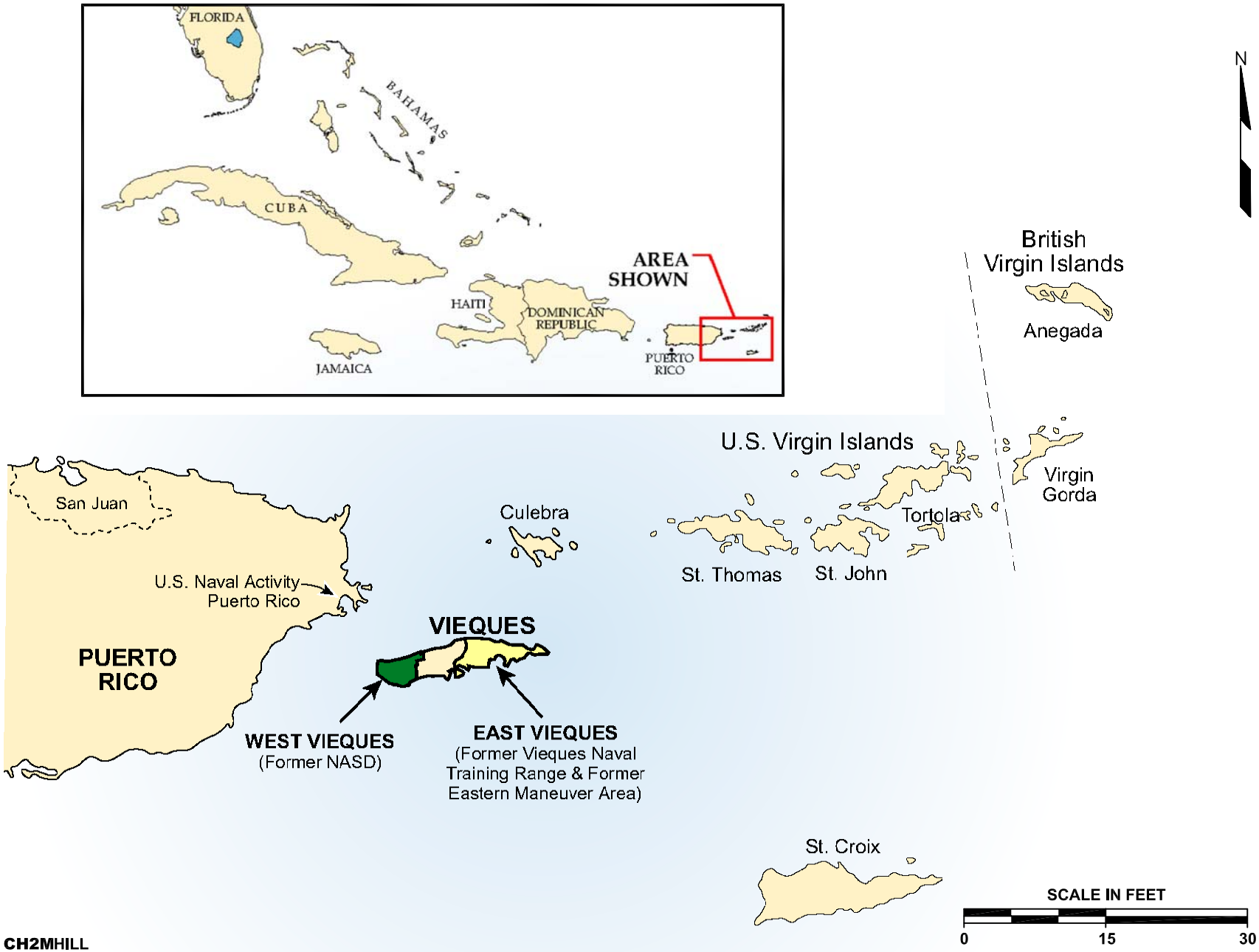

SOURCE: **CH2MHILL**

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TASK ORDER NO. 0015	EFD NO.	SCALE: NOT TO SCALE	CODE ID. NO. A

DEPARTMENT OF THE NAVY — NAVAL FACILITIES ENGINEERING COMMAND

MID-ATLANTIC DIVISION

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REGIONAL LOCATION MAP

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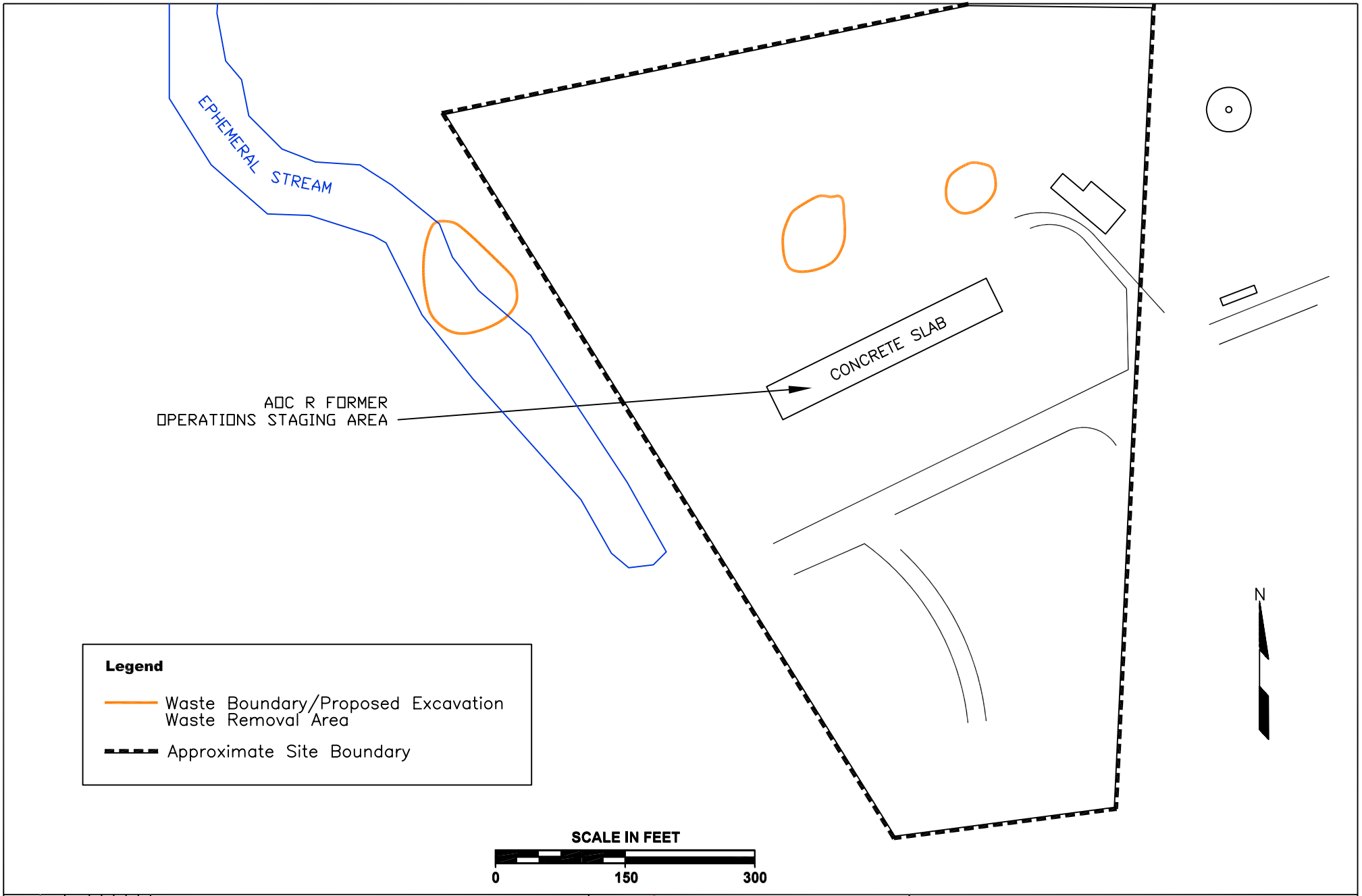

DEPARTMENT OF THE NAVY — NAVAL FACILITIES ENGINEERING COMMAND

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NAVAL STATION — NORFOLK, VIRGINIA

Former Naval Ammunitions Support Detachment — VIEQUES, PUERTO RICO

AOC R SITE MAP

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CODE ID. NO. A
SCALE: 1"=150'
EFD NO.
TASK ORDER NO. 0015
SPEC. NO.
CONSTR. CONTR. NO. N62470-03-D-4402
NAVFAC DRAWING NO. 116701-A6.DWG
FIGURE: 6

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QUALITY CONTROL PLAN
REMOVAL ACTIONS
SWMU 6, SWMU 7, and AOC J
NAVAL AMMUNITIONS SUPPORT DETACHMENT
VIEQUES, PUERTO RICO

Prepared for:

DEPARTMENT OF THE NAVY
Contract No. N62470-03-D-4402
Task Order No. 0015
NAVFAC Mid-Atlantic
Norfolk, VA

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Ernest P. Duke
Program QC Manager

James A. Dunn, Jr., P.E.
Program Manager

March 2007
Project 116701

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STATEMENT OF QC PROGRAM

Shaw Environmental Inc. (Shaw) will be performing the quality control (QC) function for Task Order 0015, Contract No. N62470-03-D-4402. This task order is for Removal Actions at SWMU 6, SWMU 7, AOC J, and AOC R, former Naval Ammunitions Support Detachment, Vieques, Puerto Rico. This Site Specific Addendum is prepared and submitted to incorporate the methods and procedures from the Shaw Program Quality Control Plan (PQCP) approved by the Atlantic Division, Naval Facilities Engineering Command, which is provided in Attachment 1. Shaw will perform the inspections and tests required to ensure that materials, workmanship, and construction conform to the Design Drawings, Project Specifications, and contract requirements.

Note to Employees – Concept of Quality Control

Quality control should not be considered a person or an organization of personnel, but a concept to perform in such a manner that the end product of our efforts provides a quality product and customer satisfaction. The quality control individual or group cannot inspect quality into the final product, but only inspect and document the results of our efforts. The only people that can build quality into the product are the individuals performing the task of producing the end product.

It should be noted by all employees that the documentation requirements of Shaw procedures, plans, and the Task Order Specifications are considered equally as important as the end product itself. When it is stated that the documentation will be approved prior to the start of work, this is exactly what is intended. To eliminate problems in this area requires careful planning and execution by everyone.

We would do well to remember that our livelihood depends on how well we satisfy our customer. To accomplish this requires teamwork and attention to detail by all employees and contractors.

I. QUALITY CONTROL ORGANIZATION

The Site QC Manager for this Task Order will have the authority to implement and manage this QC Plan, the three phases of quality control and the authority to stop work, which is not in compliance with the contract.

The Site QC Manager will function as both the QC Manager and also as the Site Health and Safety Officer for this Task Order. This individual maintains current US Army Corps of Engineers (USACE) Construction Quality Management for Contractors, QC certification.

The relationship between the QC organization, and production personnel of the TO, is provided in the Organizational Chart, Figure I-1.

II. IDENTIFICATION OF PERSONNEL ASSIGNED TO THE QC ORGANIZATION

The resume of the Site QC Manager is provided as Figure II-1.

III. APPOINTMENT LETTER

The Site QC Manager Appointment letter is presented as Figure III-1.

IV. OUTSIDE ORGANIZATIONS

A list of outside organizations such as subcontractors employed by Shaw for work under this task order is provided in Exhibit IV-1. This list provides each firm's name and address and a description of the services each firm will provide. This list will be maintained current and will be available for review.

V. INITIAL SUBMITTAL REGISTER & REVIEWER

V.1 Submittal Register

The Initial Submittal Register is provided as Exhibit V-1. The status of each submittal will be recorded and maintained by the QC Manager. The Submittal Register will be submitted at the end of each month.

V.2 Personnel Authorized to Review and Certify Submittals

Personnel authorized to review and certify submittals other than the Site QC Manager are identified on Exhibit V-2. The QC Manager shall perform the final review and approval/certification for all submittals. Any additional personnel assigned to perform submittal review and certification must be approved by the CO, prior to performance.

VI. TESTING LABORATORY ACCREDITATION

Testing laboratory accreditation requirements are addressed in the Program QC Plan Section 1, part 4.7, Construction Testing.

VII. TESTING PLAN & LOG PREPARATION

A Testing Plan and Log has been prepared for this TO and is provided as Exhibit VII-1.

VIII. REWORK ITEMS

Rework Items will be documented on the Daily QC Report and on the Rework Items Log. This will be used to report and track rework Items. The Rework Items Log will be submitted at the end of each month.

IX. DOCUMENTATION PROCEDURES

The Daily QC Report form and Daily Production Report form will be used to document daily activities at the site. Documentation will be completed as discussed in Section 1, Parts 4.5 and 4.9 of the Program QC Plan.

X. QUALITY CONTROL INSPECTION PLAN

The Quality Control Inspection Plan, Exhibit X-1, lists each specification section and definable feature of work with provisions for recording the corresponding checklist/report for each phase of the three-phase control process. As each control phase is satisfactorily preformed, the Site QC Manager will record the corresponding checklist/report number and date.

Note: A definable feature of work is a task, which is separate, and district from other tasks and requires separate control procedures.

This list has been prepared to correspond to the activities of the project schedule. It will be maintained as provided in Section 1, Part 4.4 of the PQCP. The list will be agreed upon during the Coordination and Mutual Understanding Meeting. The list will be keyed to the construction schedule. Each preparatory, initial and follow-up phase checklist/report will reflect the construction activity number derived from the construction schedule.

XI. PERSONNEL MATRIX

The Site QC Manager will prepare and maintain the personnel matrix, Exhibit XI-1, showing each section of the TO specification with identification of who will review and certify submittals, who will perform and document the three phases of control, and who will perform and document testing. This matrix should be completed as much as possible prior to and during site mobilization. The matrix will be maintained current by the Site QC Manager and will be available for review.

FIGURE I-1

QC ORGANIZATIONAL RELATIONSHIP WITH PRODUCTION PERSONNEL SHAW ENVIRONMENTAL INC., FSSI TASK ORDER NO. 0015

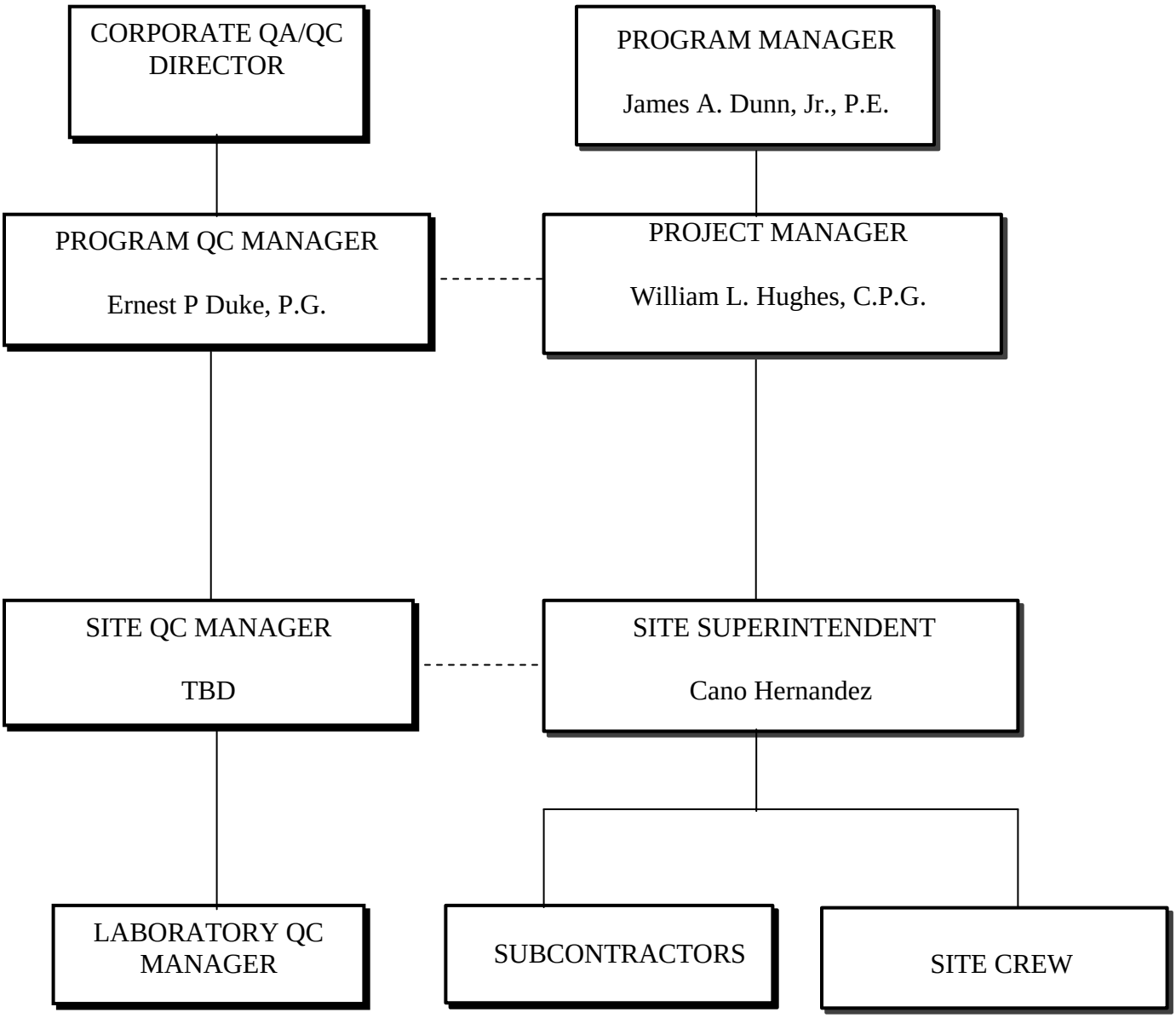

Figure II-1
Site QC Manager / Alternate Resumes

(To be provided)

Figure III-1
Site QC Manager / Letter of Appointment

Shaw Environmental, Inc.

June , 2007

Shaw Environmental, Inc.
500 E. Main Street, Suite 1630
Norfolk, VA 23517

RE: Site QC Manager
Contract N62470-03-D-4402
Task Order No. 0015

Dear :

This letter will serve as your appointment as the Site Quality Control Manager on the referenced project and will also clarify your duties and authority in this position. In this position, you will be authorized to use available resources to satisfy all applicable requirements of the Program and Task Order Quality Control Plans.

This authorization specifically gives you the authority to direct removal and replacement or correction of nonconforming materials or work and stop work authority when continuation would be unsafe to personnel, harmful to the environment, or result in a significant degradation of quality.

You will be expected to work closely with the Project Manager, Site Supervisor and other project personnel, but you will not be directly responsible to anyone but myself for resolution of quality issues when working in the capacity of Quality Control Manager.

If you have any question in this matter, please contact me.

Respectfully,

Ernest P. Duke
Shaw Program QC Manager
NAVFAC LANTDIV RAC Program

EXHIBIT IV-1

SUBCONTRACTOR LIST Field Support Services, Inc. / Shaw Environmental, Inc. Removal Actions at former NASD Task Order No. 0015	
COMPANY NAME AND ADDRESS	**DESCRIPTION OF SERVICES PROVIDED**
TBD (Private Contractor)	Utility Locations
TBD	Site Survey
Accutest Laboratories	Chemical Analytical Testing
Vieques Municipal Landfill	Disposal Services

EXHIBIT V-1

SUBMITTAL REGISTER

Task Order No. 0015 Contract No. N62470-03-D-4402	Project No. 116701 Project Title: Removal Actions at SWMU 6, SWMU 7, and AOC J, former NASD, Vieques, Puerto Rico	Revision No. 01

							Contractor Action			Approving Authority					
Spec. Sect.	SD Number and Submittal Description	Spec Paragraph Number	Approving Authority	Other Reviewers	Transmittal Control No.	Planned Submittal Date	Action Code	Date of Action	Date Forwarded to Appr Auth/ from Contr	Date Forward to Other Reviewer	Date Rcvd from Other Reviewer	Action Code	Date of Action	Date Rcvd from Appr Auth	Remarks
(a)	(b)	(c)	(d)	(e)	(f)	(g)	(h)	(i)	(j)	(k)	(l)	(m)	(n)	(o)	(p)
	SD-01 Preconstruction														
	Work Plan		G												
	Health and Safety Plan		G												
	Quality Control Plan		G												
	Sampling and Analysis Plan		G												
	Base Permits		G												
	SD-11 Closeout Submittals														
	Contractor's Closeout Report					Closeout Report									
	As-Built Survey and Records					Closeout Report									
	Status Reports					Closeout Report									
	Environmental Conditions Report					Closeout Report									
	Test Results Summary Report					Closeout Report									
	Daily Contractor Production Reports					Closeout Report									
	Daily QC Reports					Closeout Report									
	Rework Items List					Closeout Report									
	SD-13 Certifications														
	Testing Laboratory Accreditation					Closeout Report									
	SD-10 Test Reports														
	Lab Test Results of Confirmation Samples					Closeout Report									
	Lab Test Results of Waste Soils					Closeout Report									
	SD-08 Statements														
	Disposal Facility Permits					Closeout Report									
	SD-18 Records														
	Shipment Manifests					Closeout Report									

Notes:
Approved By:
G: Government
Blank: QC Manager
Reference; QMP-16.2 Submittal Management Process

ACTION CODES
A: Approved
AN: Approved as Noted
NR: Not Reviewed
RR: Disapproved; Revise and Resubmit

EXHIBIT V-2

LIST OF PERSONNEL AUTHORIZED TO REVIEW AND CERTIFY SUBMITTALS Field Support Services, Inc. / Shaw Environmental, Inc. Removal Actions at former NASD Task Order No. 0015		
	SUBMITTAL TYPE:	AUTHORIZED PERSONNEL:
Work Plan Submittals	Preconstruction	Project Manager
All Work Plan/ Addendum Sections	All Types	QC Manager (1), Program QC Manager, Project Manager and Site Engineer

(1) Note: QC Manager shall perform the final review and certification for submittals requiring Government approval.
QC Manager shall perform the final review and approval for submittals requiring Contractor approval.

EXHIBIT VI-1

TESTING PLAN AND LOG
Field Support Services, Inc. / Shaw Environmental, Inc.
Removal Actions at former NASD
Task Order No. 0015

CONTRACT NUMBER	PROJECT TITLE: Removal Actions at SWMU 6, SWMU 7, AOC J, and AOC R
N62470-03-D-4402	LOCATION: former NASD, Vieques, Puerto Rico

WORK PLAN SECTION AND PARAGRAPH NUMBER	ITEM OF WORK	TEST REQUIRED	ACCREDITED/ APPROVED LAB		SAMPLED BY	TESTED BY	LOCATION OF TEST		DATE COMPLETED	DATE FORWARDED TO CONTR. OFF.	REMARKS
			YES	NO			ON SITE	OFF SITE			
WP 2.3	Waste Characterization Testing	Full TCLP (VOCs, SVOCs, Herbicides, Pesticides, Metals) TPH DRO/GRO, total cyanide and sulfide, corrosivity, ignitability, Explosives, TAL Metals, TCL Volitiles, TCL Semivolitiles, TCL Pest/PCBs									See QAPP - Worksheet 28
WP 2.5.1	SMWU 6 Floor Confirmation Testing	Explosives, TAL Metals, TCL Volitiles, TCL SemiVolitiles, TCL Pest/PCBs, TPH DRO/GRO SW-846 methodology									See QAPP - Worksheet 28
WP 2.5.2	SMWU 7 Floor Confirmation Testing	Explosives, TAL Metals, TCL Volitiles, TCL SemiVolitiles, TCL Pest/PCBs, TPH DRO/GRO SW-846 methodology									See QAPP - Worksheet 28
WP 2.5.3	AOC J Floor Confirmation Testing	Explosives, TAL Metals, TCL Volitiles, TCL SemiVolitiles, TCL Pest/PCBs, TPH DRO/GRO SW-846 methodology									See QAPP - Worksheet 28
WP 2.5.4	AOC R Floor Confirmation Testing	Explosives, TAL Metals, TCL Volitiles, TCL SemiVolitiles, TCL Pest/PCBs, TPH DRO/GRO SW-846 methodology									See QAPP - Worksheet 28

EXHIBIT VII-1

REWORK ITEMS LIST
Field Support Services, Inc. / Shaw Environmental, Inc.
Removal Actions at former NASD
Task Order No. 0015

Contract No. N62470-03-D-4402

Removal Actions at former NASD

Location: Vieques, Puerto Rico

NUMBER	DATE IDENTIFIED	DESCRIPTION	CONTRACT REQUIREMENT (Spec. Section and Par. No., Drawing No. and Detail No., etc.)	ACTION TAKEN BY QC MANAGER	RESOLUTION	DATE COMPLETED

EXHIBIT VIII-1

QUALITY CONTROL INSPECTION PLAN
Field Support Services, Inc. / Shaw Environmental, Inc.
Removal Actions at former NASD
Task Order No. 0015

Specification Section	Definable Feature of Work	Subtasks	Control Check Verification					
			Date	Preparatory Phase Checklist/Report No.	Date	Initial Phase Checklist/Report No.		Follow-up Phase Checklist/Report No.
WP 2.1	Mobilization, Site Preparation							
WP 2.3	Disposal Characterization Sampling							
WP 2.1	Clearing and Grubbing							
WP 2.5	Soil and Debris Removal							
WP 2.5	Confirmation Sampling and Analysis							
WP 2.5	Background Sampling and Analysis							
WP 2.7.1	SWMU-6 Restoration							

EXHIBIT IX-1

PERSONNEL MATRIX Field Support Services, Inc. / Shaw Environmental, Inc. Removal Actions at former NASD Task Order No. 0015			
SPECIFICATION SECTION	SUBMITTALS TO BE REVIEWED BY:	THREE PHASE TO BE PERFORMED BY:	TESTING TO BE PERFORMED BY:
All Sectons	Site QC Manager (1), Program QC Manager, Project Manager or Project Engineer	Site QC Manager	Chemical Analysis - Accutest Laboratories

(1) Note: QC Manager shall perform the final review and certification for submittals requiring Government approval.
QC Manager shall perform the final review and approval for submittals requiring Contractor approval.

SITE SPECIFIC HEALTH AND SAFETY PLAN FOR INTERIM REMOVAL ACTIONS AT SMWU-6, SWMU-7, AOC J AND AOC-R WEST VIEQUES, PUERTO RICO

Prepared for:

DEPARTMENT OF THE NAVY
Atlantic Division
Naval Facilities Engineering Command
6506 Hampton Boulevard
Building A (South East Wing) 3rd Floor
Norfolk, VA 23508

Contract No. N62470-03-D-4402
Task Order 0015

Prepared by:

SHAW ENVIRONMENTAL, Inc.
500 E. Main Street, Suite 1630
Norfolk, VA 23517

Project No. 116701
March 2007

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Appendix J Blood Borne Pathogen Exposure Control Plan

List of Acronyms

AHA	Activity Hazard Analyses
AOC	Area of Concern
BAC	Blood Alcohol Concentration
CIH	Shaw Program Certified Industrial Hygienist
CFR	Code of Federal Regulation
CRZ	Contamination Reduction Zone
DBA	A-Weighted Decibel
°C	Degrees Celsius
°F	Degrees Fahrenheit
FSSI	Field Support Services, Inc.
EMA	Emergency Management Agency
EMS	Emergency Response Service
EPA	Environmental Protection Agency
ERCP	Emergency Response Contingency Plan
EV	Electron Volt
EZ	Exclusion Zone
F/B	Flash/Bang
FSSI	Field Support Services, Inc.
LEL	Lower Explosive Limit
mg/m^3	Milligrams per cubic meter
Mph	Miles Per Hour
MSDS	Material Safety Data Sheet
NASD	Naval Ammunitions Support Detachment
NIOSH	National Institute for Occupational Safety and Health
NRR	Noise Reduction Rating
O_2	Oxygen
OSHA	Occupational Safety and Health Administration
PEL	Permissible Exposure Level
HSM	Program Health and Safety Manager
PM	Project Manager
PPE	Personal Protection Equipment
ppm	Parts Per Million
UXO	Unexploded Ordinance

ROICC	Resident Officer In Charge of Construction
RPM	Remedial Program Manager
Shaw	Shaw Environmental, Inc.
SS/SSO	Site Superintendent/Site Safety officer
SSHSP	Site-Specific Safety and Health Plan
SWMU	Solid Waste Management Unit
SZ	Support Zone
TLV	Threshold Limit Value
TWA	Time-Weight Average
UV	Ultraviolet

1.0 INTRODUCTION

1.1 OBJECTIVE

The objective of this plan is to provide a mechanism for establishing safe working conditions at the site. The safety organization, procedures, and protective equipment have been established based upon an analysis of potential hazards. Specific hazard control methodologies have been evaluated and selected to minimize the potential of accident or injury.

1.2 POLICY STATEMENT

The policies of Field Support Services, Inc (FSSI) and Shaw Environmental, Inc (Shaw) are to provide a safe and healthful work environment for all employees. FSSI/Shaw considers no phase of operations or administration to be of greater importance than injury and illness prevention. Safety takes precedence over expediency and shortcuts. At FSSI/Shaw, it is believed all accidents and injuries are preventable. Shaw will take every reasonable step to reduce the possibility of injury, illness, or accident.

This Site Specific Health and Safety Plan (SSHSP) prescribes the procedures that must be followed during referenced site activities. Operational changes that could affect the health and safety of personnel or the community will not be made without the prior approval of the Project Manager and the Health and Safety Coordinator.

The provisions of this plan are mandatory for all personnel and subcontractors assigned to the project. All visitors to the work site must abide by the requirements of the plan.

1.3 REFERENCES

This SSHSP complies with applicable Occupational Safety and Health Administration (OSHA), U.S. Environmental Protection Agency (EPA), and Shaw Health & Safety policies and procedures. This plan follows the guidelines established in the following:

- Standard Operating Safety Guides, EPA (Publication 9285.1-03, June 1992).
- Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, National Institute for Occupational Safety and Health (NIOSH), OSHA, U.S. Coast Guard (USCG), EPA (86-116, November 1985).

- Title 29 of the Code of Federal Regulations (CFR), Part 1910.
- Title 29 of the Code of Federal Regulations (CFR), Part 1926.
- USACE Safety and Heath Requirements Manual EM 385-1-1.
- Shaw Health and Safety Procedures HS001 through HS999 (current).

1.4 DISCLAIMER

The enclosed Site Specific Health and Safety Plan has been designed for the methods presently contemplated by FSSI/Shaw for execution of the proposed work. Therefore, the Site Specific Health and Safety Plan may not be appropriate if the work is not performed by or using the methods presently contemplated by FSSI/Shaw or if the scope of work is modified. Each company or contractor is responsible for the safety and health of their personnel, for their actions, and for the work they perform. It is highly recommended that each company or contractor working at the site perform their work under the supervision of their internal health and safety professionals.

2.0 SITE HISTORY/SCOPE OF WORK

2.1 SITE HISTORY

2.1.1 SWMU 6

SWMU 6 is located approximately 100 feet south of the Vieques Passage and was used for disposal of solid wastes, generated by Navy operations within the former Naval Ammunitions Support Detachment (NASD) during the 1960's and 1970's. Waste discarded at the site includes: rusty metal, car parts, rubber tires, broken glass, rubble, and empty containers of lubricants, oil, solvents, and paints. Many items have deteriorated due to natural corrosion in the saltwater environment and are in the form of small pieces of metallic or glass debris. This Removal Action addresses only removal of the wastes, including debris and soil. No munitions and explosives of concern (MECs) have been identified at SWMU 6 during the two MEC avoidance surveys performed prior to the Expanded Preliminary Assessment/Site Investigation (PA/SI) and the Remedial Investigation (RI). However, munitions related items such as inert concrete filled bombs, empty bomb dispensers, and empty shell casings were identified. The main area of debris is just north of Highway 200 and runs along the road. The pile is approximately 165 feet long by 10 feet wide by two feet deep. Additionally, there are five large concrete filled bombs partially buried in water and sediment on the site. The total estimated waste present at SWMU 6 is 450 tons.

2.1.2 SWMU 7

SWMU 7 is approximately 1,100 feet south of the Vieques Passage. From the early 1960's to the late 1970's, a steep ravine at the site was used for disposal of solid waste materials. Discarded materials disposed on SWMU 7 include old tires, sheet metal, empty containers such as drums, cans, and bottles, old batteries, and construction rubble. No known hazardous chemical or waste disposal occurred at this site. SWMU 7 is currently located on Municipality of Vieques property. Disposal activities appear to have been concentrated in a segment of the ravine approximately 420 feet along the length of the dirt access road where waste materials were pushed over the edge. Most of the waste material is confined to the steep slopes. Debris also includes: two sea mines, six rocket launchers, a Volkswagen Beetle, and hundreds of tires. Based on the results of the RI, site conditions at SWMU 7 do not pose an unacceptable risk to human health or the environment. As a result, this Removal Action addresses only waste, including debris and soil containing debris, located at SWMU 7. The total estimated waste present is 9,000 tons.

2.1.3 AOC J

AOC J is located two miles west of the entrance to the former NASD and north of Road 200. The area extends approximately 50 feet south of the Vieques passage. AOC J was used as a solid waste disposal site for construction staging activities from 1965 to 1973. Most of the solid waste at AOC J is on the slopes of the ditch, with additional debris piles located up to 100 feet west of the ditch. The debris piles consist of metallic and non-metallic materials and one empty drum. Site conditions at AOC J do not pose an unacceptable risk to human health or ecological receptors. As a result, this Removal Action addresses only removal of the debris and soil containing debris located at AOC J. A MEC avoidance survey conducted in May 2000 found no active ordinance and explosives (OE) at the site. However, 105-mm shell casings and empty ammunition boxes were observed. A geophysical survey was conducted at AOC J to delineate the extent of buried waste. Subsurface debris does not extend beyond the ditch's western bank. Total waste present at AOC J is approximately 600 tons.

2.1.4 AOC R

AOC R is located approximately 2 miles west of the entrance to the former NASD, and several hundred feet south of the Vieques Passage along Highway 200. A portion of the western boundary is adjacent to an ephemeral stream. This stream is the same ephemeral stream adjacent to AOC J, which is topographically down gradient of AOC R. AOC R was used as a public works operational area from 1965 to 1971. The debris piles consist of a practice bomb, ammunition cans, metal pipes, rocket launchers, wood power poles, tires, white corrugated roofing sheet material, and munitions items. Total estimated waste present is 1,000 tons.

2.2 SCOPE OF WORK

This Health and Safety Plan focuses on the removal of 11,500 tons of debris and soil from the site in a manner consistent with the types of contaminate encountered at the site. The excavation may be up to three-feet below ground surface (bgs).

The principle tasks to be conducted are listed below:

- Site Mobilization/Demobilization;
- UXO Clearance;
- Clearing and Grubbing;
- Soil and Debris Segregation and Removal;
- Collection of Soil Samples;

- Material Soil Load-out and Disposal;
- Equipment Decontamination;

All site activities have been analyzed for potential hazards for which control measures are provided in Appendix C, Activity Hazard Analyses.

3.0 KEY PERSONNEL AND MANAGEMENT

The Project Manager (PM), Site Superintendent/Site Safety Officer (SS/SSO), NAVFAC Mid-Atlantic Program Certified Industrial Hygienist (CIH) and NAVFAC Mid-Atlantic Program Health and Safety Manager (HSM) are responsible for formulating and enforcing health and safety requirements, and for implementing this SSHSP. The following summarizes the health and safety responsibilities of the site management.

3.1 PROJECT SAFETY RESPONSIBILITES

The PM has the overall responsibility for the project and to assure that the requirements of the contract are attained in a manner consistent with the SSHSP requirements. The PM will coordinate with the SS/SSO, to assure that the work is completed in a manner consistent with the SSHSP. The SS/SSO is responsible for field implementation of the SSHSP. The SS/SSO will be the main contact in any on-site emergency situation and will insure off-site emergency agencies have been contacted prior to the start of work. The PM, CIH, HSM, and SS/SSO are authorized to administer this SSHSP. All site personnel are authorized to stop work when an imminent health or safety risk exists. The PM and CIH are responsible for reviewing the SSHSP and ensuring that the SSHSP is complete and accurate. The HSM will also provide technical and administrative support for the Health and Safety Program and will be available for consultation when required. Each employee is responsible for personal safety as well as the safety of others in the work area.

3.2 KEY SAFETY PERSONNEL

The following individuals share responsibility for health and safety at the site:

Shaw Project Manager	William Hughes (757) 318-5140 (office) (757) 438-8498 (cellular)
Shaw Site Superintendent/Site Safety Officer	Cano Hernandez (757)544-7687 (cellular)
US Navy Environmental Representative	Madeline Rivera 787-865-4152

Shaw Certified Industrial Hygienist	David Mummert, CIH (419) 425-6129 (office) (419) 348-1544 (cellular)
Shaw Health and Safety Manager	Kym Edelman (757) 318-5132 (office) (757) 435-5384 (cellular)
Shaw Regional Health and Safety Manager	Joe Hoyt, CHST (804) 337-6982 (cell)

4.0 ACTIVITY HAZARDS

No previous sampling for site contaminants was available to Shaw, thus the initial chemical concerns are based on suspected landfill gases. The presence of landfill gasses has not been confirmed, however, any landfill which received organic material has the potential for methane and potentially hydrogen sulfide gasses to evolve.

4.1 CHEMICAL HAZARDS

A brief summary of exposure limits and health hazards associated with hydrogen sulfide and methane are provided in Table 4.2.

TABLE 4-2
Summary of Site Contaminants

CHEMICAL	EXPOSURE ROUTES	PEL/TLV	HEALTH HAZARDS/ PHYSICAL HAZARDS
Hydrogen Sulfide	Skin, eye, inhalation	10 ppm	• Eye, upper respiratory tract irritant; rotten eggs odor; high concentrations (200+ppm) causes paralysis of sense of smell, (500+ppm) respiratory paralysis, collapse and coma, (>1000 ppm) can be fatal in minutes • Strongly corrosive to electrical connections and equipment; reacts with copper, oxidizing agents, nitric acid, soda lime, and peroxides; flammable gas, can build up in excavations and low lying areas
Methane	Inhalation	1,000 ppm	• A simple asphyxiant; displaces oxygen in low areas of poor ventilation; odorless • A severe fire, explosion hazard; flashes, explodes in contact heat sources, hot surfaces, sources of ignition; reacts with oxidizers; can flash back

The following general symptoms may indicate exposure to a hazardous chemical. Personnel will be removed from the work site and provided immediate medical attention if the following symptoms occur:

- Loss of appetite
- Weakness in wrists or ankles
- Dizziness or stupor
- Nausea, headaches, or cramps
- Irritation of the eyes, nose, or throat
- Chest pains and coughing
- Rashes or burns.

4.2 HAZARD COMMUNICATION

The purpose of hazard communication (Employee Right-to-Know) is to ensure that the hazards of all chemicals located at this field project site are transmitted (communicated) according to 29 CFR 1926.59 to all personnel and subcontractors. Hazard communication will include:

4.2.1 Container Labeling

Shaw personnel will ensure that all containers are labeled according to contents. These drums and containers will include those from manufacturers and those produced on site by operations, such as gasoline and diesel safety cans. All incoming and outgoing labels shall be checked for identity, hazard warning, and name and address of responsible party.

4.2.2 Material Safety Data Sheets (MSDS)

There will be an MSDS located on site for all site contaminants and each hazardous chemical known to be used on site. MSDS's are located in Appendix A of the Site-Specific Health Plan.

4.2.3 Employee Information and Training

Training employees on chemical hazards is accomplished through an ongoing corporate training program. All site employees shall maintain their HAZWOPER training, in accordance with 29 CFR 1910.120. Additionally, chemical hazards are communicated to employees through daily safety meetings held at Shaw field projects and by an initial site orientation program.

At a minimum, FSSI/Shaw and related subcontractor employees will be instructed on the following:

- An in-depth review of the soil and surface contaminants of concern identified and listed in Section 4.1.
- OSHA regulated chemicals and their hazards in the work area.
- How to prevent exposure to these hazardous chemicals.
- What the company has done to prevent workers' exposure to these chemicals.
- Procedures to follow if they are exposed to these chemicals.
- How to read and interpret labels and MSDS's for hazardous substances found on Shaw sites.
- Emergency spill procedures.
- Proper storage and labeling.

Before any new hazardous chemical is introduced on site, each FSSI/Shaw and related subcontractor employee will be given information in the same manner as during the safety class. The site Superintendent will be responsible for seeing that the MSDS on the new chemical is available for review by on site personnel. The information pertinent to the chemical hazards will be communicated to project personnel.

Morning safety meetings will be held and the hazardous materials used on site will be discussed. Attendance is mandatory for all on site employees.

4.3 PHYSICAL HAZARDS

To minimize physical hazards, Shaw has developed standard safety protocols that will be followed at all times. Activity Hazard Analyses, located in Appendix C, have been developed for each principal activity and identify all major hazards to which employees may be exposed.

The SS/SSO will observe the general work practices of each crewmember and equipment operator, and enforce safe procedures. The crew leaders and SS/SSO will inspect the work areas. All hazards will be corrected in a timely manner. A variety of physical hazards may be encountered during work activities at this site. Hard hats, safety glasses and steel-toe safety boots are required in all areas of the site. Site-specific hazards and all necessary precautions will be discussed at the daily safety meetings**.** Failure to follow safety protocols will result in removal of an employee from the site and appropriate disciplinary actions.

4.4 ENVIRONMENTAL HAZARDS

Environmental factors such as weather, wild animals, insects, and irritant plants may pose a hazard when performing outdoor tasks. The SS/SSO will take necessary actions to alleviate these hazards should they arise.

4.4.1 Heat Stress

The combination of warm ambient temperature and protective clothing increases the potential for heat stress. Heat stress disorders include:

- Heat rash
- Heat cramps
- Heat exhaustion
- Heat stroke.

Heat stress prevention is outlined in Shaw Health and Safety procedure HS400, Working in Hot Environments. This information will be reviewed during safety meetings. Workers are encouraged to increase consumption of water and electrolyte-containing beverages; e.g., Gatorade. Heat stress can be prevented by assuring an adequate work/rest schedule. Guidelines are presented below and should be used in conjunction with HS400.

In addition, workers are encouraged to take rests and report symptoms whenever they feel any adverse effects that may be heat-related. The frequency of breaks may need to be increased based on worker recommendation to the SS/SSO. Heat stress can be prevented by assuring an adequate work/rest schedule and adequate fluid consumption. A guide for work/rest schedules for various protection levels are given below in Table 4.2. The number of hours before a work/rest period is based on experience with similar work. The time periods should be considered maximum. It must also be remembered that individual physical variability's and differences in physical work activities may require revisions to site plans. This table should be used as a guide. Professional judgment of the SS/SSO is necessary to assure a fully protective plan to prevent heat stress disorders.

Table 4.2
Guidelines For Work-Rest Periods
Protection Level
Number Of Hours Before Rest Period

Temperature	Level D	Level C	Level B	Level A
90+ F*	2.0	1.5	1.0	0.5
87.5 F	2.5	2.0	1.5	1.0
82.5 F	3.0	2.5	2.0	1.5
77.5 F	3.5	3.0	2.5	1.5
72.5	4.0	3.5	2.5	1.5

**Work above 100º F will be reviewed with the Project HSM to determine specific requirements.*

Alternately the work/rest schedule can be calculated based on heat stress monitoring results. Each individual will count his/her radial (wrist) pulse as early as possible during each rest period. If the heart rate exceeds 75 percent of their calculated maximum heart rate (MHR = 200 – age) at the beginning of the rest period, then the work cycle will be decreased by one-third. The rest period will remain the same. An individual is not permitted to return to work until his/her sustained heart rate is below 75 percent of their calculated maximum heart rate.

Body temperature, measured orally or through the ear canal, may also be monitored to assess heat stress. Workers should not be permitted to continue work when their body temperature exceeds 100.4 ^{o}F (degrees Fahrenheit) or 38 ^{o}C (degrees ^{o}C Celsius). Monitoring should be conducted at the beginning of each break period as noted above.

Monitoring for heat stress will begin when the ambient temperature reaches or exceeds 70 ^{o}F when wearing chemical protective clothing (Level C, B, A), or 80 ^{o}F for site activities performed with no chemical protective clothing (Level D). Monitoring will include pulse rate, weight loss, oral/ or ear canal temperature, signs and symptoms of heat stress and fluid intake.

4.4.2 Noise

Hearing protection is required for workers operating or working near heavy equipment, where the noise level is greater than 85 A-weighted decimal (dBA) (Time Weighted Average [TWA]) as well as personnel working around heavy equipment. The HSM will determine the need and appropriate testing procedures (i.e., sound level meter and/or dosimeter) for noise measurement in accordance with Shaw Health and Safety procedure HS402 Hearing Conservation Program.

Noise monitoring should be conducted during the beginning of each activity, as well as, any time modifications lead to increased noise levels (e.g., adding additional equipment). A sound level meter will be used to measure noise levels at selected locations in the work area and on the site perimeter when treatment equipment is operating normally. When used, noise-monitoring equipment must be calibrated before and after each shift.

If continuous noise levels are found to exceed 85 dBA at any location within the work area, warning signs will be posted. Workers and visitors will be notified that hearing protection is required. Appropriate hearing protection (e.g., ear plugs) will be worn whenever personnel are working or visitors are present in that location. A supply of earplugs will be maintained on site.

Action levels in the following table will trigger the use of appropriate hearing protection (plugs or muffs). Hearing protection must be able to attenuate noise below 90 dBA (8-hour TWA). Each hearing protection or device has a Noise Reduction Rating (NRR) assigned by the EPA. The calculation for a hearing protection device's effectiveness is: noise reading dBA – (NRR – 7dB) < 90 dBA.

Instrument	Measurement	Action
Type I or Type II Sound Level Meter or dosimeter	>80 dBA → 85 dBA	Hearing protection recommended. Limit work duration to 8-hour shifts.
	>85 dBA → 90 dBA	Hearing protection required. Limit work duration to 8-hour shifts.
	>90 dBA → 115 dBA	Hearing protection required. Investigate use of engineering controls. Limit work duration to 8 hour shifts.
	>115 dBA	Stop work. Consult Project HSM

4.4.3 Biological Hazards

Tragia volubilis L.
Vine Nettle, Twining Cowitch, Creeping Cowitch

Pammel (1911) lists the species as being irritant. The plant bears stinging hairs (Hutchinson & Dalziel 1928, Thurston & Lersten 1969) which sting severely (Altschul 1973). Cook & Collins (1903) noted that contact with the plant causes instant stinging and burning sensations, and may result in blisters.

Stinging trichome of **Tragia volubilis**, a twining South American vine in the Euphorbiaceae. The stinging mechanism of **Tragia** is unusual because the calcium carbonate crystal is ejected from the trichome upon contact, where it pierces and enters the skin. The intial stinging sensation is mechanical; however, a secondary stinging sensation is caused by the crystal within the skin.

Poison Ivy (RHUS RADICANS). Poison Ivy may be found at the site. It is highly recommended that all personnel entering into an area with poison ivy wear a minimum of a Tyvek® coverall, to avoid skin contact.

The majority of skin reactions following contact with offending plants are allergic in nature and characterized by:

- General symptoms of headache and fever
- Itching
- Redness
- A rash.

Some of the most common and most severe allergic reactions result from contact with plants of the poison ivy group, including poison oak and poison sumac. Such plants produce severe rash characterized by redness, blisters, swelling, and intense burning and itching. The victim may develop a high fever and feel very ill. Ordinarily, the rash begins within a few hours after exposure, but may be delayed 24 to 48 hours.

A barrier cream (e.g., IvyBlock®) should be applied to the exposed skin before entering and working in areas with possible poisonous plants.

Distinguishing Features of Poison Ivy Group Plants. The most distinctive features of poison ivy and poison oak are their leaves, which are composed of three leaflets each. Both plants have greenish-white flowers and berries that grow in clusters.

First Aid

- Remove contaminated clothing; wash all exposed areas thoroughly with soap and water, followed by rubbing alcohol. A one percent hydrocortisone cream (over-the-counter) will aid in healing and reducing itch.
- Apply calamine or other soothing lotion if rash is mild.
- Seek medical advice if a severe reaction occurs, or if there is a known history of previous sensitivity.

Contaminated Clothing. The irritating substances emitted by poison ivy group plants will remain on clothing for prolonged periods of time - up to weeks or months, if not washed thoroughly. It may be necessary to wash contaminated clothing separately and more than once before reusing.

COMMON POISON IVY
(RHUS RADICANS)

- Grows as a small plant, a vine, and a shrub.
- Grows everywhere in the United States except California and parts of adjacent states. Eastern oak leaf poison ivy is one of its varieties.
- Leaves always consist of three glossy leaflets.
- Also known as three-leaf ivy, poison creeper, climbing sumac, poison oak, markweed, picry, and mercury.

WESTERN POISON OAK
(RHUS DIVERSILOBA)

- Grows in shrub and sometimes vine form.
- Grows in California and parts of adjacent states.
- Sometimes called poison ivy, or yeara.
- Leaves always consist of three leaflets.

POISON SUMAC
(RHUS VERNIX)

- Grows as a woody shrub or small tree from 5 to 25 feet tall.
- Grows in most of eastern third of United States.
- Also known as swamp sumac, poison elder, poison ash, poison dogwood, and thunderwood.

Ticks. Heavily vegetated areas of a site may have ticks. It is highly recommended that all personnel walking through such areas wear a minimum of a Tyvek® and latex boot covers. The ticks will stand out against the light colors. A tick repellent or insect containing DEET is also recommended.

If you have been bitten, place the tick in a jar labeled with the date, location of the bite, and the location acquired. If any symptom appears, such as an expanding red rash, contact a physician immediately.

First Aid

- Carefully (slowly and gently) remove the tick with tweezers, taking care that all parts are removed.
- With soap and water, thoroughly, but gently, scrub the area from which the tick has been removed, because disease germs may be present on the skin; also wipe the bite area with an antiseptic.

Dengue

Dengue fever and dengue hemorrhagic fever (DHF) are viral diseases transmitted by *Aedes* mosquitoes, usually *Ae. aegypti*. The four dengue viruses (DEN-1 through DEN-4) are immunologically related, but do not provide cross-protective immunity against each other. The principal vector mosquito, *Ae. aegypti*, is most frequently found in or near human habitations and prefers to feed on humans during the daytime. It has two peak periods of biting activity: in the morning for several hours after daybreak and in the late afternoon for several hours before dark. The mosquito may feed at any time during the day, however, especially indoors, in shady areas, or when it is overcast. Mosquito breeding sites include artificial water containers such as discarded tires, uncovered barrels, buckets, flower vases or pots, cans, and cisterns.

Estimates derived from studies of military and relief workers allow for an estimate of risk near one illness per thousand travelers. This estimate may overstate the danger for tourists who will have less contact with the vector, who stay only a few days in air-conditioned hotels with well-kept grounds, and who participate in outdoor recreational activities where the vector mosquito may be absent (such as sunbathing or playing golf in the middle of the day). Travelers who stay at other types of accommodations or with friends and relatives in locations with intense disease transmission may have a higher risk of illness. Cases of dengue are confirmed every year in travelers returning to the United States after visits to tropical and

subtropical areas. Travelers to endemic and epidemic areas, therefore, should take precautions to avoid mosquito bites.

Dengue fever is characterized by sudden onset after an incubation period of 3-14 days (most commonly 4-7 days), high fevers, severe frontal headache, and joint and muscle pain. Many patients have nausea, vomiting, and rash. The rash appears 3-5 days after onset of fever and can spread from the torso to the arms, legs, and face.

West Nile Virus and West Nile Encephalitis

West Nile Virus/West Nile Encephalitis is rapidly becoming a health concern in the United States. West Nile Virus was first identified in the U.S. in the New York area in 1999 and is closely related to the St. Louis Encephalitis Virus, which is routinely found in the United States.

"Encephalitis" means an inflammation of the brain and it can be caused by viral and bacterial infections. West Nile Encephalitis can be a serious or even fatal illness although this is rare in humans. This illness develops in approximately one of every 150 infections and is generally confined to older and physically compromised individuals.

West Nile Encephalitis is a viral infection of the brain transmitted through the bite of a mosquito, which has previously fed on birds and/or horses that were infected with West Nile Virus. Dead birds in an area may mean that West Nile Virus is circulating between the birds and the mosquitoes in that area. West Nile Virus is not transmitted from one person to another. Human illness from West Nile Virus is rare, even in areas where the virus has been reported.

Symptoms of Exposure Most people who become infected with West Nile Virus will have either no symptoms or only mild ones. Symptoms of West Nile Encephalitis include high fever, headache, confusion, muscle aches and weakness, seizures, or paralysis. At its most serious, the infection can result in coma, permanent neurological damage, and death. Symptoms usually occur five to 15 days following the bite of an infected mosquito. Because West Nile Encephalitis is a viral infection, antibiotics are not effective and there is no specific treatment available other than general support therapy.

Protective Measures at Projects There is no vaccine to protect humans against West Nile Virus. Individuals at project sites can reduce their risk from being infected with West Nile Virus by taking the following actions to protect against mosquito bites:

- Review the hazards of West Nile Virus periodically in morning safety meetings.
- Increase protective measures when working at dawn, dusk, and in the early evening.
- Reduce the area of exposed skin when working outdoors. Long-sleeved shirts with sleeves rolled down are recommended. Understand that mosquitoes may bite through thin clothing, so personnel should evaluate the actual Level D clothing worn, e.g., heavy long sleeve work shirts and heavy jeans may be indicated. Also, the risk or threat of mosquito bites is reduced for those activities that require the use of disposable coveralls.
- For activities where only Level D Personal Protective Equipment (PPE) is specified, consider using disposable coveralls when working in wooded, highly vegetated, or swampy areas.
- Use an insect repellent containing approximately 25% DEET (N, N-diethyl-meta-toluamide). DEET in concentrations greater than 25% provides no additional protection but repel insects longer. However, at some point there is no direct correlation between concentration and repellency. For example, 50% DEET provides about four hours of protection against mosquitoes, but increasing the concentration to 100% provides only one additional hour of protection. Use the repellent according to the manufacturer's directions provided on the container. Use just enough repellent to cover exposed skin and clothing. Do not treat unexposed skin. Frequent re-application is unnecessary for effectiveness. Avoid prolonged and excessive use of DEET.
- After returning from outdoor field activities, wash treated skin with soap and water.
- Personnel should report flu-like symptoms to the SSHO.

DEET is safe for pregnant and lactating women and is generally safe for children. You should avoid applying it to open wounds and irritated skin as it may further irritate the skin or cause discomfort.

Sweating, perspiration and getting wet may wash away the repellent and may require that DEET containing repellent be re-applied.

To remove the breeding places on a project, the following precautions will be followed as practical:

- Cut tall grass and weeds
- Drain accumulated water in such items as drums, buckets, pools and plastic containers
- Repair holes in door and window screens

- Eliminate stagnate water puddles as practical
- Limit outdoor activities at dawn, dusk and early evening, when mosquitoes are most active, as practical

Other Animal/Insect Hazards:
Potential exists to encounter mongoose, rats and mice which could potentially carry rabies. Individuals should avoid contact with any wild animals. In addition, feral dogs and cats have been observed and could also carry rabies. Those exhibiting abnormal behavior such agitation or viciousness or seem to be excessively lethargic or paralytic should be reported.

No poisonous snakes are indigenous to the island.

Fire ants, centipedes, bees, wasps, hornets and spiders are also present on the island. All of these insects can bite and may be venomous. Bites should be reported immediately. Individuals with known allergies should use extreme caution working in areas that may provide refuge for these insects. If bitten, allergic reactions could occur. Any adverse reaction or symptom requires medical attention.

Schistosomes (blood flukes) may be present in wet or swampy areas. Individuals working in these areas must utilize water proof clothing and avoid water contact, especially with open sores, cuts, or blisters. If contact occurs, the area must be washed with isopropyl alcohol. Symptoms of infection include diarrhea, itchy skin, fever and central nervous system damage.

4.4.4 Lightning

The procedures provided below will be used to protect site personnel from lightning related injuries.

Training. A tailgate safety meeting will be conducted to increase awareness to the hazards and prevention of lightning related incidents.

Detection of Lightning. The SS/SSO will be proactive in monitoring conditions that may produce thunderstorms and lightning. A daily and weekly weather forecast will be tracked and communicated to site personnel. When signs of impending storms, i.e., increasing wind, darkening skies, or lightning appear, local weather monitoring will be increased. The

National Weather Service (www.nws.noaa.gov/) should be consulted frequently. Personnel will be notified when thunderstorms may impact the site.

The "flash/bang" (f/b) technique of measuring the distance to lightning will be reviewed with all personnel. The f/b technique is defined as: for each five seconds from the time of observing the lightning flash to hearing the associated thunder, the lightning is one mile away.

Suspension/Resumption of Activities. All outside activities will be suspended when a lightning flash is immediately in the area or a f/b of 20 seconds (4 miles away) is noted. Personnel may continue indoor work activities. Outdoor activities will resume when 30 minutes has passed since the last observable f/b is 20 seconds or greater.

Lightning Protection. When notification is given, all outside work activities will stop and personnel will gather in the support zone for a head count and further instructions. Indoor work will continue, except for the use of electrical equipment, telephones and computers. When a safe location is not present and personnel are caught by a sudden lightning event, employees should seek the lowest possible area, away from large objects which might attract lightning or fall over, e.g., trees, utility poles. The employee should assume a crouching position with their head lowered and hands over their ears. AVOID: WATER, HIGH GROUNDS, HEAVY EQUIPMENT AND TALL, ISOLATED OBJECTS.

First Aid. An employee that is struck by lightning needs immediate assistance (call 911). The body will not carry an electrical charge, but receives an electrical shock and may be burned. Personnel certified in first-aid/CPR should inspect for shock and burns around fingers, toes, buckles and jewelry. Stay with the injured employee until medical help arrives.

4.5 VEHICLE AND HEAVY EQUIPMENT SAFETY MANAGEMENT

4.5.1 Vehicle Safety

Motor vehicle incidents are the number one cause of occupational fatalities, accounting for one in three deaths. Fifty percent or more of vehicle safety incidents occur while backing up. Shaw employees involved in the operation and use of Shaw and/or leased or rented vehicles will comply with the Shaw Health and Safety procedures HS800 Motor Vehicle Operation: General Requirements and HS810 Commercial Vehicle Operation and Maintenance. Shaw requires employees to use seat belts at all times when traveling in Shaw owned or leased/rented vehicles. The SS/SSO will develop a parking area plan, including backing

vehicles into parking spaces, using spotters for backing vehicles and policy mandated vehicle inspections.

Shaw employees are expected to incorporate safe actions and preparations to avoid vehicle accidents and personal injury during work and off-hours. Breaks should be planned into lengthy job mobilizations and demobilizations, including rotation of drivers at regular intervals. If parking areas are busy or crowded and more than one worker is traveling in the same vehicle, one worker should remain outside the vehicle as it leaves the parking space to assist the driver with traffic observation. Vehicles traveling before dawn and at dusk in rural or wooded areas should be prepared to brake for wildlife, e.g. deer crossing roadways.

Shaw employees arriving at work areas should park vehicles away from delivery, heavy equipment and vehicle loading/unloading locations to prevent parked vehicles from damage by various deliveries. Heavy equipment operators should inspect areas and request vehicles to be moved or spotters used if necessary, to maneuver equipment in tight areas. Employees who observe near misses or potential risks to parked or moving vehicles must report these to the SS/SSO immediately.

Shaw employees are expected to use the vehicle inspection form and check/test the safety systems on the vehicle on a daily basis. Check the following: brakes, mirrors, seat belts, tires, leakage from the undercarriage, lights and turn signals. Vehicles with safety deficiencies must be reported immediately and not driven until properly repaired. Vehicles running errands from different project sites should have telephone numbers of the job site in the vehicle in case calls for assistance are required.

Because of the different ways alcohol can affect behavior, even in very small amounts, the best and safest course is not to drink before driving. At Shaw, a driver with blood alcohol concentration (BAC) over 0.04 percent is considered to be under the influence and subject to disciplinary action. Personnel involved in motor vehicle incidents are subject to drug and alcohol testing.

Weather conditions can have a profound effect on driving. On slippery roads, drive more slowly. Stop and turn with care. Keep several car lengths from other vehicles. At speeds in excess of 35 mph, the chances of hydroplaning increase with speed. In general, keep back 1 car length for every 10 miles per hour (mph) to prevent striking the car ahead.

Vehicles will be operated in accordance with the requirements listed below:

- Seatbelt use is mandatory for all passengers
- Personnel may not ride in the back of cargo vehicles
- The driver must make a 360 degree walk around the assigned vehicle prior to vehicle movement
- A ground guide is used to back up any vehicle
- Vehicle speed is limited to the posted speed limits for developed roadways, 25 mph maximum on dirt roads and 10 mph maximum off-road (based on conditions)
- Vehicle driven in four wheel low and low gear when on dirt roads or off road driving where steep grades dictate
- All operators must possess a valid drivers license
- Fuel or gasoline are not to be transported inside the passenger compartment
- No vehicle is left running when unattended
- Parking brakes are used when vehicles are parked.

In the event of a vehicle incident, notify your Project Manager ***immediately*** and complete all required reports.

4.5.2 Heavy Equipment Safety

Forklifts, excavators, loaders other material handling equipment present various physical hazards on remediation sites. The following critical safety practices shall be followed to prevent safety incidents during heavy equipment operation.

- All equipment will be inspected prior to each use.
- All operators will have training or equivalent experience to be permitted to operate heavy equipment.
- Spotters will be used to back-up equipment and direct traffic in all “blind” areas.
- Standard hand signals will be used to communicate between operators and ground crew.
- All heavy equipment will have operable back-up alarms.

- Heavy equipment will be parked in areas where operators will not be exposed to strains or slip/trip/fall hazards during mounting and dismounting of equipment
- All heavy equipment will be equipped with operable seat belts; belts will be used by all operators.
- Written lifting plans will be developed and reviewed for all critical lifts.

4.6 MANUAL MATERIAL LIFTING

Many different types of objects may be handled manually during site operations. Care should be taken when lifting and handling heavy or bulky items because they are the cause of many back injuries. The following fundamentals address the proper lifting techniques that are essential in preventing back injuries:

- The size, shape, and weight of the object to be lifted must first be considered. No individual employee is permitted to lift any object that weighs more than 60 pounds. Multiple employees or the use of mechanical lifting devices are required for objects over the 60-pound limit.
- The anticipated path to be taken by the lifter should be inspected for the presence of slip, trip, and fall hazards.
- The feet shall be placed far enough apart for good balance and stability (typically shoulder width). THE FOOTING SHALL BE SOLID.
- The worker shall get as close to the load as possible. The legs shall be bent at the knees.
- The back shall be kept as straight as possible and abdominal muscles should be tightened.
- To lift the object, the legs are straightened from their bending position.
- A worker shall never carry a load that cannot be seen over or around.
- When placing an object down, the stance and position are identical to that for lifting. The legs are bent at the knees and the object lowered.

When two or more workers are required to handle the same object, coordination is essential to ensure that the load is lifted uniformly and that the weight is equally divided between the individuals carrying the load. When carrying the object, each worker, if possible, shall face the direction in which the object is being carried. In handling bulky or heavy items, the following guidelines shall be followed to avoid injury to the hands and fingers:

- A firm grip on the object is essential; leather gloves shall be used if necessary.
- The hands and object shall be free of oil, grease, and water that might prevent a firm grip, and the fingers shall be kept away from any points that could cause them to be pinched or crushed, especially when setting the object down.
- The item shall be inspected for metal slivers, jagged edges, burrs, and rough or slippery surfaces prior to being lifted.

4.7 UNEXPLODED ORDNANCE

The potential exists for unexploded ordnance (UXO) to be encountered. This is of particular concern during intrusive activities excavation activities. A UXO Technician will be on-site during all intrusive activities. A separate UXO plan is included in the main Scope of Work. All site personnel will be required to attend UXO Awareness training.

The basic guidelines for UXO safety are listed below:

- Do not continue to move towards a suspected UXO
- Once you recognize a UXO hazard, do not move any closer
- Stop all work
- Make all radio transmissions at least 100 meters away from a UXO hazard
- Do not try to remove anything that is on or near a UXO
- Do not move or disturb a UXO
- Stay away from UXOs
- Mark a UXO hazard area properly so that other personnel will stay away from it
- Evacuate all nonessential personnel from a UXO hazard area
- Report through your chain of command all UXO hazards that affect operations.

4.8 ACTIVITY HAZARD ANALYSES

Appendix C contains Activity Hazard Analyses (AHA) for primary site tasks. They contain detailed information on physical and chemical hazards, and provide control measures for these hazards. The AHA's will be field checked by the SS/SSO on an ongoing basis and revised as necessary. All revisions will be communicated to the work crew. In addition to the AHAs, a Job Safety Analysis (JSA) must be completed daily in accordance with Shaw Health and Safety Procedure HS045. (See Appendix B)

5.0 WORK AND SUPPORT AREAS

To prevent migration of contamination from personnel and equipment, work areas will be clearly specified as designated below prior to beginning operations. Each work area will be clearly identified using signs or physical barriers.

5.1 SUPPORT ZONE (SZ)

The uncontaminated SZ, or clean zone, will be located upwind, in an area outside the EZ and CRZ and within the geographic perimeters of the site. The area is used for material staging, vehicle parking, office facilities, sanitation facilities, and receipt of deliveries. Personnel entering this zone may include delivery personnel, visitors, security guards, etc., who will not necessarily be permitted in the EZ. All personnel arriving in the SZ will, upon arrival, report to the HSM and sign the site visitor log.

5.2 CONTAMINATION REDUCTION ZONE (CRZ)

Personnel and equipment decontamination will be performed in the CRZ that is adjacent to the EZ. All personnel entering or leaving the EZ will pass through this area to prevent any cross-contamination and for the purpose of accountability. Personal protective outer garments and respiratory protection will be removed in the CRZ and properly labeled. All water generated from equipment and personal decontamination will be contained on site and disposed of in an appropriate manner.

5.3 EXCLUSION ZONE (EZ)

The EZ will be the area around the excavation, segregation, sampling and load-out activities. These areas have the highest potential for exposure to contamination by contact, ingestion, or inhalation. All employees will use proper PPE when working in these areas. The location of the EZ will be identified by fencing or other appropriate means primarily around the excavation areas and the stockpiles. A daily entry log records the time of entry and exit from the EZ for each person.

A log of all personnel visiting, entering or working on the site shall be maintained by the SS/SSO. Visitors will attend a site orientation given by the SS/SSO and sign the SSHSP.

The following are standard safe work practices that apply to all site personnel and will be discussed in the safety briefing prior to initiating work on the site:

- Eating, drinking, chewing gum or tobacco, smoking is prohibited in the EZ/CRZ's.
- Hands and face must be washed upon leaving the EZ and before eating, drinking, chewing gum or tobacco and smoking.
- A buddy system will be used. Hand signals will be established to maintain communication.
- During site operations, each worker will consider himself as a safety backup to his partner. Off-site personnel provide emergency assistance.
- Visual contact will be maintained between buddies on site when performing hazardous duties.
- All personnel must comply with established safety procedures. Any staff member who does not comply with safety policy, as established by the SS/SSO, will be immediately dismissed from the site.
- Proper decontamination procedures must be followed before leaving the site.
- All employees and visitors must sign in and out of the site.

6.0 PROTECTIVE EQUIPMENT

This section specifies the levels of personal protective equipment (PPE), which is required for each principal activity performed at this site. All site personnel must be trained in the use of all PPE utilized. The PPE procedures outlined in Shaw Health and Safety procedures HS600 Personal Protective Equipment and HS601 Respiratory Protection Program will be applied to project activities.

6.1 ANTICIPATED PROTECTION LEVELS

The following protection levels have been established for the site work activities.

Table 6.1
Anticipated Protection Levels

Task	Initial PPE Level	Upgrade PPE Level	Skin Protection	Respiratory Protection	Other PPE
All site activities, (except for soil Sampling) providing there is not physical contact with landfill materials and no air monitoring levels are detected above the action levels noted in Section 8.1	Level D	Modified Level D	Leather-work gloves. Tyvek® coverall as necessary to protect against biological hazards	None	Hard-hat, steel-toe work boots, safety glasses and hearing protection >85 dBA. Goggles/face shield when in contact with liquid contamination or flying debris.
Soil Sampling and any work activities in direct contact with the landfill materials (air monitoring readings are below the action levels)	Modified Level D	Level C (Elevated PID Readings) Elevated H2S Readings: Stop Work and contact HSM for upgrade	Tyvek® coverall, inner latex sample gloves, outer nitrile gloves and latex boot covers	Level C: Initial: Full-face air purifying respirator	Hard-hat, steel-toe work boots, safety glasses and hearing protection >85 dBA. Goggles/face shield when in there is a potential for splash hazards
General Support Zone Activities	Level D	Not Anticipated	None	None	Hard-hat, steel-toe boots, safety glasses and hearing protection >85dBA.

***Note**- Level C cannot be used with elevated levels of hydrogen sulfide therefore if hydrogen sulfide is encountered, work will stop and the HSM will determine the need and type of additional PPE.

6.2 PROTECTION LEVELS DESCRIPTIONS

This section lists the minimum requirements for each protection level. Modification to these requirements may have been noted above.

6.2.1 Level D

Level D consists of the following:

- Safety glasses with side shields
- Hard hat
- Steel-toed work boots
- Work clothing as prescribed by weather
- Leather work gloves
- Cut resistant gloves when handling sharp objects
- Reflective vests for ground personnel working around heavy equipment or roadways
- Hearing protection in areas >85 dBA
- Cut resistant gloves, as needed

6.2.2 Modified Level D

Modified Level D consists of the following:

- Safety glasses with side shields
- Hard hat
- Steel-toed work boots
- Tyvek® coverall (when handling dry materials)
- Poly-coated Tyvek® coverall or PVC rain suit (when handling wet materials)
- Latex over-boots
- Inner latex sample gloves
- Outer nitrile gloves
- Hearing protection in areas >85 dBA
- Full-face shield when splash hazards are present
- Metatarsal and shin guard for pressure sprayer operations
- Cut resistant gloves, as needed

6.2.3 Level C

Level C consists of the following:

- Full-face, air-purifying respirator
- Hooded Tyvek Coveralls
- Hard hat
- Safety-toed work boots
- Vinyl under booties
- Neoprene over boots
- Nitrile outer gloves
- Face shield (when projectiles or splashes pose a hazard)
- Hearing protection >85 dBA

6.3 RESPIRATORY PROTECTION

6.3.1 Air-Purifying Respirators and Cartridge Selection

A Survivair 20/20 full-face respirator with 1053 cartridges will be used for Level C work.

6.3.2 Fit Testing

Annual respirator fit tests are required of all personnel wearing negative-pressure respirators. Quantitative fit-testing is required.

6.3.3 Inspection and Cleaning

Respirators shall be checked periodically by a qualified individual and inspected before each use by the wearer. All respirators and associated equipment will be decontaminated and hygienically cleaned after each use.

6.3.4 Fit Testing

Annual respirator fit tests are required of all personnel wearing negative-pressure respirators. The test will use isoamyl acetate or irritant smoke. The fit test must be for the style and size of the respirator to be used. Quantitative fit-testing is required for use of respirators in chemical environments where the respirator effective use limit exceeds 10 (exposure of 1 ppm inside the respirator for 10 ppm outside the respirator). Therefore, quantitative fit-testing is dependent on the PEL/TLV of the chemical substance involved. Quantitative fit-

testing is required for potential exposure to airborne particulate levels that exceed 10 times the established PEL/TLV.

6.3.5 Facial Hair

No personnel who have facial hair which interferes with the respirator's sealing surface will be permitted to wear a respirator and will not be permitted to work in areas requiring respirator use.

6.3.6 Corrective Lenses

Normal eyeglasses cannot be worn under full-face respirators because the temple bars interfere with the respirator's sealing surfaces. For workers requiring corrective lenses, special spectacles designed for use with respirators will be provided. Contact lenses are permitted to be used with full-face respirators based on a decision by OSHA.

6.3.7 Medical Certification

Only workers who have been certified by a physician, as being physically capable of respirator usage will be issued a respirator. Personnel unable to pass a respiratory fit test or without medical clearance for respirator use will not be permitted to enter or work in areas on site that require respiratory protection. Employees will receive a written physicians opinion that they are fit for general hazardous waste operations as per 29 CFR 1910.120(f)(7).

6.4 SITE- SPECIFIC PERSONAL PROTECTIVE EQUIPMENT (PPE)

The primary objective of the PPE program is to ensure employee protection and to prevent employee exposure to site contaminants during site operations. Engineering controls are not feasible for many tasks and, therefore, require the use of PPE.

The SS/SSO will be responsible for monitoring all aspects of the PPE program. This includes donning and doffing, temperature related stress monitoring, inspection, and decontamination. PPE selection is identified in Table 6.1 for each specified task. The SS/SSO, in consultation with the HSM will direct changes in PPE based on changing conditions. The site-specific SSHSP will serve as written certification that the workplace was evaluated concerning PPE requirements.

7.0 DECONTAMINATION PROCEDURES

This section describes the procedures necessary to ensure that both personnel and equipment are free from contamination when they leave the work site.

7.1 PERSONNEL DECONTAMINATION

Decontamination procedures will ensure that material which workers may have contacted in the EZ do not result in personal exposure and is not spread to clean areas of the site. This sequence describes the general decontamination procedure. The specific stages will vary depending on the site, the task, the protection level, etc.

7.1.1 Modified Level D Decontamination

1. Go to end of EZ
2. Remove and discard latex booties
3. Remove outer gloves and discard
4. Remove protective suit
5. Remove inner sample gloves and discard
6. Wash face and hands.

7.1.2 Level C Decontamination

1. Go to end of EZ
2. Remove and discard latex booties
3. Remove outer gloves and discard
4. Remove outer suit (Poly-coated Tyvek®, Tyvek® or PVC rain suit)
5. Cross into CRZ (dirty side of respirator wash area)
6. Remove and wash respirator (4 stages)
   a. Soap and water solution
   b. First rinse
   c. Disinfect respirator (1 cap full of bleach to 1 gallon of water)
   d. Final rinse
   e. Hang respirator to dry
9. Remove inner sample gloves and discard
10. Wash face and hands.

7.1.3 Suspected Contamination

Any employee suspected of sustaining skin contact with chemical materials will first use the emergency shower. Following a thorough drenching, the worker will proceed to the decontamination facility. Here the worker will remove clothing, shower, don clean clothing, and immediately be taken to the first-aid station. Medical attention will be provided based on the degree of injury.

7.1.4 Personal Hygiene

Before any eating, smoking, or drinking, personnel will wash hands, arms, neck and face.

7.2 EQUIPMENT DECONTAMINATION

All contaminated equipment will be decontaminated before leaving the site. Decontamination procedures will vary depending upon the contaminant involved, but may include sweeping, wiping, scraping, hosing, or steaming the exterior of the equipment. Personnel performing this task will wear the proper PPE as prescribed by the SS/SSO.

7.3 DISPOSAL

All decontamination liquids and disposable clothing will be treated as contaminated waste unless determined otherwise by accepted testing methods. Wastes will be disposed of according to state and federal regulations.

7.4 SUSPECTED CONTAMINATION

Any employee suspected of sustaining skin contact with chemical materials will remove clothing, shower, don clean clothing, and immediately be taken to the first-aid station. Medical attention will be provided based on the degree of injury.

8.0 AIR MONITORING

Air monitoring will be conducted in order to characterize personnel exposures and fugitive emissions from site contaminants. The principal contaminant of concern is lead.

8.1 WORK AREA AIR MONITORING

Work area air monitoring at the site will be by direct reading methods. A personal Data RAM will be used to monitor for airborne particulate during grubbing in contaminated areas, soil excavation and material segregation activities. A hydrogen sulfide meter will be used during excavation and material segregation activities. A LEL/O_2 meter will be on-site for use by the SSO if necessary. A PID will be available on-site to monitor for any potential volatile organic materials. Air monitoring results will be used to determine the effectiveness and/or need for dust control methods and to trigger action levels as specified in Table 8-1.

Table 8.1
Direct Reading Air Monitoring Requirements

Monitoring Device	Monitoring Location/ Personnel	Monitoring Frequency	Action Level	Action
Data RAM	EZ During grubbing in contaminated areas, excavation and material loadout activities/ Equipment Operators, Recovery Technicians and Sample Technicians	Initially and then periodically at the discretion of the HSM	<.05 mg/m^{3*}	Modified Level D & dust control
			≥.05 – 0.5 mg/m^{3*}	Evaluate dust control measures. Contact the Health and Safety Manager. Upgrade to Level C PPE
			>0.5 mg/m^3	Stop work and re-evaluate. Contact Health and Safety Coordinator
PID	EZ During grubbing in contaminated areas, excavation and material loadout activities/ Equipment Operators, Recovery Technicians and Sample Technicians	Initially and then periodically at the discretion of the HSM	<5 ppm*	Modified Level D
			5 - 50 ppm*	Level C. Attempt to identify the source of the readings. Contact the HSM or CIH
			>50 ppm*	Stop work. Contact the HSM or CIH.

Monitoring Device	Monitoring Location/ Personnel	Monitoring Frequency	Action Level	Action
Hydrogen Sulfide meter	EZ During grubbing in contaminated areas, excavation, material loadout and sampling activities/ Equipment Operators, Recovery Technicians and Sample Technicians	Continuous	<5.0 ppm ≥ 5.0 ppm	Level D+ Stop work, contact HSM
LEL/O_2	EZ As determined necessary by the SSO	Initially and then as suspect material is are discovered	>10% LEL <20.8% O_2 or 23.5% O2	Evacuate area

*Sustained levels above background for 5 minutes in the breathing zone.

8.2 INSTRUMENTATION

The following is a description of the air monitoring equipment to be used at this site.

8.2.1 Time Aerosol Monitor (Model Pr1000) Data RAM)

8.2.1.1 Principle of Operation

Detection of light in the near infrared region back-scattered to a sensor (photovoltaic detector) by airborne particulate in a sensing volume

Device calibrated at the factory against an air sampling filter/gravimetric analysis reference method

8.2.1.2 Calibration Methods/Frequencies

There is no calibration method or procedure for calibrating the mini-ram monitor. However, it is recommended that the Data RAM monitor be re-zeroed once daily.

8.2.1.3 Preventative Maintenance

Maintenance of the Data RAM consists of replacement battery replacement and cleaning of the optical detection assembly.

8.2.2 Lower Explosive Limit/Oxygen (LEL/O_2) Meter

8.2.2.1 Types and Operational Aspects

MSA Watchman LEL/O2 Meter or equivalent

8.2.2.2 Principle of Operation

- Oxygen detector uses an electrochemical sensor; produces a minute electric current proportional to the oxygen content.

- Combustible gas indicators use a combustion chamber containing a filament that ignites flammable vapors; filament is heated or coated with a catalyst (platinum) to facilitate combustion.

- Filament is part of a balanced resistor circuit; combustion in the chamber causes the filament temperature to increase; results in increased filament resistance.

- Change in the filament's resistance causes an imbalance in the circuit proportional to the percent of the lower explosive limit (% LEL).

- Concentrations greater than the LEL and lower than the upper explosive limit (UEL) will read 100% LEL; combustible atmosphere present.

- Concentrations greater than the UEL will read above 100% LEL then return to zero. (NOTE: Some devices have catchment mechanisms which will cause the needle to remain at 100% until the meter is reset.) This type of response indicates the gas mixture is too rich to burn and is not combustible. The danger is that the addition of air to the gas mixture could bring it into the flammable range (less than the UEL).

- Oxygen meter set at the factory to alarm at 19.5% (oxygen deficient atmosphere) combustible gas meter set by the user to alarm at 10% LEL.

8.2.2.3 Calibration Methods/Frequencies

Before the calibration of the combustible gas indicator can be checked, the unit must be in operating condition. The combustible gas indicator (LEL) is normally calibrated on pentane as being representative of the flammability characteristics of most commonly encountered combustible gases. The meter scale is calibrated from zero to 100% LEL, which corresponds

in actual volume concentrations of 0 to approximately 14% pentane in air. A booklet of response curves is supplied with the Watchman Meter. These curves may be used to interpret meter readings when sampling combustible gases other than pentane.

It is recommended that calibration be checked before and after using each time. The PSO will record and log such calibration information into an air monitoring notebook. The O_2 meter is calibrated by adjusting the O_2 control knob to 20.8% while the meter is operated in a fresh air atmosphere.

8.2.2.4 Preventative Maintenance

The primary maintenance of unit is the rechargeable 2.4 volt nickel cadmium battery. Recommended charging time is 16 hours. It may be left on charge for longer periods without damaging the battery. The battery sometimes will not supply full power capacity after repeated partial use between charging. Therefore, it is recommended that the battery be exercised at least once a month by running for eight to 10 hours and recharged. If the instrument has not been used for 30 days, the battery should be charged prior to use.

8.2.3 Photoionization Detector (PID)

8.2.3.1 Type and Operational Aspects

PID Model Photovac 2020

- Principle of Operation

  Ionization potential (IP) - The energy required to remove the outermost electron from a molecule; measured in electron volts (eV); characteristic property of a specific chemical.

  Photoionization - Using ultraviolet (UV) light to remove the outermost electron from a molecule.

  Energy of UV light (10.2, 9.5, 11.7 eV) must be equal to or greater than the IP to photoionize the molecule.

  Fan or pump is used to draw air into the detector where the contaminants are exposed to a UV light source (lamp).

Ions are collected on a charged plate and produce a current directly proportional to the number of ionized molecules; current is amplified and displayed on the meter.

8.2.3.2 Calibration Method/Frequencies

The Photovac 2020 is designed for trace gas analysis in ambient air and is calibrated with certified standards of benzene, vinyl chloride, and isobutylene. Other optional calibrations are available (e.g., ammonia, ethylene oxide, H_2S, etc.).

Shaw will use a PID with a 10.6 eV lamp. This lamp is responsive to a wide range of contaminants, has a longer service life and is less affected by humidity than the other lamps. Optional lamps of 10.2 and 11.7 eV will be used when a specific contaminant is identified which is more responsive to that particular lamp.

The approximate span settings for the probe that would give different readings of the amounts of trace gas of a particular species in a sample are based upon the relative photoionization sensitivities of various gases twice daily (beginning and end of shift).

It is recommended that calibration be checked twice each day (beginning and end of shift). The SSO will record and log such calibration information into an air monitoring notebook.

8.2.3.3 Preventative Maintenance

Maintenance of the Photovac consists of cleaning the lamp and ion chamber, and replacement of the lamp or other component parts or sub-assemblies.

8.2.4 HYDROGEN SULFIDE MONITOR

Hydrogen sulfide monitors are required to measure personnel breathing zones when site personnel are potentially exposed to H_2S during site remedial operations. An action level of 5 ppm for 5 minutes requires that work be stopped immediately and the HSM be contacted for further direction.

8.3 AIR MONITORING LOG

The HSM will ensure that all air-monitoring data is logged into a notebook. Data will include instrument used, wind direction, work process, etc. The Program CIH will periodically review this data.

8.4 CALIBRATION REQUIRMENTS

The Hydrogen Sulfide monitor, PID and LEL/O_2 will be calibrated daily before use and the Data RAM will be zeroed before use. A separate log will be kept by the HSM detailing date, time, span gas, or other standard, and name of person performing the calibration.

8.5 AIR MONITORING RESULTS

Air monitoring results will be available for personnel inspection, and will be discussed during morning safety meetings. Personal air sampling results will be forwarded to the Corporate Health and Safety Manager for Medical Surveillance, to be incorporated into the employee(s)' medical records.

9.0 EMERGENCY RESPONSE

9.1 PRE-EMERGENCY PLANNING

Prior to engaging in remediation activities at the site, Shaw will plan for possible emergency situations and have available adequate supplies and manpower to respond. In addition, site personnel will receive training during the site orientation concerning proper emergency response procedures.

The following situations would warrant implementation of the Emergency Response and Contingency Plan (ERCP):

Fire/Explosion	• The potential for human injury exists. • Toxic fumes or vapors are released. • The fire could spread on-site or off-site and possibly ignite other flammable materials or cause heat-induced explosions. • The use of water and/or chemical fire suppressants could result in contaminated run-off.
Natural Disaster	• A rainstorm exceeds the flash flood level. • The facility is in a projected tornado path or a tornado has damaged facility property. • Severe wind gusts are forecasted or have occurred and have caused damage to the facility. • Hurricanes
Medical Emergency	• Overexposure to hazardous materials. • Trauma injuries (broken bones, severe lacerations/bleeding, burns). • Eye/skin contact with hazardous materials. • Loss of consciousness. • Heat stress (Heat stroke). • Heart attack. • Respiratory failure. • Allergic reaction.

The following measures will be taken to assure the availability of adequate equipment and manpower resources:

- Sufficient equipment and materials will be kept on site and dedicated for emergencies only. The inventory will be replenished after each use.
- On-site emergency responders will be current in regards to training and medical surveillance programs. Copies of all applicable certificates will be kept on file for on-site personnel required to respond.

- It will be the responsibility of the Site Superintendent to brief the on-site response team on anticipated hazards at the site. The Emergency Coordinator shall also be responsible for anticipating and requesting equipment that will be needed for response activities.
- Emergency response activities will be coordinated with the Local Emergency Management Agency (EMA) in compliance with SARA Title III requirements.

Communications will be established prior to commencement of any activities at the remediation site. Communication will be established so that all responders on site have availability to all pertinent information to allow them to conduct their activities in a safe and healthful manner. The primary communication device will be air horns.

9.2 EMERGENCY RECOGNITION AND PREVENTION

Because unrecognized hazards may result in emergency incidents, it will be the responsibility of the Site Superintendent and the Site Safety Officer, through daily site inspections and employee feedback (Safety Observation Program, daily safety meetings, daily JSAs and AHAs) to recognize and identify all hazards that are found at the site. These may include:

Chemical Hazards	• Materials at the site • Materials brought to the site
Physical Hazards	• Fire/explosion • Slip/trip/fall • Excessive noise
Mechanical Hazards	• Pinch points • Vehicle traffic
Environmental Hazards	• Electrical Storms • High winds • Heavy Rain/Snow • Temperature Extremes (Heat/Cold Stress) • Poisonous Plants/Animals

Once a hazard has been recognized, the SS/SSO and the HSM will take immediate action to prevent the hazard from becoming an emergency. This may be accomplished by the following:

- Daily safety meeting
- Task-specific training prior to commencement of activity
- PPE selection/use
- Following all Shaw standard operating procedures

Table 9.1
Emergency Telephone Numbers

Local Agencies – Ambulance Fire Police	 787-741-2111 787-741-2111 787-741-2020
Hospital Susanna Centro Hospital **Directions to the hospital are to be inserted into Appendix D by the SS/SSO**	 787-741-3282
Regional Poison Control Center	800-552-6337
Federal Agencies Agency for Toxic Substances and Disease Registry National Response Center	 (404) 639-0615 (24 hr.) 800-424-8802
US Navy Environmental Representative Madeline Rivera	 787-865-4152
Shaw Personnel Project Manager – Bill Hughes Site Superintendent/Site Safety Officer – Cano Hernandez Program CIH – David Mummert, CIH Program Health & Safety Manger – Kym Edelman	 (757) 318-5140 (office) (757)544-7687 (cellular) (419) 425-6129 (office) (419) 348-1544 (cellular) 757-435-5384 (cellular)
Shaw Corporation (24 hour)	800-537-9540

9.3 PERSONNEL ROLES, LINES OF AUTHORITY, AND COMMUNICATIONS

This section of the ERCP describes the various roles, responsibilities, and communication procedures that will be followed by personnel involved in emergency responses.

The primary Emergency Coordinator for this site is the Site Superintendent. In the event an emergency occurs and the Emergency Coordinator is not on site, the SS/SSO or the highest-ranking employee on site will serve as the Emergency Coordinator until he arrives. The Emergency Coordinator will determine the nature of the emergency and take appropriate action as defined by this ERCP.

The Emergency Coordinator will implement the ERCP immediately as required. The decision to implement the plan will depend upon whether the actual incident threatens human

health or the environment. Immediately after being notified of an emergency incident, the Emergency Coordinator or his designee will evaluate the situation to determine the appropriate action.

9.3.1 Responsibilities and Duties

This section describes the responsibilities and duties assigned to the Emergency Coordinator.

It is recognized that the structure of the "Incident Command System" will change as additional response organizations are added. Shaw will follow procedures as directed by the Fire Department, LEPC, State and Federal agencies as required. Shaw will defer to the local Fire Department Chief to assume the role of Incident Commander upon arriving on site. Additional on-site personnel may be added to the Site Emergency Response Team as required to respond effectively.

9.3.2 On-Site Emergency Coordinator Duties

The on-site Emergency Coordinator is responsible for implementing and directing the emergency procedures. All emergency personnel and their communications will be coordinated through the Emergency Coordinator. Specific duties are as follows:

- Identify the source and character of the incident, type and quantity of any release. Assess possible hazards to human health or the environment that may result directly from the problem or its control.

- Discontinue operations in the vicinity of the incident if necessary to ensure that fires, explosions, or spills do not recur or spread to other parts of the site.

- Notify the ROICC. The ROICC will contact the local Emergency Response Teams if their help is necessary to control the incident. Table 9.1 provides telephone numbers for emergency assistance.

- Direct on-site personnel to control the incident until, if necessary, outside help arrives.

- Ensure that the building or area where the incident occurred and the surrounding area are evacuated, and shut off possible ignition sources, if appropriate. The Emergency Response Team is responsible for directing site personnel such that they avoid the area of the incident and leave emergency control procedures unobstructed.

- If fire or explosion is involved, notify facility Fire Department.
- Notify Shaw Project Manager
- Notify US Navy Environmental Representative
- Have protected personnel, in appropriate PPE, on standby for rescue.

If the incident may threaten human health or the environment outside of the site, the Emergency Coordinator should immediately determine whether evacuation of area outside of the site maybe necessary and, if so, notify the US Navy Environmental Representative (USNER). The USNER will contact the local Police Department and the Office of Emergency Management.

If hazardous waste has been released or produced through control of the incident, ensure that:

- Waste is collected and contained.
- Containers of waste are removed or isolated from the immediate site of the emergency.
- Treatment or storage of the recovered waste, contaminated soil or surface water, or any other material that results from the incident or its control is provided.
- Ensure that no waste that is incompatible with released material is treated or stored in the facility until cleanup procedures are completed.
- Ensure that all emergency equipment used is decontaminated, recharged, and fit for its intended use before operations are resumed.

9.3.3 Safe Distances and Places of Refuge

The Emergency Coordinator for all activities will be the SS/SSO. No single recommendation can be made for evacuation or safe distances because of the wide variety of emergencies that could occur. Safe distances can only be determined at the time of an emergency based on a combination of site and incident-specific criteria. However, the following measures are established to serve as general guidelines.

In the event of minor hazardous materials releases (small spills of low toxicity), workers in the affected area will report initially to the contamination reduction zone. Small spills or leaks (generally less than 55 gallons) will require initial evacuation of at least 50 feet in all directions to allow for cleanup and to prevent exposure. After initial assessment of the extent of the release and potential hazards, the Emergency Coordinator or his designee will determine the specific boundaries for evacuation. Appropriate steps such as caution tape, rope, traffic cones, barricades, or personal monitors will be used to secure the boundaries.

If an incident may threaten the health or safety of the surrounding community, the public will be informed and, if necessary, evacuated from the area. The Emergency Coordinator, or his designee, will inform the proper agencies in the event that this is necessary. Telephone numbers are listed in Table 9.1.

Places of refuge will be established prior to the commencement of activities. These areas must be identified for the following incidents:

- Chemical release
- Fire/explosion
- Medical emergency
- Hazardous weather.

In general, evacuation will be made to the main entrance to the Shaw site, unless the Emergency Coordinator determines otherwise. It is the responsibility of the Emergency Coordinator to determine when it is necessary to evacuate personnel to off-site locations.

In the event of an emergency evacuation, all the employees will gather at the entrance to the site until a head count establishes that all are present and accounted for. No one is to leave the site without notifying the Emergency Coordinator.

9.3.4 Evacuation Routes and Procedures

All emergencies require prompt and deliberate action. In the event of an emergency, it will be necessary to follow an established set of procedures. Such established procedures will be followed as closely as possible. However, in specific emergency situations, the Emergency Coordinator may deviate from the procedures to provide a more effective plan for bringing the situation under control. The Emergency Coordinator is responsible for determining which situations require site evacuation.

9.3.5 Evacuation Signals and Routes

Two-way radio communication and an air horn will be used to notify employees of the necessity to evacuate an area or building involved in a release/spill of a hazardous material. The crew Superintendent will have a two-way radio. Only the Emergency Coordinator will initiate total site evacuation, however, in his absence, decision to preserve the health and safety of employees will take precedence.

9.3.6 Evacuation Procedures

In the event evacuation is necessary, the following actions will be taken:

- The emergency signal will be activated.
- No further entry of visitors, contractors, or trucks will be permitted. Vehicle traffic within the site will cease in order to allow safe exit of personnel and movement of emergency equipment.
- Shut off all machinery if safe to do so.
- ALL on-site personnel, visitors, and contractors in the support zone will assemble at the entrance to the site for a head count and await further instruction from the Emergency Coordinator.
- ALL persons in the exclusion zone and contamination reduction zone will be accounted for by their immediate crew leaders (e.g., foreman). Leaders will determine the safest exits for employees and will also choose an alternate exit if the first choice is inaccessible.
- During exit, the crew leader should try to keep the group together. Immediately upon exit, the crew leader will account for all employees in his crew.
- Upon completion of the head count, the crew leader will provide the information to the Emergency Coordinator.
- Contract personnel and visitors will also be accounted for.
- The names of emergency response team members involved will be reported to the emergency spill control coordinator.
- The Emergency Coordinator, or designee, will make a final tally of persons. No attempt to find persons not accounted for will involve endangering lives of Shaw or other employees by re-entry into emergency areas.

In all questions of accountability, immediate crew leaders will be held responsible for those persons reporting to them. Visitors will be the responsibility of those employees they are seeing. Contractors and truck drivers are the responsibility of the Site Superintendent.

- Personnel will be assigned by the Emergency Coordinator to be available to direct and brief emergency responders.

- Re-entry into the site will be made only after the Emergency Coordinator gives clearance. At his direction, a signal or other notification will be given for re-entry into the facility.

- Drills will be held annually, at a minimum, to practice all of these procedures and will be treated with the same seriousness as an actual emergency.

9.4 EMERGENCY SPILL RESPONSE PROCEDURES AND EQUIPMENT

In the event of an emergency involving a hazardous material spill or release, the following general procedures will be used for rapid and safe response and control of the situation. Emergency contacts found in Table 9.1 (Page 9-4) provide a quick reference guide to follow in the event of a major spill.

9.4.1 Notification Procedures

If an employee discovers a chemical spill or process upset resulting in a vapor or material release, he or she will immediately notify the on-site Emergency Coordinator.

On-site Emergency Coordinator will obtain information pertaining to the following:

- The material spilled or released.
- Location of the release or spillage of hazardous material.
- An estimate of quantity released and the rate at which it is being released.
- The direction in which the spill, vapor or smoke release is heading.
- Any injuries involved.
- Fire and/or explosion or possibility of these events.
- The area and materials involved and the intensity of the fire or explosion.

This information will help the on-site Emergency Coordinator to assess the magnitude and potential seriousness of the spill or release.

9.4.2 Procedure for Containing/Collecting Spills

The initial response to any spill or discharge will be to protect human health and safety, and then the environment. Identification, containment, treatment and disposal assessment will be the secondary response.

- Construction of a temporary containment berm utilizing on-site clay absorbent earth
- Digging a sump, installing a polyethylene liner
- Diverting the spill material into the sump placing drums under the leak to collect the spilling material before it flows over the ground
- Transferring the material from its original container to another container.

The Emergency Coordinator will notify the ROICC of the spill and steps taken to institute clean up. Emergency response personnel will clean up all spills following the spill clean-up plan developed by the Emergency Coordinator. Supplies necessary to clean up a spill will be immediately available on-site. Such items may include, but are not limited to:

- Shovel, rake
- Sorbent materials
- Personal safety equipment
- Steel drums
- Miscellaneous hand tools.

As called for in regulations developed under the Comprehensive Environmental Response Compensation Liability Act of 1980 (Superfund), Shaw practice is to report a spill of a pound or more of any hazardous material for which a reportable quantity has not been established and which is listed under the Solid Waste Disposal Act, Clean Air Act, Clean Water Act, or TSCA. Shaw also follows the same practice for any substances not listed in the Acts noted above but which can be classified as a hazardous waste under RCRA.

Clean up personnel will take the following measures:

1. Make sure all unnecessary persons are removed from the hazard area.
2. Put on protective clothing and equipment.

3. If a flammable material is involved, remove all ignition sources, and use spark and explosion proof equipment for recovery of material.

4. Remove all surrounding materials that could be especially reactive with materials in the waste. Determine the major components in the waste at the time of the spill.

5. If wastes reach a storm sewer, try to dam the outfall by using sand, earth, sandbags, etc. If this is done, pump this material out into a temporary holding tank or drums as soon as possible.

6. Place all small quantities of recovered liquid wastes (55 gallons or less) and contaminated soil into drums for incineration or removal to an approved disposal site.

7. Spray the spill area with foam, if available, if volatile emissions may occur.

8. Apply appropriate spill control media (e.g., clay, sand, lime, etc.) to absorb discharged liquids.

9. For large spills, establish diking around leading edge of spill using booms, sand, clay or other appropriate material. If possible, use diaphragm pump to transfer discharged liquid to drums or holding tank.

9.4.3 Emergency Response Equipment

The following equipment will be staged in the support zone and throughout the site, as needed, to provide for safety and first aid during emergency responses.

- ABC-type fire extinguisher
- First-aid kit, industrial size
- Eyewash
- Emergency signal horn.

9.4.4 Emergency Spill Response Clean-Up Materials and Equipment

A sufficient supply of appropriate emergency response clean-up and personal protective equipment will be inventoried and inspected, visually, on a weekly basis.

The materials listed below may be kept on site for spill control, depending on the types of hazardous materials present on site. The majority of this material will be located in the support zone. Small amounts will be placed on pallets and located in the active work areas.

- Appropriate solvents (e.g., CITRIKLEEN, for decontamination of structures or equipment).

- Sand or clay to solidify/absorb liquid spills.

9.4.5 Medical Emergency Contingency Measures

The procedures listed below will be used to respond to medical emergencies. The HSM will contact the local hospital and inform them of the site hazards and potential emergency situations. A minimum of two first-aid/CPR trained personnel will be maintained on site.

9.4.6 Response

The nearest workers will immediately assist a person who shows signs of medical distress or who is involved in an accident. The work crew Superintendent will be summoned.

The work crew Superintendent will immediately make radio contact with the on-site Emergency Coordinator to alert him of a medical emergency situation. The Superintendent will advise the following information:

- Location of the victim at the work site
- Nature of the emergency
- Whether the victim is conscious
- Specific conditions contributing to the emergency, if known.

The Emergency Coordinator will notify the Site Safety Officer. The following actions will then be taken depending on the severity of the incident:

Life-Threatening Incident. If an apparent life-threatening condition exists, the crew Superintendent will inform the Emergency Coordinator by radio, and the local Emergency Response Services (EMS) will be immediately called. An on-site person will be appointed who will meet the EMS and have him/her quickly taken to the victim. Any injury within the EZ will be evacuated by Shaw personnel to a clean area for treatment by (EMS) personnel. No one will be able to enter the EZ without showing proof of training, medical surveillance and site orientation.

Any personnel requiring emergency medical attention will be evacuated from exclusion and contamination reduction zones if doing so would not endanger the life of the injured person or otherwise aggravate the injury. Personnel will not enter the area to attempt a rescue if

their own lives would be threatened. The decision whether or not to decontaminate a victim prior to evacuation is based on the type and severity of the illness or injury and the nature of the contaminant. For some emergency victims, immediate decontamination may be an essential part of life-saving first aid. For others, decontamination may aggravate the injury or delay life-saving first aid. Decontamination will be performed if it does not interfere with essential treatment.

If decontamination can be performed, wash external clothing and cut it away.

If decontamination cannot be performed, observe the following procedures.

- Wrap the victim in blankets or plastic to reduce contamination of other personnel.
- Alert emergency and off-site medical personnel to potential contamination, instruct them about specific decontamination procedures.
- Send site personnel familiar with the incident and chemical safety information (e.g., MSDS) with the affected person.

An accident/injury/illness report will be completely and properly filled out and submitted to the Program Health and Safety /Project CIH, in accordance with Shaw reporting procedures.

A list of emergency telephone numbers is given in Table 9.1.

Non Life-Threatening Incident. All injuries, no matter how small, will be reported to the SS/SSO and HSM. If it is determined that no threat to life is present, the Site Superintendent will direct the injured person through decontamination procedures appropriate to the nature of the illness or accident. Appropriate first-aid or medical attention will then be administered.

*NOTE: The area surrounding an accident site must not be disturbed until the scene has been cleared by the Site Superintendent.

9.4.7 Notification

The following personnel/agencies will be notified in the event of a medical emergency:

- Local Fire Department or EMS
- On-site Emergency Coordinator
- Workers in the affected areas
- Notify Shaw PM
- Notify ROICC

9.4.8 Fire Contingency Measures

Shaw personnel and subcontractors are not trained professional firefighters. Therefore, if there is any doubt that a fire can be quickly contained and extinguished, personnel will notify the Emergency Coordinator by radio and vacate the structure or area. The Emergency Coordinator will immediately notify the local Fire Department.

The following procedures will be used to prevent the possibility of fires and resulting injuries:

- Sources of ignition will be kept away from where flammable materials are handled or stored.

- "No smoking" signs will be conspicuously posted in areas where flammable materials are present and throughout the exclusion and contamination reduction zones.

- Fire extinguishers will be located in all Shaw site dedicated vehicles and placed in all areas where a fire hazard may exist.

- Before workers begin operations in an area the foreman will give instruction on egress procedures and assembly points. Egress routes will be posted in work areas and exit points clearly marked.

The following procedures will be used in the event of a fire:

- Anyone who sees a fire will notify his or her Superintendent who will then contact the Emergency Coordinator by radio. The Emergency Coordinator will activate the emergency air horns and contact the local Fire Department.

- When the emergency siren sounds, workers will disconnect electrical equipment in use (if possible) and proceed to the nearest fire exit.

- Work crews will be comprised of pairs of workers (buddy system) who join each other immediately after hearing the fire alarm and remain together throughout the emergency. Workers will assemble at a predetermined rally point for a head count.

- When a worker has extinguished a small fire, the Emergency Coordinator will be notified.

9.5 HAZARDOUS WEATHER CONTINGENCY MEASURES

Operations will not be started or continued when the following hazardous weather conditions are present:

- Lightning
- Heavy Rains
- High Winds

9.5.1 Response

- All equipment will be shut down and secured to prevent damage.

- Personnel will be moved to safe refuge. The Emergency Coordinator will determine when it is necessary to evacuate personnel to off-site locations and will coordinate efforts with fire, police, and other agencies.

9.5.2 Notification

The Emergency Coordinator will be responsible for assessing hazardous weather conditions and notifying personnel of specific contingency measures. Notifications will include:

- Shaw employees and subcontractors
- Notify Shaw PM
- Notify Air Force Representative

10.0 TRAINING REQUIRMENTS

As a requirement for work at this site, in any hazardous waste work area, all field personnel will be required to take a 40-hour training class. This training must cover the requirements in 29 CFR 1910.120: personal protective equipment, toxicological effects of various chemicals, hazard communication, blood borne pathogens, handling of unknown tanks and drums, confined-space entry procedures, electrical safety, etc. In addition, all personnel must receive annual 8-hour refresher training and three-day on-site training under a trained, experienced Superintendent. Supervisory personnel shall have received an additional 8-hour training in handling hazardous waste operations.

All personnel entering the exclusion zone will be trained in the provisions of this site safety plan and be required to sign the Site Safety Plan Acknowledgment in Appendix H.

Site-specific training for activities at SWMU-6, SWMU-7 and AOC-J will include potential site contaminants, UXO Awareness training, Hazard Communication, as per 29 CFR 1926.59, site physical and environmental hazards, emergency response and evacuation procedures, and emergency telephone numbers will be held at the site location by the SS/SSO before any site work activities begin.

Outlines of the orientation for Shaw personnel and subcontract personnel and visitors are presented below:

Shaw/SUBCONTRACTORS	**VISITOR ORIENTATION**
• SSHSP sign off • Sign in/out procedures • Site background • Chain of command • Rules and regulations • Hours of work • Absences • Equipment • Emergency Information • Emergency signal • Gathering point • Responsibilities/roles • Emergency phone numbers • Work Zones • Contaminants, MSDS's [Hazard Communication Program] • AHA's (Activity Hazard Analyses) • Forms, site-specific Incident Reporting	• SSHSP signoff • Review of Site map • Work Zones in progress • Hazard Communication • Emergency plan/signals • Training/medical requirements • Zones/areas open to visitors

11.0 MEDICAL SURVEILLANCE PROGRAM

All Shaw personnel participate in a medical and health monitoring program. This program is initiated when the employee starts work with a complete physical and medical history and is continued on a regular basis. A listing of Shaw's worker medical profile is shown below. This program was developed in conjunction with a consultant toxicologist and Shaw's occupational health physician. Other medical consultants are retained when additional expertise is required.

All personnel performing intrusive work will receive pre- and post-project blood lead testing meeting the requirements of 29CFR1926.62. In the event of an un-protected exposure to lead dust or signs of over-exposure personnel may be required to submit to blood lead testing during the course of the project.

All field personnel performing activities in a designated EZ or CRZ shall within the past 12 months have completed a comprehensive medical examination that meets the requirements of OSHA regulations 29 CFR 1910.120 and 29 CFR 1926.65. The annual medical includes the following elements:

- Medical and occupational history questionnaire
- Physical examination
- Complete blood count, with differential
- Liver enzyme profile
- Chest x-ray, once every 3 years, for non-asbestos workers
- Pulmonary function test
- Audiogram
- Electrocardiogram for persons older than 35 years of age, or if indicated during the physical examination
- Visual acuity
- Follow-up examinations, at the discretion of the examining physician or the corporate medical director.

The medical surveillance program meets the requirements of the OSHA Standard 29 CFR 1910.120/1926.65(f).

The following information is provided in the event that medical attention is necessary.

The Shaw Medical Director is:

Dr. Jerry H. Berke
MD, MPH
Health Resources
600 West Cumming Park
Suite 3400
Woburn, Mass 01801-6350
781-935-8581 (direct dial)
800-350-4511 (toll free)

The Shaw Medical Director and the HSM will be immediately notified of any suspected exposures to hazardous materials/wastes.

APPENDIX A
MATERIAL SAFETY DATA SHEETS (MSDS's)

APPENDIX B
SPECIFIC HEALTH AND SAFETY PROCEDURES

NOTE: Health and safety procedures that will be utilized during the project are listed below. A copy of all the Health and Safety Procedures (HS001-999) is available on the Intranet (ShawNet).

HS020	Accident Prevention Program: Reporting Investigation and Review
HS021	Accident Prevention Program: Management Safety Reviews
HS045	Job Safety Analysis
HS051	Tailgate Safety Meeting
HS060	Hazard Communication Program
HS303	Pressurized Cleaning and Cutting Equipment
HS307	Excavation and Trenching
HS308	Underground/Overhead Utility Contact Prevention
HS400	Working in Hot Environments
HS401	Cold Stress
HS402	Hearing Conservation
HS600	Personal Protection Program
HS601	Respiratory Protection
HS800	Motor Vehicle Operation: General Requirements

PROCEDURE

Subject: ACCIDENT PREVENTION PROGRAM: REPORTING, INVESTIGATION, AND REVIEW

1.0 PURPOSE AND SUMMARY

The purpose of this procedure is to establish the requirements for incident reporting, investigation, and review. This procedure is an integral part of the company's overall accident prevention program and aids in the identification of potential causal factors and corrective actions. Key elements of this procedure include:

- **All occupational injuries/illnesses, vehicle accidents, and near miss incidents must be promptly reported and investigated.**

- All Occupational Safety and Health Administration (OSHA) recordable injuries/illnesses and chargeable vehicle accidents must be reviewed by an Accident Review Board. The Accident Review Board report is submitted to the Baton Rouge Corporate Safety Department, for production to and retention on behalf of the Legal Department.

- All incidents involving a fatality, major injury/illness, or resulting in significant property damage will be immediately reported to: the business line Health & Safety Manager; the Corporate Health and Safety Department; Business Line Vice President and the Legal Department.

- All investigations and associated materials obtained and/or produced, in association with OSHA recordable injuries/illnesses, chargeable vehicle accidents, fatalities, major injury/illness, or incidents resulting in significant property damage, are to be performed for & on behalf of the legal department and will be subject to being classified as Confidential Attorney-Client / Attorney Work Product.

- All business line Health & Safety Managers are required to prepare a Monthly Loss Report summarizing all current month, and year-to date, chargeable vehicle accidents, injury/illness cases (requiring outside medical care), lost work day totals and restricted work day totals. This report shall then be forwarded, by the 10th day of the following month, to the Baton Rouge Corporate Safety Office.

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3.0 RESPONSIBILITY MATRIX

3.1 Procedure Responsibility

The Corporate Health & Safety Department is responsible for the issuance, revision, and maintenance of this procedure.

3.2 Action/Approval Responsibilities

The Responsibility Matrix is Attachment 1.

4.0 DEFINITIONS

Chargeable Vehicle Accident - Any **at-fault** vehicle accident meeting any **one** of the following criteria:

- An individual other than an employee of the company is a party in the accident
- Property owned by a person or entity other than the company is damaged
- When company owned, leased or rented vehicles are involved and damage exceeds $2,500.00.
- When an employee is driving a personal vehicle while on company business and damage exceeds $2,500.00.

Company - All affiliates, indirect and wholly owned subsidiaries of Shaw Environmental & Infrastructure, Inc. (Shaw E & I).

Days Away From Work - Days away from work are the number of **calendar** days following the injury or illness, excluding the date of the injury.

Job Safety Analysis (JSA) – The JSA is an effective management technique for identifying hazardous conditions and unsafe acts in the workplace. A JSA is intended to analyze the individual steps or activities, which together create a job or specific work duty, and to detect any actual or potential hazards that may be present. (See HS045: Job Safety Analysis)

Restricted Work – Occurs when, as the result of a work-related injury or illness:

- A physician or other licensed health care professional recommends that the employee not perform one or more of the routine functions of his or her job, or not work the full workday that he or she would otherwise have been scheduled to work

Near Miss Incident - Any incident where no injury occurred, but where the potential for injury existed.

OSHA Recordable Case – See Attachment 8

Vehicle - Any passenger vehicle, including trucks, used upon the highway or in private facilities for transporting passengers and/or property. For the purpose of this procedure, off-road vehicles such as earthmoving equipment, forklifts, non-highway use trucks, etc., are not considered vehicles. (See HS800 Motor Vehicle Operation: General Requirements)

5.0 TEXT

5.1 Incident Reporting Process

Employees are required to immediately report to their direct supervisor all occupational injuries, illnesses, accidents and near miss incidents having the potential for injury. Site Business Line Managers or Supervisors (supervisor directly responsible for the employee involved in the incident) with first-hand knowledge of an incident is required to:

- Immediately arrange for appropriate medical attention and notify the responsible health and safety representative.

- **As soon as practical, but not longer than one hour after gaining knowledge of the occurrence**, notify the Shaw Notification Hotline/Helpdesk by calling 1-866-299-3445 (Attachment 10) of any injury requiring off-site medical treatment, any chargeable vehicle accident or equipment incident involving property damage exceeding $2,500 in value (Shaw or third party).

- Inform Health Resources of all incidents requiring off-site medical attention by calling 1-800-350-4511. This call should be made **prior** to transporting the employee such that they can coordinate physicians services prior to arrival of the employee to the clinic, and provide the following information:

  - Company Name (Shaw E&I) & Business Line (e.g. DOD, Commercial)
  - Employee Name
  - Name of anticipated, treating medical facility and phone number
  - Brief description of incident.

  Health Resource's role is to interface with the treating physician, to ensure that appropriate care is provided to the injured employee.

- Complete the *Authorization for Treatment, Release of Medical Information*, and *Return to Work* (Attachment 9A, 9B, 9C) and the *Supervisor's Employee Injury Report* (Attachment 2) for all cases requiring off-site medical attention. The Site Safety and Health Representative or responsible supervisor shall ensure that the forms are completed and faxed to Health Resources at (800) 853-2641 prior to leaving the medical facility or as soon as reasonably possible.

- Post accident drug and alcohol testing shall occur in accordance with HS101 Drug and Alcohol Testing, immediately following an incident.

  NOTE: Prior to performing non-DOT post accident testing, it is the responsibility of the employee's supervisor to ensure that Health Resources has verified that this testing is not prohibited or restricted by state or local regulations.

- Prior to an injured employee returning to his/her job duties, a follow-up call by Health Resources will be made to the project site. The purpose of this call is to ensure work restrictions are clarified and planned work activities are consistent with medical recommendations.

- The Supervisor shall initiate/complete the appropriate company documentation in accordance with the following incident classifications: (note: if a Site Safety and Health Representative is on site, he should work in concert with the supervisor)

  - OSHA Recordable Cases
    a. Supervisor's Employee Injury/Illness Report (Attachment 2)
    b. Incident Investigation Report (Attachment 5)
    c. Witness Statement Form (Attachment 6)
    d. Accident Review Board (Attachment 7)

  - First Aid Cases
    a. Supervisor's Employee Injury/Illness Report (Attachment 2)
    b. Incident Investigation Report (Attachment 5)
    c. Witness Statement Form (Attachment 6)

  - Chargeable Vehicle Accidents
    a. Vehicle Accident Report (Attachment 3)
    b. Incident Investigation Report (Attachment 5)
    c. Witness Statement (Attachment 6)
    d. Accident Review Board (Attachment 7)
    e. Driving Record Certification (Procedure HS800)

  - Non-Chargeable Vehicle Accidents
    a. Vehicle Accident Report (Attachment 3)
    b. Incident Investigation Report (Attachment 5)
    c. Witness Statement (Attachment 6)

- Equipment, Property Damage and General Liability Incidents
  a. Incident Investigation Report (Attachment 5)
  b. Witness Statement Form (Attachment 6)
  c. Equipment, Property Damage and General Liability Loss Report (Attachment 4).

- Near Miss
  a. Incident Investigation Report (Attachment 5)

5.2 Supervisor's Employee Injury/Illness Report (Attachment 2)

The Supervisor's Employee Injury Report is to be completed for all incidents that result in an employee occupational injury or illness requiring off-site medical attention. It is to be initiated by the supervisor of the injured employee and forwarded to the respective Business Line Safety Manager for review / comments. Upon completion of review and comments the report should be forwarded, **within 24 Hours,** to the Shaw Corporate Claims department in Baton Rouge, via the corporate claims fax number (225.932.2636).

5.3 Vehicle Accident Report (Attachment 3)

The Vehicle Accident Report must be completed for any vehicle accident in which a company vehicle is involved. This includes company-owned or leased vehicles, rental vehicles, and personal vehicles being used for company business. This report is to be initiated by both the employee involved in the accident and his/her direct supervisor and forwarded to the respective Business Line Safety Manager for review / comments. Upon completion of review and comments the report should be forwarded to the Shaw Corporate Claims department in Baton Rouge (fax number 225.932.2636).

5.4 Equipment, General Liability, Property Damage, and Loss Report (Attachment 4)

The General Liability, Property Damage, and Loss Report is to be used for all losses or damage to company property in excess of $2,500.00. This form must be completed for all third party property, regardless of value, damaged as a result of company activities. The employee most familiar with the events that contributed to the loss or damage will complete the form, and then forward it to the project/location manager. The Corporate Claims Department and the respective Business Line Safety Manager must receive a copy of the report within one business day of the incident.

5.5 Incident Investigation Report (Attachment 5)

All injuries, illnesses, accidents, and near miss incidents will be investigated. Once arrangements for immediate medical care have been made, the employee's direct supervisor, with assistance from the health and safety representative and Business Line Health and Safety Manager, will:

- Collect the facts;

- Describe and document (include sketch, photos, etc.) how the incident occurred;

- Collect support documentation (JSA's, AHA's, Tailgate Safety Meetings, Work Orders, etc.);

- List witnesses and collect written statements;

  <u>If applicable, contact the employee's Functional Manager in an effort to gain relevant information</u>

- Identify the causative factors;

- Identify potentially unsafe acts or unsafe conditions that may have contributed to the incident;

- Identify potential curative action; and

- List the corrective actions which are to be executed, appropriate curative action, the person(s) responsible for the corrective action, and the date by which action is to be completed.

The investigation will be started as soon as possible following the incident and the relevant reports and support documentation (JSA's, AHA's, Tailgate Safety Meetings, Work Orders, etc.) shall be submitted to the appropriate Business Line Health and Safety Manager within 72 hours. In addition to the previous information, reports from external sources (police, insurance carriers, testing laboratories, etc.) are to be obtained as soon as they become available and forwarded by the Business Line Safety Manager to the Corporate Claims department in Baton Rouge.

5.6 Injured Employee Statement & Witness Statement Forms (Attachment 6a & 6b)

The Injured Employee and Witness Statement Forms allow for consistency in the development of the investigation process. The Injured Employee Statement must be completed in all cases where an employee injury results in off site medical treatment. If there are witnesses to the accident/incident, the Witness Statement form should be completed and signed by the subject witness. Both of these forms should be attached to the incident investigation report. It is essential that these statements are executed immediately following the incident to ensure an accurate account of the events.

5.7 Accident Review Board (ARB) (Attachment 7)

The purpose of the Accident Review Board is to collect and review the information gathered for each incident, report that information to the Legal Department and take appropriate curative action. In all cases, the purpose of the entire investigative process, inclusive of conducting an ARB, is to identify curative actions as it relates to the incident / injury. Accordingly, a diligent and concerted effort to accomplish these tasks must be established at the onset of all of the subject incidents.

In order to assist the Legal Department in evaluating the risk to, or liability of, the company, associated with OSHA recordable injuries, chargeable vehicle accidents, fatalities or incidents resulting in significant property damage, the responsible Project / Location Manager is required to coordinate with all parties and set up the ARB such

that it occurs **within 10 days of the accident**. The respective Business Line Health and Safety Manager, whose project/location experiences accident is then required to conduct the subject ARB.

The Accident Review Board shall be composed of the project/location manager, the employee's direct supervisor (at time of incident), a health and safety representative, and the employee(s) involved in the incident.

Additionally, there may be cases that involve an employee that has been assigned to a project and the Functional Manager of that employee may not have direct knowledge of an incident. In cases such as these, the Functional Manager shall be notified of the incident and requested to participate in the ARB. Also, as determined by the Business Line Health and Safety Manager, a representative of other internal sources of expertise should be involved where applicable.

All investigations and associated materials obtained and/or produced, in association with injuries/illnesses resulting in OSHA recordable classification, chargeable vehicle accidents, fatalities or incidents resulting in significant property damage, are to be performed for and on behalf of the legal department and will be subject to being classified as Confidential Attorney-Client / Attorney Work Product. If the ARB is initiated under a Confidential Attorney-Client / Attorney Work Product status, all documents and other work product arising out of, or associated with, the investigation process, including the ARB, shall be prepared in anticipation of litigation. The Accident Review Board report, and associated documents, is submitted to the Corporate Safety Department, for production to and retention on behalf of the Legal Department.

The ARB report, and all associated documents, shall be completed as soon as practicable, but not more than 5 business days following the ARB meeting, and forwarded by the Business Line Safety Manager to the Corporate Safety Department, via the Corporate Claims fax number. The original documents shall then be mailed to the Corporate Safety Department. These documents shall then be filed in a lockable cabinet, separate from files not meeting the subject criteria, by the Corporate Safety Department, for production to and retention on behalf of the Legal Department. In the event that copies of these files are maintained by Business Line Safety Managers and / or the respective location in which the injury occurred, the same filing criteria shall be followed. The criteria shall be that these documents are filed in lockable cabinets, separate from files not meeting the subject Attorney-Client / Attorney Work Product criteria.

It is generally not acceptable to discipline an employee for having an accident. However, if in the opinion of the Accident Review Board, it is determined that the accident resulted from an intentional unsafe act or intentional violation of company procedure on the employee's part, the employee may be subject to disciplinary action in accordance with the company's progressive disciplinary action system (see Human Resources Procedure HR207).

5.8 Monthly Loss Report

Each business line Health and Safety Manager is responsible to submit a Monthly Loss Report summarizing incidents that took place within their business line during the previous month. The business line Health and Safety Manager is responsible for submitting a consolidated package for the entire business line to the corporate health and safety office for **receipt no later than the 10th working day of the following month**.

6.0 EXCEPTION PROVISIONS

Variances and exceptions may be requested pursuant to the provisions of Procedure HS013, Health and Safety Procedure Variances.

7.0 CROSS REFERENCES

HR207 Disciplinary Action
HS013 Health and Safety Procedure Variances
HS101 Drug and Alcohol Testing
HS800 Motor Vehicle Operations - General Requirements
HS810 Commercial Motor Vehicles

8.0 ATTACHMENTS

1. Responsibility Matrix
2. Supervisor's Employee Injury/Illness Report
3. Vehicle Accident Report
4. Equipment, Property Damage and General Liability Loss Report
5. Incident Investigation Report
6. a. Injured Employee Statement
   b. Witness Statement
7. Accident Review Board Report
8. Injury/Illness Classification Guidelines
9. Medical Forms
   a. Authorization for Treatment of Occupational Injury/Illness
   b. Authorization for Release of Medical Information
   c. Return to Work Examination Form.
10. Help Desk / Hotline Notification Guidelines

ATTACHMENT 1

ACCIDENT PREVENTION PROGRAM: REPORTING, INVESTIGATION, AND REVIEW RESPONSIBILITY MATRIX

Action	Procedure Section	*Responsible Party*					
		Employee	Supervisor	Project/ Location Manager	Site Health and Safety Rep. / Officer	Business Line Health and Safety Manager	Corporate Health & Safety Manager
Issue, Revise, and Maintain Procedure	3.1						X
Report All Incidents to Supervisor	5.1	X					
Notify Health and Safety Representative	5.1		X				
Arrange Medical Care	5.1		X		X		
Notify Health Resources or Gates McDonald of Incident	5.1		X		X		
Initiate/Complete Company Forms	5.1		X		X		
Complete Investigation of incident	5.5		X	X	X	X	
Complete Equipment, Property Damage and General Liability Loss Report Incident	5.4	X					
Coordinate and Set up Accident Review Board	5.7			X			
Conduct Accident Review Board	5.7					X	
Participate in Accident Review Board	5.7	X	X	X	X	X	
Complete Monthly Loss Report	5.8					X	

Attachment 2

REPORT ALL WORKER'S COMPENSATION INJURIES TO SHAW CLAIMS DEPARTMENT
FAX REPORT WITHIN 24 HOURS OF INCIDENT TO **225-932-2636**.
Phone all injuries/ illnesses to Shaw Notification Hotline/Helpdesk
1-866-299-3445

Supervisor's Employee Injury/Illness Report Form

EMPLOYEE INFORMATION

Employee's Social Security Number:		Claim Number:	
Employee's Name:		Home Phone Number:	
Home Address:			Business Line Code:
Male Female	Date of Birth:	Hire Date:	
Dependents:	Dependents Under 18:	Marital Status:	
Occupation:		Department Name:	
State Hired:	Currently Weekly Wage:	Hourly Wage:	
Hours/Days Worked Per Week:	Days Per Week	Hours Worked Per Day:	
Employment Status:	Employee Report No.: N/A		Employee ID No.: N/A
Salaried Continued:	Paid For Date of Injury:		Education No. of Years:
Ever Injured on the Job:	Supervisor Name & Phone:		

EMPLOYER INFORMATION

Employer Name:	**The Shaw Group, Inc.**		
Work Location:			
Contact Name:	**John Mollere**	Telephone Number:	**(800)747-3322, Ext. 572**
Employer SIC:		Employer Location Code:	
Employer FED ID:		Employer Code:	N/A
Nature of Business:			
Policy Number:			

ACCIDENT INFORMATION

Date and Time of Injury:			
Did the Accident Occur at the Work Location:		If no, where did the accident occur?	N/A
Accident Address:			
Nature of Accident:			

Give a Full Description of the Accident: (Be as Factually Complete As Possible)

Are Other WC Claims Involved?	No	Date and Time Reported to Employer:	
Person Reported To:			

WITNESS INFORMATION

Were There Any Witnesses?

If Yes, List Names and How to Contact Them:

INJURY INFORMATION

Which Part of the Body Was Injured? (e.g. Head, Neck, Arm Leg)

What Was the Nature of Injury? (e.g. Fracture, Sprain, Laceration)

Part of Body Location: (e.g. Left, Right, Upper, Lower)

Injury Description:

Source of Injury: | Is Employee Hospitalized?

Lost Time: If Yes, What was First Full Day Out:

Date Last Day Worked: | Date Disability Began: N/A

Date Returned to Work: | Estimated Return Date: N/A

MEDICAL INFORMATION

ER Treated & Released: | Hospitalized: | Phy./Clinic:

Hospital - Name, Address, Phone Number: | Was Employee Transported via Ambulance: Yes No

N/A

Clinic - Name, Address, Phone Number:

ADDITIONAL COMMENTS & INFORMATION

REPORT PREPARED BY

Name: | Title:

Signature: | Phone:

ATTACHMENT 3
VEHICLE ACCIDENT REPORT
Page 1 of 2

ACCIDENT DESCRIPTION

This report is to be initiated by the employee involved in the accident or his/her direct supervisor. Please answer all questions completely. This report must be forwarded to the appropriate health and safety representative within 24 HOURS of the accident. Attach police report.

ACCIDENT DATE _____ TIME _____ ☐ A.M. or ☐ P.M.

LOCATION OF ACCIDENT (CITY, STATE) _____

DESCRIPTION OF ACCIDENT _____

_____

_____

WITNESS _____ PHONE NO. _____

ADDRESS _____ CITY _____ STATE _____ ZIP _____

POLICE OFFICER'S NAME AND BADGE #_____ DEPARTMENT _____

COMPANY VEHICLE

DRIVER _____ DRIVERS LICENSE NO. _____ STATE _____

ADDRESS _____ _____ CITY _____ STATE _____ ZIP _____

WORK PHONE NO. _(_____)_____ S.S. NO. _____ PROJECT NAME/NO. _____

VEHICLE NO. _____ YEAR _____ MAKE _____ MODEL _____ LICENSE PLATE NO. _____

STATE _____ VEHICLE OWNER: ☐ COMPANY ☐ LEASED/RENTED ☐ PRIVATE VEHICLE

VEHICLE TYPE: ☐ COMMERCIAL MOTOR VEHICLE ☐ NON-COMMERCIAL

IF NOT COMPANY-OWNED: OWNER _____ PHONE NO _(____)_____

ADDRESS _____ _____ CITY _____ STATE _____ ZIP _____

VEHICLE DAMAGE _____

NO. OF VEHICLES TOWED FROM SCENE _____ NUMBER OF INJURIES _____ NUMBER OF FATALITIES _____

WERE HAZARDOUS MATERIALS RELEASED? ☐ NO ☐ YES IF YES, DESCRIBE MATERIALS _____

_____

OTHER VEHICLE

DRIVER _____ DRIVERS LICENSE NO. _____ STATE _____

ADDRESS _____ CITY _____ STATE _____ ZIP _____

PHONE NO. _(_____)_____ S.S. NO. _____

OWNER'S NAME (☐ CHECK IF SAME AS DRIVER) _____

ADDRESS _____ CITY _____ STATE _____ ZIP _____

INSURANCE COMPANY _____ POLICY NO. _____

AGENT'S NAME _____ PHONE NO. _(_____)_____

ADDRESS _____ CITY _____ STATE _____ ZIP _____

VEHICLE YEAR _____ MAKE _____ MODEL _____ PLATE NO. _____ STATE _____

VEHICLE I.D. NO. _____

VEHICLE DAMAGE _____

PASSENGERS: ☐ NO ☐ YES INJURIES: ☐ NO ☐ YES (If Yes, list names and telephone numbers below)

_____

_____

_____

VEHICLE ACCIDENT REPORT

WEATHER: ☐ Clear ☐ Cloudy ☐ Fog ☐ Rain ☐ Sleet ☐ Snow Other _____

PAVEMENT: ☐ Asphalt ☐ Steel ☐ Concrete ☐ Wood ☐ Gravel/Dirt
☐ Brick/Stone Other _____

CONDITION: ☐ Dry ☐ Wet ☐ Icy ☐ Pot Holes Other _____

TRAFFIC CONTROL: ☐ Traffic Light ☐ Stop Sign ☐ Railroad ☐ No Intersection ☐ No Control

ROADWAY: Number of Lanes Each Direction: _____ ☐ Residential ☐ Divided Highway ☐ Undivided Highway

Draw and name roadways showing each vehicle, direction of travel, and point of impact. Indicate travel before the accident with a solid line, and post-accident movement with a broken line.

SYMBOLS:

Your Vehicle ①

Other Vehicle(s) ②③

Pedestrian

Stop Sign

Yield

Railroad

ADDITIONAL INFORMATION:

_____

_____

_____

_____

EMPLOYEE _____ (Print) _____ (Signature) _____ (Date)

SUPERVISOR _____ (Print) _____ (Signature) _____ (Date)

HEALTH & SAFETY REP. _____ (Print) _____ (Signature) _____ (Date)

ATTACH POLICE REPORT TO VEHICLE ACCIDENT REPORT

REPORT MUST BE FAXED TO:
CORPORATE CLAIMS DEPARTMENT (FAX: 225-932-2636)
WITHIN 24 HOURS, OR NOT LATER THAN NEXT BUSINESS DAY.

REPORT ALL CHARGEABLE VEHICLE ACCIDENTS TO SHAW NOTIFICATION HOTLINE/HELPDESK
(PHONE: 1-866-299-3445)

ATTACHMENT 4

EQUIPMENT, PROPERTY DAMAGE AND GENERAL LIABILITY LOSS REPORT

This report is to be completed for all losses or damage to company property in excess of $2,500.00 and all third party damage, regardless of value, resulting from company activities.

PROJECT/LOCATION _____ PROJECT NO. _____ DATE _____

ADDRESS _____

HOW DID DAMAGE OR LOSS OCCUR: _____

_____

_____

DESCRIPTION AND VALUE ($) OF DAMAGED/LOST/STOLEN PROPERTY: _____

_____

LOCATION OF DAMAGED/LOST/STOLEN PROPERTY (Before Loss): _____

_____

DATE AND TIME OF DAMAGE, LOSS, OR THEFT: Date: _____ Time: _____ a.m./p.m.

OWNER OF DAMAGED/LOST/STOLEN PROPERTY:

Name _____ Phone No. (_____) _____

Address _____ City _____

Employer and Address _____

INJURED PARTIES (Also complete a Supervisor's Employee Injury Report if a Company Employee):

Name _____ Phone No. (_____) _____

Address _____ City _____

Employer and Address _____

Description of Injury _____

WITNESSES:

1. Name _____ Home Phone (_____) _____
   Home Address _____ City _____
   Employer and Address _____
2. Name _____ Home Phone (_____) _____
   Home Address _____ City _____
   Employer and Address _____

WERE PICTURES TAKEN? ☐ YES ☐ NO

WERE POLICE NOTIFIED? ☐ YES ☐ NO DEPT. _____ REPORT NO. _____

COMPLETED BY: _____ _____ _____

(Print) (Signature) (Date)

PROJECT/LOCATION MANAGER: : _____ _____ _____

(Print) (Signature) (Date)

REPORT MUST BE FAXED TO:
CORPORATE CLAIMS DEPARTMENT (FAX: 225-932-2636)
WITHIN 24 HOURS, OR NOT LATER THAN NEXT BUSINESS DAY

ATTACHMENT 5
INCIDENT INVESTIGATION REPORT

*** Must Be Completed Within 72 HOURS & Relevant Support Documentation Must Be Attached / Submitted***

Investigation Date _____ Date of Incident _____

Employee Name _____

Supervisor Name _____

Project Number/Name _____ / _____

Location of Incident _____

- Incident Classification

Injury	☐ First Aid	Vehicle	☐ Chargeable	DOT	☐ DOT Vehicle
	☐ OSHA Recordable		☐ Non-chargeable		☐ DOT Reportable
	☐ Lost Workday				
	☐ Restricted Workday	Near Miss	☐	General Liability	☐

- Description (Provide facts, describe how incident occurred, provide diagram [on back] or photos)

_____

_____

- Analysis (What unsafe acts or conditions contributed to the incident?)

_____

_____

_____

_____

- Corrective Action(s) (List corrective action items, responsible person, scheduled completion date)

_____

_____

_____

- Witness Names (Complete Attachment 6 – Employee Witness Statement)

_____

_____

_____

_____

Investigated By	_____	_____	_____
	Print Name	Signature	Date
Project/Location Mgr.	_____	_____	_____
	Print Name	Signature	Date

ATTACHMENT 6a
Injured Employee Statement
MUST BE COMPLETED WITHIN 24 HOURS OF THE INCIDENT

This form should be completed by the injured employee involved in the incident. Describe only the facts for which you have personal knowledge. If you have no knowledge of a particular question, write "no knowledge".

Company _____

Exact Location of Incident/Accident _____

Name of Injured Employee _____

Date of Incident/Accident _____ Time _____ am pm

Date of this Statement _____ Time _____ am pm

Time your shift begins? Time _____ am pm Time your shift ends? Time _____ am pm

Name of Known Witnesses:

Name _____

Name _____

Name _____

Name _____

Your Immediate Supervisors Name _____

If not employed by Shaw E&I, enter name of company and phone number _____

Have you had a prior injury similar to this injury? _____

Was it while you were at work? _____

What date did the prior injury occur? _____

Stating Only Factual Information, Describe in Detail What Happened and Include Any Applicable Events Leading to the Incident/Accident.

_____

_____

_____

_____

_____

_____

_____

_____

I certify that, to the best of my knowledge, all of the above information is complete, accurate and factual. I acknowledge that the intentional falsification or altering of facts or making misleading statements may be grounds for disciplinary action.

_____ _____

Signature/Date Print Name

ATTACHMENT 6b
Employee Witness Statement
MUST BE COMPLETED WITHIN 24 HOURS OF THE INCIDENT

This form should be completed by every employee working in the crew of the injured employee and by every other employee with knowledge of events or circumstances involved in the incident. This information is being solicited from you so that the company can accurately assess the reported incident to avoid similar occurrences in the future. Describe only the facts for which you have personal knowledge. If you have no knowledge of the incident, write "no knowledge".

Company _____

Exact Location of Incident/Accident _____

Name of Injured Employee _____

Date of Incident/Accident _____ Time _____ am pm

Date of this Statement _____ Time _____ am pm

Time your shift begins? Time _____ am pm Ends _____ am pm

Witness Information:

Name _____

Home Phone No. _____

Home Address _____

County _____ Zip _____

Witness' Supervisor Name _____

If not employed by Shaw E&I, enter name of company _____

Company Phone Number _____

Did You See the Incident/Accident? _____

How Far From You (approx., in feet) Did the Incident/Accident Occur? _____

Stating Only Factual Information, Describe in Detail What Happened and Include Any Applicable Events Leading to the Incident/Accident.

_____

_____

_____

_____

_____

_____

_____

_____

I certify that, to the best of my knowledge, all of the above information is complete, accurate and factual. I acknowledge that the intentional falsification or altering of facts or making misleading statements may be grounds for disciplinary action.

_____ _____

Witness Signature/Date Print Name

ATTACHMENT 7

ACCIDENT REVIEW BOARD

DATE:	LOCATION:
BOARD MEMBERS:	
ACCIDENT DATE:	EMPLOYEE(S) INVOLVED IN INCIDENT:
INVESTIGATION COMPLETE: YES ☐ NO ☐	ACCIDENT CLASSIFICATION:

THE FOLLOWING INFORMATION MUST BE PROVIDED BY THE REVIEW BOARD FOR THIS INCIDENT (PRINT):

SUPERVISOR: _____ **PROJECT/LOCATION MGR.:** _____

POTENTIAL CAUSE OF ACCIDENT:

ACTION BY BOARD*:

* ALL ACTIONS BY THE ACCIDENT REVIEW BOARD ARE SUBJECT TO FINAL REVIEW BY THE HUMAN RESOURCES AND LEGAL DEPARTMENTS.

ACCEPTED: _____ (Employee Signature)	_____ (Supervisor Signature)
APPROVED: _____ (Project/Location Manager)	REJECTED FOR: _____ _____
APPROVED: _____ (Business Line Health and Safety Manager or Designee)	REJECTED FOR: _____ _____
APPROVED: _____ (Business Line Vice President)	REJECTED FOR: _____ _____

ATTACHMENT 8

INJURY/ILLNESS CLASSIFICATION GUIDELINES

First Aid Treatment – If the incident requires only the following types of treatment, consider it first aid. **Do Not** record the case if it involves only:

- Using non-prescription medications at non-prescription strength
- Administering tetanus immunizations
- Cleaning, flushing, or soaking wounds on the skin surface
- Using wound coverings such as bandages, Band-Aids™, gauze pads, etc., or using SteriStrips™ or butterfly bandages
- Using hot or cold therapy
- Using any totally non-rigid means of support, such as elastic bandages, wraps, non-rigid back belts, etc.
- Using temporary immobilization devices while transporting an accident victim (slings, neck collars, or back boards)
- Drilling a fingernail or toenail to relieve pressure, or draining fluids from blisters
- Using eye patches
- Using simple irrigation or a cotton swab to remove foreign bodies not embedded in or adhered to the eye
- Using irrigation, tweezers, cotton swab or other simple means to remove splinters or foreign material from areas other than the eye
- Using finger guards
- Using massages
- Drinking fluids to relieve heat stress

Medical Treatment – Includes managing and caring for a patient for the purpose of combating disease or disorder. The following are **not** considered medical treatments and are not recordable:

- Visits to a doctor or Licensed Health Care Professional (LHCP) solely for the purpose of observation or counseling
- Diagnostic procedures, including administering prescription medications that are used solely for diagnostic purposes
- Any procedure that can be labeled first aid (see above descriptions)

OSHA Recordable Injuries and Illnesses

Work related injuries and illnesses that result in the following should be recorded on the OSHA 300 Log:

- Death
- Loss of consciousness
- Days away from work
- Restricted work activity or job transfer
- Medical treatment beyond first aid.

You must also record any **work related** injury or illness that involves cancer, chronic irreversible disease, a fractured or cracked bone, or a punctured eardrum.

Additional Recordable Criteria

You must also record the following conditions when they are work related:

- Any needle stick injury or cut from a sharp object that is contaminated with another person's blood or other potentially infectious material
- Any case requiring an employee to be medically removed from a site under the requirements of an OSHA health standard
- Any Standard Threshold Shift (STS) in hearing (i.e., cases involving an average hearing loss of 10dB or more in either ear)
- Tuberculosis infection as evidenced by a positive skin test or diagnosis by a physician or other licensed health care professional after exposure to a known case of active tuberculosis.

ATTACHMENT 9B
MEDICAL FORMS

AUTHORIZATION FOR TREATMENT OF OCCUPATIONAL INJURY/ILLNESS

Employee Name: _____
Social Security #: _____ Injury: ☐ Illness: ☐
Job Title: _____ Incident Date: _____
Project/Location _____ Location of Accident/Exposure:
Telephone #: _____ _____
H&S Representative: _____
Body Part(s) Injured: _____
Describe in detail how incident occurred: _____
_____

TO TREATING PHYSICIAN:

In the case of occupational injury/illness, please examine the employee and render necessary conservative treatment directly related to the occupational injury/illness.

Light Duty Work:
It is the policy of our company to provide work assignments, whenever possible, for employees with physical activity restrictions resulting from an occupational injury/illness. If the employee will be subject to a restriction, please contact **Health Resources** before releasing the employee, so that a light duty assignment may be arranged.

Medically Unfit to Return to Work:
It is the policy of our company to assist employees unable to return to work, due to an injury/illness, in obtaining needed medical care and other available benefits. Medical findings are also used to help evaluate unsafe conditions that may have led to the incident. Please help us assist our employees by contacting **Health Resources** with your findings as soon as possible, preferably before the employee leaves your office, but not later than the close of business on the day of initial treatment.

Health Resources: Telephone: 1-800-350-4511 Fax: (800) 853-2641

Please Send Reports To Both of the Following: **Health Resources** 600 West Cummings Park, Suite 3400 Woburn, Massachusetts 01801 ***AND*** **The Shaw Group, Inc. Corporate Claims Department** 4171 Essen Lane Baton Rouge, LA 70809

Please Send Bills To: **The Shaw Group, Inc. Corporate Claims Department**
4171 Essen Lane
Baton Rouge, LA 70809

DOCTOR, Please provide:
Medical Diagnosis: _____
Treatment Provided: _____
_____
Recommended Work Limitation/Restriction: _____
Return Visit Needed: No ☐ Yes ☐ Date if Yes _____ First Aid Only ☐
Physician Name: _____ Physician Telephone: _____
Physician Signature: _____ Date: _____

YOU MUST CALL HEALTH RESOURCES FOR ALL OCCUPATIONAL INJURIES/ILLNESSES REQUIRING OUTSIDE MEDICAL TREATMENT: 1-800-350-4511.

FAX COMPLETED FORM TO HEALTH RESOURCES (800) 853-2641.

Send Bills to Shaw Corporate Claims Department

ATTACHMENT 9B
MEDICAL FORMS
AUTHORIZATION FOR RELEASE OF PROTECTED MEDICAL INFORMATION

Printed Name: _____ Date of Birth: _____

Address:_____

Social Security #:_____ Home Telephone:_____

Authority to Release Protected Health Information

I hereby authorize the release of medical information, identified in this authorization form, and provide such information to:

HEALTH RESOURCES
600 West Cummings Park, Suite 3400
Woburn, Massachusetts 01801
Phone: (800) 350-4511
Fax: (800) 853-2641

AND

The Shaw Group Inc.
4171 Essen Lane
Baton Rouge, Louisiana 70809
Phone: 225-932-2500
Fax: 225-932-2636

The Information To Be Released includes the following:

Complete health record	Discharge summary	Progress notes
History and physical exam	Consultation reports	X-ray films / images
Laboratory test results	X-ray & Image reports	Itemized bill
Diagnosis & treatment codes	Complete billing record	

Other, (specify)_____

Purpose of the Requested Disclosure of Protected Health Information

I am authorizing the release of my Protected Health Information.

Drug and/or Alcohol Abuse, and/or Psychiatric, and/or HIV/AIDS Records Release

I understand if my medical or billing record contains information in reference to, psychiatric care, sexually transmitted disease, hepatitis B or C testing, previous drug and/or alcohol abuse and/or other sensitive information, I agree to its release. ***Check One:*** ☐ **Yes** ☐ **No**

I understand if my medical or billing record contains information in reference to HIV/AIDS (Human Immunodeficiency Virus/Acquired Immunodeficiency Syndrome) testing and/or treatment I agree to its release. ***Check One:*** ☐ **Yes** ☐ **No**

Right to Revoke Authorization

Except to the extent that action has already been taken in reliance on this authorization, the authorization may be revoked at any time by submitting a written notice to ***The Corporate Claims Dept.*** **at** ***The Shaw Group Inc., 4171 Essen Lane, Baton Rouge, Louisiana 70809***. Unless revoked, this authorization will expire at which time completion of treatment for the injury or illness has been accomplished.

Re-disclosure

I understand the information disclosed by this authorization may be subject to re-disclosure by the recipient and no longer be protected by the Health Insurance Portability and Accountability Act of 1996.

Signature of Patient or Personal Representative Who May Request Disclosure

I understand that I do not have to sign this authorization. However, if health care services are being provided to me for the purpose of providing information to a third-party (e.g. fitness-for-work test), I understand that services may be denied if I do not authorize the release of information related to such health care services to the third-party. I can inspect or copy the protected health information to be used or disclosed. **I hereby release and discharge** ***The Shaw Group Inc*** **of any liability and the undersigned will hold** ***The Shaw Group Inc*** **harmless for complying with this Authorization.**

Signature: _____ **Date:** _____

Description of relationship if not patient: _____

ATTACHMENT 9C
MEDICAL FORMS

RETURN-TO-WORK EXAMINATION FORM

Exam Date: _____ / _____ / _____ **Employee Name:** _____

Birth Date: _____ / _____ / _____ **Social Security #:** _____ - _____ - _____

Job Title: _____ **Sex:** ❑ Male ❑ Female

Examining Provider: **Please complete this form and fax to Health Resources at (800) 853-2641. Please contact Health Resources at (800) 350-4511 to report status of employee post-treatment.**

DIAGNOSIS: _____

TREATMENT PLAN: _____

MEDICATIONS: _____

PHYSICAL THERAPY: _____

OTHER: _____

❑ May return to full duty work effective ___/___/___

❑ May return to limited duty from ___/___/___ to ___/___/___

❑ Unable to return to work from ___/___/___ to ___/___/___

WORK LIMITATIONS:

❑ Restricted lifting/pushing/pulling: maximum weight in lbs: _____ (company limits all lifting to ≤ 60 lbs).

❑ Work only with right/left hand.

❑ Restricted repetitive motion right/left hand.

❑ Sitting job only.

❑ Restricted operation of moving equipment.

❑ Other: _____

FOLLOW-UP PLAN:

❑ Release from care.

❑ Schedule for follow-up appointment on ___/___/___.
Time _____ AM/PM

❑ Referral to _____
Appointment date ___/___/___ Time _____ AM/PM

Comments: _____

_____	_____	_____
Examiner's Name (*print*)	Examiner's Signature	Date

ATTACHMENT 10

HELP DESK / HOTLINE NOTIFICATION GUIDELINES

Any incident, as defined in the bulleted items below, requires corporate notification **as soon as practical but not longer than one hour after occurrence**, via the Health and Safety Help Desk / Hotline. This requirement is a corporate wide directive and applies to all Shaw Group companies, not just Shaw E&I. As such, the responsibility for whom makes this notification has purposefully not been defined. This is due to the various types of projects in which The Shaw Group performs activities. Some projects may only consist of three technicians at a site; others may involve multiple levels of site management and consist of 200+ employees. Therefore, the intent is for the supervisory/management person to communicate the notification requirements to his/her employees and make the appropriate determination as to how the notification takes place.

Immediate Corporate Notification via Help Desk: 1-866-299-3445

- Illness and/or injury (doctors cases and above);
- Property damage (dollar amount greater than $2,500);
- Automobile accidents (All);
- Criminal activity (i.e. bomb threat, theft);
- Natural disaster (i.e. earthquakes, flood, storm damage, hurricanes);
- Explosion and/or fires (that results in property damage greater then $2,500 or result in injury);
- Environmental spills/releases (incidents that requires regulatory notification or have an offsite impact);
- Regulatory visit (i.e. OSHA, EPA, DEQ, MSHA, etc.);
- Fatalities

Note:

- Help Desk / Hotline notification is in addition to the requirement to inform Health Resources of all incidents requiring off-site medical attention by calling 1-800-350-4511. This call should be made **prior** to transporting the employee such that they can coordinate physicians' services prior to arrival of the employee to the medical facility.

- As stated above, the notification requirements are a corporate directive and apply to the entire Shaw Group. . Accordingly, Shaw E&I managers/supervisors should use sound judgment as it pertains to the two bulleted items that have been highlighted above. Although they may not be desired events, some Environmental spills/releases that occur may not be an uncommon situation at a particular site. In addition, there may be projects in which the EPA or some other regulatory agency visits on some normal frequency. Events such as these, which would typically be unusual at a construction or fabrication site, are not so unusual to some of our environmental projects. As such, a notification to the helpdesk would not be required.

PROCEDURE

Subject: **ACCIDENT PREVENTION PROGRAM: MANAGEMENT SAFETY INSPECTIONS**

1.0 PURPOSE AND SUMMARY

This procedure establishes the requirement for management safety inspections of project and office locations. These inspections are an integral part of the overall accident prevention program and help to demonstrate management's commitment to safety. Key requirements of this procedure include:

- Project managers are required to conduct one inspection per month and ensure that at least one other inspection is conducted during the month;
- Office managers are required to conduct an office safety inspection once every six months.
- Completed inspection reports are given to the project/office health and safety representative for review. A copy of the completed report will then be forwarded to the respective business line health and safety manager.

2.0 TABLE OF CONTENTS

1.0 Purpose and Summary
2.0 Table of Contents
3.0 Responsibility Matrix
  3.1 Procedure Responsibility
  3.2 Action/Approval Responsibilities
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    4.1.1 Management Site Visits
    4.1.2 Project Managers
    4.1.3 Office Managers
    4.1.4 Project Supervisors
    4.1.5 Health and Safety Representative
  4.2 Workshops
5.0 Exception Provisions
6.0 Cross References
7.0 Attachments

3.0 RESPONSIBILITY MATRIX

3.1 Procedure Responsibility

The Director of Health and Safety is responsible for the issuance, revision, and maintenance of this procedure.

3.2 Action/Approval Responsibilities

The Responsibility Matrix is Attachment 1.

4.0 TEXT

Inspections of project and office locations by managers, supervisors, and the health and safety staff are critical factors in a comprehensive accident prevention program. Management safety inspections help demonstrate management's commitment to safety and verify that proper work practices are in use. These inspections are also used to verify the existence of safe work conditions and regulatory compliance. All employees are afforded the opportunity to participate in the inspection process via the safety interview process.

4.1 Safety Inspections and Documentation

Safety inspections are required by various tiers of the management structure. The objective is for operation managers to visibly demonstrate their concern for safety in the workplace by direct contact with employees while in the workplace. Each inspection is to be documented on the appropriate Safety Inspection Report (Attachment 2 or 3).

The primary responsibilities of the inspector include:

- Interviewing employees with regard to health and safety issues and how they might be corrected;
- Observing and correcting unsafe conditions and acts; and
- Verifying that corrective actions have been assigned to a responsible employee and implemented.

Positive safety observations and safety issues not specifically addressed in the Safety Inspection Report can be documented on the last page of the report. A list of all corrective action items will be maintained showing the corrective action, responsible person, and the date action is to be completed. Completed reports are to be given to the project/office health and safety representative, then forwarded to the respective business line health and safety manager.

4.1.1 Management Site Visits

Each senior manager is encouraged to make an informal safety inspection and review previously conducted inspection reports, during each site visit, to demonstrate their commitment to safety and reinforce the responsibilities of project management. Findings during this informal inspection are to be brought to the attention of the project manager so that corrective action can be initiated.

4.1.2 Project Managers

All project managers are required to complete at least one safety inspection per month and ensure that at least one other safety inspection per month is conducted. In the event that the project manager is not present at the project site during the month, this responsibility may be delegated to the project supervisor.

4.1.3 Office Managers

Office managers are required to conduct an office safety inspection once every six months. Managers are encouraged to conduct more frequent inspections if the office location is being remodeled or if new space is being occupied that was not previously inspected.

4.1.4 Project Supervisors

Project supervisors are expected to inspect their projects monthly and ensure that corrective actions are implemented. Dependent upon project manager participation, project supervisors may also be required to conduct an additional monthly inspection. The requirement to conduct these inspections cannot be delegated.

4.1.5 Health and Safety Representative

Health and safety representatives must continually observe activities and correct unsafe acts/conditions as soon as reasonably possible. They are also required to review each Safety Inspection Report completed at their location to ensure that corrective actions are implemented. Once this review is complete, they will forward the reports to the appropriate business line health and safety manager.

4.2 Workshops

Health and safety representatives will present workshops and/or conduct joint inspections to help managers and supervisors develop their inspection skills.

5.0 EXCEPTION PROVISIONS

Variances and exceptions may be requested pursuant to the provisions of Procedure HS013, Health and Safety Procedure Variances.

6.0 CROSS REFERENCES

HS013 Health and Safety Procedure Variances

7.0 ATTACHMENTS

1. Responsibility Matrix
2. Project Safety Inspection Report
3. Office Safety Inspection Report

ATTACHMENT 1

ACCIDENT PREVENTION PROGRAM : MANAGEMENT SAFETY INSPECTIONS RESPONSIBILITY MATRIX

Action	Procedure Section	*Responsible Party*				
		Senior Managers	Project/ Office Manager	Project Supervisors	Health and Safety Representative	Director of Health and Safety
Issue, Revise, and Maintain Procedure	3.1					X
Conduct Informal Safety Inspections and Review Previously Completed Reports	4.1.1	X				
Conduct Safety Inspections	4.1.2 4.1.3 4.1.4		X	X		
Give Completed Reports to Health and Safety Representative	4.1.2 4.1.3 4.1.4		X	X		
Review Reports and Forward to Health and Safety Manager	4.1.5				X	
Conduct Inspection Workshops	4.2				X	

ATTACHMENT 2

PROJECT SAFETY INSPECTION REPORT

PROJECT _____ **DATE** _____

BUSINESS LINE: _____ PROJECT NAME/NUMBER:
PROGRAM MANAGER: _____ PROJECT MANAGER:
GENERAL PROJECT DESCRIPTION:
SITE ACTIVITIES AT TIME OF INSPECTION:

INTERVIEWED EMPLOYEE:
SAFETY ISSUE:
CORRECTIVE ACTION:

ASSIGNED TO: _____ FOLLOW-UP DATE:
CORRECTION VERIFIED BY: _____ DATE:

INTERVIEWED EMPLOYEE:
SAFETY ISSUE:
CORRECTIVE ACTION:

ASSIGNED TO: _____ FOLLOW-UP DATE:
CORRECTION VERIFIED BY: _____ DATE:

INSPECTION COMPLETED BY: _____ DATE:

HEALTH AND SAFETY REVIEW BY: _____ DATE:

PROJECT SAFETY INSPECTION REPORT

PROJECT _____ **DATE** _____

	YES	NO	N/A
FIRST AID			
1. Are first aid kit locations identified and accessible?			
2. Are emergency eye wash/safety showers available and inspected monthly?			
3. Are first aid kits inspected weekly?			
4. Is a qualified first aid/CPR provider on site?			
PERSONAL PROTECTIVE EQUIPMENT			
1. Have levels of personnel protection been established?			
2. Are respirators decontaminated, inspected, and stored according to standard procedures?			
3. Have employees been fit-tested?			
4. Is defective personal protective equipment tagged and taken out of service?			
5. Does compressed breathing air meet CGA Grade "D" minimum?			
6. Are there sufficient sizes and quantities of protective equipment?			
7. At a minimum, are employees utilizing safety glasses, hard hats, and steel toe boots?			
FIRE PREVENTION			
1. Are employees smoking only in designated outdoor areas?			
2. Are fire lanes established and maintained?			
3. Are flammable liquid dispensing systems bonded?			
4. Are approved safety cans available for storage of flammable liquids?			
5. Has the local fire department been contacted?			
6. Are fire extinguishers available and inspected monthly?			
7. Are flammables and combustibles properly stored?			
8. Are flammable storage cabinets available and used when needed?			
AIR MONITORING			
1. Is required air monitoring being conducted?			
2. Are air monitoring instruments calibrated daily?			
3. Are air monitoring logs up to date?			
4. Are instrument user manuals available?			
5. Are instruments being maintained?			
6. Are employees notified of personal sampling results within 5 days of receipt?			
WELDING AND CUTTING			
1. Are fire extinguishers present at welding and cutting operations?			
2. Are confined spaces evaluated prior to and during cutting and welding operations?			
3. Have Hot Work Permits been completed?			
4. Are proper helmets, goggles, aprons, and gloves available for welding and cutting operations?			
5. Are welding machines properly grounded?			
6. Are oxygen and fuel gas cylinders stored a minimum of 20 feet apart?			
7. Are only trained personnel permitted to operate welding and cutting equipment?			
8. Are gas cylinders transported in a secured vertical position with caps in place?			
HAND AND POWER TOOLS			
1. Are defective hand and power tools tagged and taken out of service?			
2. Is eye protection available and used when operating power tools?			

PROJECT SAFETY INSPECTION REPORT

PROJECT _____ **DATE** _____

	YES	NO	N/A
3. Are guards and safety devices in place on power tools?			
4. Are power tools inspected before each use?			
5. Are nonsparking tools available when necessary?			
6. Is the correct tool being used for the job?			
MOTOR VEHICLES			
1. Are vehicles regularly inspected?			
2. Are personnel licensed for the vehicles they operate?			
3. Are unsafe vehicles tagged and reported to supervision?			
4. Is vehicles safety equipment operating properly?			
5. Are loads secure?			
6. Are vehicle occupants using safety belts?			
7. Are current insurance cards and blank accident report forms located in vehicles?			
EMERGENCY PLANS			
1. Are emergency telephone numbers posted?			
2. Have emergency escape routes been designated?			
3. Are employees familiar with the emergency signal?			
4. Has the emergency route to the hospital been established and posted?			
5. Is a vehicle on site that can transport injured employees to the hospital?			
MATERIALS HANDLING			
1. Are materials stacked and stored to prevent sliding or collapsing?			
2. Are tripping hazards identified?			
3. Are semi-trailers chocked?			
4. Are fixed jacks used under semi-trailers?			
5. Are riders prohibited on materials handling equipment?			
6. Are approved manlifts provided for the lifting of personnel?			
7. Are personnel in manlifts wearing approved fall protection devices?			
FIRE PROTECTION			
1. Has a fire alarm system been established?			
2. Do employees know the location and use of all fire extinguishers?			
3. Are fire extinguisher locations posted?			
4. Are combustible materials segregated from open flames?			
5. Have fire extinguishers been professionally inspected during the last year?			
6. Are fire extinguishers visually inspected monthly?			
ELECTRICAL			
1. Is electrical equipment and wiring properly guarded and maintained in good condition?			
2. Are extension cords kept out of wet areas?			
3. Is damaged electrical equipment tagged and taken out of service?			
4. Have underground electrical lines been identified by proper authorities?			
5. Has a lockout/tagout system been established?			
6. Are GFCIs being used on all temporary electrical systems and as needed?			
7. Are extension cords being inspected daily (i.e., group pin in place, no unapproved splices)?			
8. Are warning signs exhibited on high voltage equipment (250V or greater)?			

PROJECT SAFETY INSPECTION REPORT

PROJECT _____ **DATE** _____

	YES	NO	N/A
9. Is adequate distance maintained from overhead electrical lines?			
10. Are switches, circuit breakers, and switchboards installed in wet locations enclosed in weatherproof enclosures?			
CRANES AND RIGGING			
1. Are cranes inspected daily prior to use?			
2. Are crane swing areas barricaded or demarked?			
3. Is all rigging equipment tagged with an identification number and rated capacity?			
4. Is rigging equipment inspection documented?			
5. Are slings, chains, and rigging inspected before each use?			
6. Are damaged slings, chains, and rigging tagged and taken out of service?			
7. Are slings padded or protected from sharp corners?			
8. Do employees keep clear of suspended loads?			
9. Are rated load capacities and special hazard warnings posted on crane?			
10. Are the records of annual crane inspection available?			
11. Has accessible areas within the swing radius of the rear of the crane been barricaded?			
12. Do crane operators have required training/certification?			
COMPRESSED GAS CYLINDERS			
1. Are breathing air cylinders charged only to prescribed pressures?			
2. Are like cylinders segregated and stored in well ventilated areas?			
3. Is smoking prohibited in cylinder storage areas?			
4. Are cylinders stored secure and upright?			
5. Are cylinders protected from snow, rain, etc.?			
6. Are cylinder caps in place before cylinders are moved?			
7. Are fuel gas and oxygen cylinders stored a minimum of 20 feet apart?			
8. Are propane cylinders stored and used only outside of buildings?			
SCAFFOLDING			
1. Is scaffolding placed on a flat, firm surface?			
2. Are scaffold planks free of mud, ice, grease, etc.?			
3. Is scaffolding inspected before each use?			
4. Are defective scaffold parts taken out of service?			
5. Have employees completed scaffold user training?			
6. On scaffolds where platforms are overlapped, is planking overlapped a minimum of 12 inches?			
7. Does scaffold planking extend over end supports between 6 to 18 inches (dependent upon platform length)?			
8. Are employees restricted from working on scaffolds during storms and high winds?			
9. Are all pins in place and wheels locked?			
10. Is required perimeter guarding (top rail, mid rail, and toe board) present?			
11. Has a competent person been designated to oversee scaffold construction?			
12. Are employees prohibited from moving mobile scaffold horizontally while employees are on them?			
13. Are all scaffold components manufactured by the same company?			
WALKING AND WORKING SURFACES			
1. Are ladders regularly inspected?			
2. Are access ways, stairways, ramps, and ladders clean of ice, mud, snow, or debris?			

PROJECT SAFETY INSPECTION REPORT

PROJECT _____ **DATE** _____

	YES	NO	N/A
3. Are ladders being used in a safe manner?			
4. Are ladders kept out of passageways, doors, or driveways?			
5. Are broken or damaged ladders tagged and taken out of service?			
6. Are metal ladders prohibited in electrical service?			
7. Are stairways and floor openings guarded?			
8. Are safety feet installed on straight and extension ladders?			
9. Is general housekeeping being maintained?			
10. Are ladders tied off?			
11. Are handrails and side rails installed along the unprotected sides of stairways having 4 or more risers or rising more than 30 inches?			
SITE SAFETY PLAN			
1. Is a site safety plan available on site or accessible to all employees?			
2. Does the safety plan accurately reflect site conditions and tasks?			
3. Have potential hazards been described to employees on site?			
4. Is there a designated safety official on site?			
5. Have all employees signed the safety plan acknowledgment form?			
SITE POSTERS			
1. Are the following posters displayed in a prominent and accessible area?			
A. Minimum Wage			
B. OSHA Job Protection			
C. Equal Employment Opportunity			
2. Are all required state-specific posters displayed?			
SITE CONTROL			
1. Are work zones clearly marked?			
2. Are support trailers located to minimize exposure from a potential release?			
3. Are support trailers accessible for approach by emergency vehicles?			
4. Is the site properly secured during and after work hours?			
5. Is an exclusion zone sign-in/sign-out log maintained?			
6. Are only employees with current training and physicals permitted in exclusion zone?			
HEAVY EQUIPMENT			
1. Is heavy equipment inspected as prescribed by the manufacturer?			
2. Is defective heavy equipment tagged and taken out of service?			
3. Are project roads and structures inspected for load capacities and proper clearances?			
4. Is heavy equipment shut down for fueling and maintenance?			
5. Are backup alarms installed and working on mobile equipment?			
6. Have qualified equipment operators been designated?			
7. Are riders prohibited on heavy equipment?			
8. Are guards and safety appliances in place and used?			
9. Are operators using the "three point" system when mounting/dismounting equipment?			
EXCAVATION			

PROJECT SAFETY INSPECTION REPORT

PROJECT _____ **DATE** _____

	YES	NO	N/A
1. Has a "competent person" been designated to oversee excavation activities?			
2. Prior to opening excavations, are utilities located and marked?			
3. Has a professional engineer evaluated all excavations greater than 20 feet deep?			
4. Is there rescue equipment on site and accessible to the excavation area?			
5. Is excavated material placed a minimum of 24 inches from the excavation?			
6. Are the sides of excavations sloped or shored to prevent cave ins?			
7. Have excavations greater than 4 feet deep been monitored for hazardous atmospheres (i.e., LEL/O_2 deficiency)?			
8. Are ladders or ramps used in excavations over 4 feet deep?			
9. Are means of egress available so as to require no more than 25 feet of lateral travel?			
10. Are barriers, i.e., guardrails or fences, placed around excavations near pedestrian or vehicle thoroughfares?			
11. Is excavation inspected daily by competent persons and documented?			
CONFINED SPACES			
1. Have employees been trained in the hazards of confined spaces?			
2. Are confined space permits posted at entrance to confined space?			
3. Is a copy of the confined space entry procedure available?			
4. Has a rescue plan been established?			
5. Is an entry supervisor present at each permit-required entry?			
6. Are required extraction/fall protection devices being used?			
DECONTAMINATION			
1. Are decontamination stations set up on site?			
2. Is decontamination water properly contained and disposed of?			
3. Are all pieces of equipment inspected for proper decontamination before leaving the site?			
4. Are shin/metatarsal guards being used during power washing activities?			
HAZARD COMMUNICATION			
1. Is there a copy of the HAZCOM procedure on site?			
2. Are there MSDSs for required materials/chemicals present on site?			
3. Are all containers properly labeled, as to content, hazard?			
4. Have employees been trained in accordance with the HAZCOM procedure?			
5. Do employees (including subcontractors) know and understand the effects of exposure from the chemicals on site?			
6. Have all personnel signed the HAZCOM acknowledgment form?			
7. Is there an updated list of chemicals maintained on site?			
TRAINING			
1. Are tailgate safety meetings being conducted daily?			
2. Are current training/medical records maintained on site?			
DOCUMENTATION			
1. Is an OSHA 300 Log maintained on site and posted during February 1, to April 30,?			
2. Are accident report forms available?			
3. Is a copy of health and safety policy and procedures available on site?			

PROJECT SAFETY INSPECTION REPORT

PROJECT _____ **DATE** _____

ALL NEGATIVE RESPONSES	CORRECTIVE ACTION	ASSIGNED TO	DATE ASSIGNED	DATE COMPLETED	VERIFIED BY

DESCRIBE POSITIVE SAFETY OBSERVATIONS

ATTACHMENT 3

OFFICE SAFETY INSPECTION REPORT

OFFICE _____ **DATE** _____

DATE: _____ OFFICE NAME:
OFFICE MANAGER:
AREA(S) OF OFFICE INSPECTED:

INTERVIEWED EMPLOYEE:
SAFETY ISSUE:
CORRECTIVE ACTION:

ASSIGNED TO: _____ FOLLOW-UP DATE:
CORRECTION VERIFIED BY: _____ DATE:

INTERVIEWED EMPLOYEE:
SAFETY ISSUE:
CORRECTIVE ACTION:

ASSIGNED TO: _____ FOLLOW-UP DATE:
CORRECTION VERIFIED BY: _____ DATE:

INSPECTION COMPLETED BY: _____ DATE:

HEALTH AND SAFETY REVIEW BY: _____ DATE:

OFFICE SAFETY INSPECTION REPORT

OFFICE _____ **DATE** _____

	YES	NO	N/A
FIRST AID			
1. Are first aid kits accessible and identified?			
2. Are emergency eye wash/safety showers available where needed and inspected?			
3. Are first aid kits inspected weekly?			
FIRE PREVENTION			
1. Are employees smoking only in designated outdoor areas?			
2. Are fire lanes/evacuation routes established and maintained?			
3. Are approved safety cans/cabinets available for storage of flammable liquids?			
4. Are fire exits clearly identified and unobstructed?			
FURNITURE AND EQUIPMENT			
1. Are desks, file cabinets, etc. arranged so that drawers do not open into aisles or walkways?			
2. Are desk and file drawers closed after use?			
3. Is weight distributed in file cabinets so that upper drawer contents does not create a top-heavy condition?			
4. Are cabinets, bookcases, and shelves secured to prevent their falling over?			
5. Are faulty desks, chairs, or other office equipment repaired or taken out of service?			
6. Is adequate and sufficient lighting provided in all work areas?			
7. Are paper cutter blades in fully down and locked position when not in use?			
8. Are work stations arranged to be comfortable without unnecessary strains on backs, arms, necks, etc.?			
9. Do machines with exposed moving parts have appropriate guards?			
AISLES AND FLOORS			
1. Is aisle clearance adequate for two-way traffic and for unobstructed access to all parts of the office and building?			
2. Does office arrangement allow easy egress under emergency conditions?			
3. Are wastebaskets, briefcases, or other objects placed where they are not a tripping hazard?			
4. Are floors clear of pencils, bottles, and other loose objects?			
5. Are tripping hazards from electrical cords, phone outlets, or other protrusions on the floor prevented by arrangement of furniture or other means?			
6. Are floors free of loose tiles and projections that can create a tripping hazard?			
7. Is carpeting in good condition and not badly worn or torn?			
HAND AND POWER TOOLS			
1. Are defective hand and power tools tagged and taken out of service?			
2. Is eye protection available and used when operating power tools?			
3. Are guards and safety devices in place on power tools?			
4. Are power tools inspected before each use?			
5. Is the correct tool being used for the job?			
6. Do knife blades have guards when not in use?			
MOTOR VEHICLES			
1. Are vehicles regularly inspected?			
2. Are personnel licensed for the vehicles they operate?			
3. Are unsafe vehicles reported to supervision?			

OFFICE SAFETY INSPECTION REPORT

OFFICE _____ **DATE** _____

	YES	NO	N/A
4. Is safety equipment on vehicles?			
5. Are loads secure on vehicles?			
6. Are vehicle occupants using safety belts?			
7. Are current insurance cards and blank accident report forms located in vehicles?			
EMERGENCY PLANS			
1. Are emergency telephone numbers posted?			
2. Have emergency escape routes been designated?			
3. Are employees familiar with the emergency signal?			
4. Has an emergency route to the hospital been established and posted?			
MATERIALS HANDLING			
1. Are materials stacked and stored to prevent sliding or collapsing?			
2. Are flammables and combustibles stored in approved containers?			
3. Are tripping hazards identified?			
4. Are riders prohibited on material handling equipment?			
FIRE PROTECTION			
1. Has a fire alarm system been established?			
2. Do employees know the location and use of all fire extinguishers?			
3. Are fire extinguisher locations marked?			
4. Have fire extinguishers been professionally inspected during the last year?			
5. Are fire extinguishers visually inspected monthly?			
6. Is there an operating fire detection system?			
ELECTRICAL			
1. Are extension cords kept out of wet areas?			
2. Are certified electricians used for electrical work?			
3. Are GFCIs being used as needed?			
4. Are extension cords not being used in lieu of permanent wiring?			
5. Are warning signs exhibited on high voltage equipment (250V or greater)?			
6. Are switches, circuit breakers, and switchboards installed in wet locations enclosed in weatherproof enclosures?			
7. Are electric fans protected with guards of not over one-half inch mesh, which prevents fingers getting inside guard?			
8. Are cords, panels, receptacles, and plugs in good condition?			
9. Are multi-outlet strips not plugged into other multi-outlet strips?			
10. Are extension cords not plugged into other extension cords?			
11. Are circuit breakers or fuse panels properly labeled, kept closed, and accessible?			
12. Are extension cords arranged so that they are not placed over radiators, steam pipes, through doorways, or under carpets?			
13. Do space heaters have automatic shut-offs that will actuate if the heater tips over?			
14. Are space heaters UL listed and plugged directly into a wall receptacle?			
15. Are space heaters located at least 3 feet from combustible material?			
WALKING AND WORKING SURFACES			
1. Are cords, cables, and other items not placed in walkways?			
2. Are ladders regularly inspected?			
3. Are access ways, stairways, ramps, and ladders clean of ice, mud, snow, or debris?			

OFFICE SAFETY INSPECTION REPORT

OFFICE _____ **DATE** _____

	YES	NO	N/A
4. Are ladders being used in a safe manner?			
5. Are ladders kept out of passageways, doors, or driveways?			
6. Are broken or damaged ladders tagged and taken out of service?			
7. Are metal ladders prohibited in electrical service?			
8. Are stairways and floor openings guarded?			
9. Are safety feet installed on straight and extension ladders?			
10. Are employees walking instead of running?			
11. Are handrails and side rails installed along the unprotected sides of stairways having 4 or more risers or rising more than 30 inches?			
12. Are there torn, loose, or curled carpets?			
HOUSEKEEPING			
1. Is good housekeeping maintained?			
2. Are paper and materials stored properly?			
3. Are cleaning fluids used only in small quantities and stored in closed containers that are kept in well-ventilated areas?			
4. If cleaning fluids are flammable, are they not used near a flame or an open heating element?			
5. Are wastebaskets emptied on a daily basis?			
SITE POSTERS			
1. Are the following posters displayed in a prominent and accessible area?			
A. Minimum Wage			
B. OSHA Job Protection			
C. Equal Employment Opportunity			
2. Are all required state-specific posters displayed?			
HAZARD COMMUNICATION			
1. Is the written HAZCOM program available?			
2. Is there a MSDS FOR EACH HAZARDOUS CHEMICAL present in the office?			
3. Are all containers properly labeled, as to content, hazard?			
4. Have employees been trained on chemical hazards?			
5. Have all employees signed the HAZCOM acknowledgment form?			
6. Is there a list of chemicals maintained on site?			
DOCUMENTATION			
1. Is an OSHA 300 Log maintained and posted during February1, to April 30?			
2. Are accident report forms available?			
3. Is a copy of health and safety policy and procedures available?			

OFFICE SAFETY INSPECTION REPORT

OFFICE _____ **DATE** _____

ALL NEGATIVE RESPONSES	CORRECTIVE ACTION	ASSIGNED TO	DATE ASSIGNED	DATE COMPLETED	VERIFIED BY

DESCRIBE POSITIVE SAFETY OBSERVATIONS

APPENDIX C
ACTIVITY HAZARD ANALYSES

ACTIVITY HAZARD ANALYSIS FOR MOBILIZATION/DEMOBILIZATION				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Mobilization/ Demobilization	Slips, Trips, Falls	• Clear walkways work areas of equipment, tools, vegetation, excavated material and debris • Mark, identify, or barricade other obstructions • Halt exterior work in high winds, severe weather	Body harnesses/ lanyard (elevated platforms above 6 ft.)	
	Struck By/ Against Heavy Equipment	• Wear reflective warning vests when exposed to vehicular traffic • Isolate equipment swing areas • Make eye contact with operators before approaching equipment • Understand and review hand signals	Warning vests, hard hat, safety glasses, steel toe work boots	
	Electrical Shock	• De-energize or shut off utility lines at their source before work begins • Use double insulated or properly grounded electric power-operated tools • Maintain tools in a safe condition • Provide an equipment-grounding conductor program or employ ground-fault circuit interrupters • Use qualified electricians to hook up electrical circuits • Inspect all extension cords daily for structural integrity, ground continuity, and damaged insulation • Cover or elevate electric wire or flexible cord passing through work areas to protect from damage • Keep all plugs and receptacles out of water • Use approved water-proof, weather-proof type if exposure to moisture is likely • Inspect all electrical power circuits prior to commencing work • Label all electrical boxes > 200 volts with circuit voltage • Follow Lockout-Tagout procedures in accordance w/ Shaw Health and Safety Procedure HS315.	Lockout/Tagout Devises	Voltage Meter or Tic Tracer

ACTIVITY HAZARD ANALYSIS FOR MOBILIZATION/DEMOBILIZATION				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Mobilization/ Demobilization (continued)	Sharp Objects	• Wear cut resistant work gloves when lacerations or other injury may be caused by sharp edges or objects • Maintain all hand and power tools in a safe condition • Keep guards in place during use	Leather gloves	
	Handling Heavy Objects	• Observe proper lifting techniques • Obey sensible lifting limits (60 lb. maximum per person manual lifting) • Use mechanical lifting equipment (hand carts, trucks) to move large, awkward loads • Avoid carrying heavy objects above shoulder level • Avoid actions/activities that contribute to overexertion • Warm up muscles before engaging in manual lifting activities • Review lifting posture/techniques regularly at safety meeting		
	Awkward Motion and Position Strains and Sprains	• Maintain a safe stance and body position operating pressurized equipment • Avoid rushing Take breaks to reduce repetitive strain. • Avoid awkward positions and intense stretching. • Alternate hands periodically. • Do not overexert yourself in any way. • To not provide a forced-grip for prolonged periods of time. • Stretch hands and arms every 30 to 60 minutes		
	High Noise Levels	• Use hearing protection when exposed to excessive noise levels (greater than 85 dBA over an 8-hour work period) • Assess noise level with sound level meter if possibility exists to exceed 85 db A TWA	Hearing protection	

ACTIVITY HAZARD ANALYSIS FOR MOBILIZATION/DEMOBILIZATION				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Mobilization/ Demobilization (continued)	High/Low Ambient Temperature	• Monitor for Heat/Cold stress in accordance with Shaw Health and Safety Procedures HS400, HS401 • Provide fluids to prevent worker dehydration • Follow work/rest schedule in the HASP	Insulated Clothing (subject to ambient temperature)	Meteorological Equipment
EQUIPMENT REQUIRED		**INSPECTION REQUIREMENTS**	**TRAINING REQUIREMENTS**	
• Excavator and/or dozer • Power clearing tools (brush saws, weed wakers)		• Daily equipment inspections as per manufacturers requirements • Inspect all safety equipment (fire extinguishers, first aid kits and eye washes)	• Review AHA with all task personnel • Review the Health and Safety Plan • Review any potential site contaminants • Review operations/safety manuals for all equipment utilized • Review potential hazardous plants and insects/animals	

ACTIVITY HAZARD ANALYSIS FOR UXO CLEARANCE				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
UXO cLEARANCE	Handling Heavy Objects	• Observe proper lifting techniques • Obey sensible lifting limits (60 lb. maximum per person manual lifting) • Warm up muscles before engaging in manual lifting activities • Avoid actions/activities that contribute to over exertion • Review lifting posture/techniques regularly at safety meetings		
	Insect/ Animal Bites	• Review injury potential with workers • Avoid insect nests areas, habitats outside work areas • Emphasize the “Buddy System” where such injury potential exists • Use insect repellant to protect against sting injuries • Wear PPE and tape joints to keep insects away from the skin • Use protective insect repellents containing 25% DEET to prevent insect bites • Check limbs/body for insects/ insect bites during decontamination and/or shower	Tyvek coveralls, duct tape bottom of coveralls to boots in areas with possible insect/animal contact	

ACTIVITY HAZARD ANALYSIS FOR UXO CLEARANCE				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
UXO Clearance (continued)	Slips, Trips, Falls	• Clear walkways, work areas of equipment and tools • Mark, identify, or barricade other obstructions		
	Contact Dermatitis/ Poison Ivy	• Wear long sleeve shirts / trousers to avoid skin contact with plants or other skin irritants • Identify and review poisonous plants with workers • Avoid unnecessary clearing of plant/vegetation areas • Cover vegetation with plastic (visqueen) where survey position raises exposure potential • Apply protective cream/lotion to exposed skin to prevent poison ivy or similar reactions • Identify workers who are known to contract poison ivy	Tyvek coveralls, duct tape bottom of coveralls to boots if possibility of contact with poisonous plants	
	Adverse weather conditions:	• Monitor weather forecast • Shut down operations should severe weather conditions exist		
	Inhalation and Contact with Hazardous Substances (Sub-surface survey activities)	• Provide workers proper skin, eye and respiratory protection based on the exposure hazards present • Review hazardous properties of site contaminants with workers before operations begin if known • Monitor breathing zone air to determine levels of contaminants • Dampen soil using light water spray to prevent fugitive dust emissions • Establish an up-wind evacuation area • Hand dig around any unidentified debris, drums and cylinders.	Modified Level D	H_2S meter and LEL/O_2

ACTIVITY HAZARD ANALYSIS FOR UXO CLEARANCE				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**

EQUIPMENT REQUIRED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
• Fire Extinguisher	• Daily equipment inspections as per manufacturers requirements • Inspect all safety equipment (fire extinguishers, first aid kits and eye washes)	• Review AHA with all task personnel • Review the Health and Safety Plan • Review operations/safety manuals for all equipment utilized • Review potential hazardous plants and insects/animals

ACTIVITY HAZARD ANALYSIS FOR CLEARING AND GRUBBING				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Clearing and Grubbing	Struck By/ Against Heavy Equipment	• Isolate equipment swing areas • Make eye contact with operators before approaching equipment • Understand and review hand signals	Hard hat, safety glasses and steel toe work boots	
	Slips, Trips, Falls	• Clear walkways, work areas of equipment, tools, vegetation, and debris • Clean mud and grease from your boots before mounting equipment. Watch for slippery/unstable ground when dismounting equipment • Exit equipment slowly and maintain three point contact		
	Handling Heavy Objects	• Observe proper lifting techniques • Use Dozer or Track hoe to move logs and brush		
	Eye Injuries	• Wear face shield, goggles when operating powered clearing / grubbing equipment	Face shield, goggles	
	Sharp Objects	• Wear cut resistant work gloves when the possibility of lacerations or other injury may be caused by sharp edges or objects • Maintain all hand and power tools in a safe condition • Keep guards in place during use • Close doors, windows on heavy equipment to prevent injuries from tree branches and other vegetation	Leather gloves	

ACTIVITY HAZARD ANALYSIS FOR CLEARING AND GRUBBING				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Clearing and Grubbing (continued)	Insect/ Snake Bites	• Review injury potential and types of snakes with workers • Avoid insect nests areas, likely habitats of snakes outside work areas • Emphasize The Buddy System where such injury potential exists • Use insect repellant, wear PPE to protect against sting/bite injuries	Tyvek® coveralls (duct tape bottom of coveralls to boots)	
	Contact Dermatitis	• Wear PPE to avoid skin contact with contaminated soil, plants, or other skin irritants • Identify and review poisonous plants with workers • Apply protective cream/lotion to exposed skin to prevent poison ivy or similar reactions	Tyvek® coveralls, (duct tape bottom of coveralls to boots)	
	Operations of power clearing tools (brush saws, weed whackers)	• Wear eye, face, hand & hearing protection when operating power clearing equipment • Shut-off / idle power tools walking between work areas • Store flammable liquids in well ventilated areas, away from work areas • Shut off equipment during re-fueling • Prohibit smoking while operating clearing equipment • Provide ABC (or equivalent) fire extinguishers for all work	Full-face shield, goggles, leather work gloves, chainsaw chaps, (if applicable), hearing protection, and steel toe work boots	
	High Noise Levels	• Use hearing protection when exposed to excessive noise levels (greater than 85 dBA over an 8-hour work period)	Hearing protection	

ACTIVITY HAZARD ANALYSIS FOR CLEARING AND GRUBBING				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Clearing and Grubbing (continued)	High/Low Ambient Temperature	• Monitor for Heat/Cold stress in accordance with Shaw Health and Safety Procedures # HS400, HS401 • Provide fluids to prevent worker dehydration		
	Inhalation and Contact with Hazardous Substances (Grubbings)	• Provide workers proper skin, eye and respiratory protection based on the exposure hazards present • Review hazardous properties of site contaminants with workers before operations begin if known • Monitor breathing zone air to determine levels of contaminants • Dampen soil using light water spray to prevent fugitive dust emissions • Establish an up-wind evacuation area • Hand dig around any unidentified debris, drums and cylinders.	Modified Level D	Data RAM, PID, H_2S meter and LEL/O_2

EQUIPMENT REQUIRED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
• Excavator and/or dozer • Power clearing tools (brush saws, weed whackers)	• Daily equipment inspections as per manufacturers requirements • Inspect all safety equipment (fire extinguishers, first aid kits and eye washes)	• Review the Site Specific Health and Safety Plan and Addendum • Review AHA with all task personnel • Review operations/safety manuals for all equipment utilized • Review potential hazardous plants and insects/animals

ACTIVITY HAZARD ANALYSIS FOR SOIL AND DEBRIS SEGREGATION AND REMOVAL				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Soil and Debris Segregation and Removal	Underground/ Overhead Utilities	• Identify all utilities around the site before work commences • Cease work immediately if unknown utility markers are uncovered • Use manual excavation within 3 feet of known utilities • Utility clearance shall conform with 29 CFR 1926.955 (high voltage >700 kv) 15 feet phase to ground clearance; 31 feet phase to phase clearance		
	Excavation Wall Collapse	• Construct diversion ditches or dikes to prevent surface water from entering excavation • Provide good drainage of area adjacent to excavation • Collect ground water/rain water from excavation and dispose of properly • Store excavated material at least 2 feet from the edge of the excavation; prevent excessive loading of the excavation face • Provide sufficient stairs, ladders, or ramps when workers enter excavations over 4 feet in depth • Place ladders no more than 25 feet apart laterally • Treat excavations over 4 feet deep as confined spaces • Complete confined space permit entry procedure • Monitor atmosphere for flammable/toxic vapors, and oxygen deficiency • Slope, bench, shore, or sheet excavations over 5 feet deep if worker entry is required • Assign a competent person to inspect, decide soil classification, proper sloping, the correct shoring, or sheeting • Inspect excavations (when personnel entry is required) daily, any time conditions change • Provide at least two means of exit for personnel working in excavations	Hard hat, safety glasses, steel toe work boots	

ACTIVITY HAZARD ANALYSIS FOR SOIL AND DEBRIS SEGREGATION AND REMOVAL				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Soil and Debris Segregation and Removal (continued)	Struck By/ Against Heavy Equipment	• Wear reflective warning vests when exposed to vehicular traffic • Isolate equipment swing areas • Make eye contact with operators before approaching equipment • Understand and review hand signals	Warning vests, hard hat, safety glasses, steel toe work boots	
	Handling Heavy Objects	• Observe proper lifting techniques • Obey sensible lifting limits (60 lb. maximum per person manual lifting) • Use mechanical lifting equipment (hand carts, trucks) to move large, awkward loads		
	Sharp Objects	• Wear cut resistant work gloves when the possibility of lacerations or other injury may be caused by sharp edges or objects • Maintain all hand and power tools in a safe condition • Keep guards in place during use	Leather gloves	
	Slips, Trips, Falls	• Clear walkways, work areas of equipment, vegetation, excavated material, tools, and debris • Mark, identify, or barricade other obstructions • Evaluate fall hazards above 4 ft.; use fall protection equipment (harness/lanyard), standard guardrails or other fall protection systems when working on elevated platforms above 6 ft. • Use heavy duty industrial (type IA) ladders • Install and inspect scaffolds according to manufacturers requirements • Only trained operators are permitted to use aerial lifts • Tie-off all straight/extension ladders or manually hold by co-worker at base • Anchorage points for fall arrest systems must support at least 5,400 pounds for each worker		

ACTIVITY HAZARD ANALYSIS FOR SOIL AND DEBRIS SEGREGATION AND REMOVAL				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Soil and Debris Segregation and Removal (continued)	High Noise Levels	• Use hearing protection when exposed to excessive noise levels (greater than 85 dB A over an 8-hour work period) • Assess noise level with sound level meter if possibility exists that level may exceed 85dB A TWA	Hearing protection	
	Inhalation, Ingestion and Contact with Hazardous Substances	• Provide workers proper skin, eye and respiratory protection based on the exposure hazards present • Avoid unnecessary contact with contaminated materials • Review hazardous properties of site contaminants with workers before operations begin • Monitor breathing zone air to determine levels of contaminants • Dampen soil using light water spray to prevent fugitive dust emissions • Cover stockpiled soil with plastic sheeting to prevent fugitive dust emissions • Conduct air monitoring / sampling to determine exposure levels	Modified Level D	Data RAM, PID and H_2S meter
	High/Low Ambient Temperature	• Monitor for Heat/Cold stress in accordance with Shaw Health and Safety Procedures HS400, HS401 • Provide fluids to prevent worker dehydration		

EQUIPMENT REQUIRED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
• Back hoe, Excavators • Dump trucks, other vehicles • Barriers/cones/flagging tape • Miscellaneous tools • Data RAM, PID and H_2S Meter	• Daily equipment inspections as per manufacturers requirements and forms • Inspection of all emergency equipment (i.e.: first aid kits, fire extinguishers, eye wash stations)	• Review AHA with all task personnel • Review the Health and Safety Plan • Review operations/safety manuals for all equipment utilized

ACTIVITY HAZARD ANALYSIS FOR SAMPLING				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Sampling	Handling Heavy Objects	• Observe proper lifting techniques • Obey sensible lifting limits (60 lb. maximum per person manual lifting) • XUse mechanical lifting equipment (hand carts, trucks) to move large, awkward loads		
	Slips, Trips, Falls	• Clear walkways, work areas of equipment, tools, vegetation, excavated material, and debris • XMark, identify, or barricade other obstructions		
	Spills	• Wear splash protection as necessary to prevent dermal contact • Ensure spill cleanup equipment/material on hand/ready for use (i.e.: Baking soda to neutralize and sample preservative spills) • Cleanup spills immediately	Full face shield	
	Inhalation and Contact with Site Contaminants	• Provide workers proper skin and eye protection based on the exposure hazards present. • Review hazardous properties of site contaminants with workers before operations begin. • Dampen work area to control dust	Level D with sample gloves.	H_2S meter
	High/Low Ambient Temperature	• Monitor for heat/cold stress in accordance with Health and Safety Procedure HS400 and HS401 • Provide fluids to prevent worker dehydration		Meteorological Equipment

EQUIPMENT TO BE USED	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
• Sample containers • Coolers • Hand tools • H_2S meter	• Excavation inspections/permits • Inspection of all emergency equipment (i.e.: first aid kits, fire extinguishers)	• Review AHA with all task personnel • Review the Site-specific Health and Safety Plan. • Review site specific chemical hazards. • Review all applicable Shaw safety procedures.

ACTIVITY HAZARD ANALYSIS FOR MATERIAL LOADOUT				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Material Loadout	Struck By/ Against Heavy Equipment	• Wear reflective warning vests when exposed to vehicular traffic • Obey posted speed limits • Isolate equipment swing areas • Make eye contact with operators before approaching equipment • Understand and review hand signals • Exit equipment slowly and maintain three point contact • Report minor incidents to site supervision • Park equipment in areas where operator can see clearly to dismount equipment • Step away from equipment when bucket adjustments are made • Do not attempt verbal communication in high noise backgrounds • Follow hand signals of ground workers for equipment manipulation when placing/loading equipment into loader bucket.	Warning vests, hard hat, safety glasses, steel toe work boots	
	Slips, Trips, Falls	• Clear walk ways, work areas of equipment, tools and debris • Mark, identify, or barricade other obstructions • Clean mud from boots before climbing on equipment		
	Sharp Objects	• Wear cut resistant work gloves when the possibility of lacerations or other injury may be caused by sharp edges or objects • Maintain all hand and power tools in a safe condition • Keep guards in place during use	Leather gloves	

ACTIVITY HAZARD ANALYSIS FOR MATERIAL LOADOUT				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Material Loadout (continued)	High Noise Levels	• Use hearing protection when exposed to excessive noise levels (greater than 85 dBA over an 8-hour work period) • Assess noise level with sound level meter if possibility exists that level may exceed 85dBA TWA	Hearing protection	
	Caught In/ Between Moving Parts	• Identify and understand parts of equipment which may cause crushing, pinching, rotating or similar motions • Assure guards are in place to protect from these parts of equipment during operation • Wear proper work gloves when the possibility of pinching, or other injury may be caused by moving/ handling large or heavy objects • Keep all guards in place during use • Avoid moving hydraulic, dump or loading equipment • Isolate/block pinch points to limit motion when inserting pins, closing tackles, ect.		
	Walking on machine tracks	• Avoid walking on machine tracks whenever possible; clean tracks for safe walking/working surfaces • Observe track surfaces when walking, move cautiously on uneven, slippery surfaces • Avoid sudden awkward motions (pulling/jerking fuel hoses)		
	Handling Heavy Objects	• Observe proper lifting techniques • Obey sensible lifting limits (60 lb. Maximum per person manual lifting) • Use mechanical lifting equipment (hand carts, trucks) to move large, awkward loads • Avid carrying heavy objects above shoulder level • Avoid actions/activities that contribute to overexertion • Warm up muscles before engaging in manual lifting. • Review lifting posture/techniques regularly at safety meetings		

ACTIVITY HAZARD ANALYSIS FOR MATERIAL LOADOUT				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Material Loadout (continued)	Defective Vehicles	• Inspect all trucks before loading • Do not load soil or equipment into defective equipment		
	Roadways	• Ensure that the roadways on the route to the landfill or final destination are designed to handle the weight of the vehicles and allow HAZMAT materials		
	Horseplay	• Prohibit horseplay on all project sites • Review rules about horseplay with subcontractor supervisors and workers • Remind workers not to respond/participate in horseplay stared by others		
	High/Low Ambient Temperature	• Provide fluids to prevent worker dehydration • Monitor for Heat/Cold stress in accordance with Shaw Health and Safety Procedures # HS400, HS401		

EQUIPMENT REQUIREMENTS	INSPECTION REQUIREMENTS	TRAINING REQUIREMENTS
• Heavy Equipment	• Emergency Safety Equipment-Shower, Eyewashes, Fire Extinguishers, First aid supplies • Daily Equipment Inspection	• Review AHA with all task personnel • Review the Health and Safety Plan

ACTIVITY HAZARD ANALYSIS FOR EQUIPMENT DECONTAMINATION				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Equipment Decontamination	Slips, Trips, Falls	• Clear walkways, work areas of equipment, vegetation, tools and debris • Mark, identify, or barricade other obstructions • Clean heavy objects of oil/grease or other slippery contamination before attempting to lift/remove • Wear gloves with grip improving surfaces for handling large, slippery objects • Clean up spills or water accumulation in walkways		
	Struck by/Against Heavy Equipment, Protruding Objects	• Wear reflective warning vests when exposed to vehicular traffic • Isolate equipment swing areas • Make eye contact with operators before approaching equipment • Understand and review hand signals • Step away from equipment when bucket adjustments are made • Do not attempt verbal communication in high noise backgrounds	Warning vests hard hat safety glasses, goggles and face shield, steel toe work boots	
	Inhalation and Contact with Hazardous Substances, & Splashes	• Provide workers proper skin, eye and respiratory protection based on the exposure hazards present • Review hazardous properties of site contaminants with workers before operations begin • Wear hard hats, safety glasses with side shields, or goggles with splash shields and steel-toe safety boots	PVC rain suit or poly-coated Tyvek® coverall, inner latex sample gloves, outer nitrile gloves, latex boot covers, goggles/ face shield when in contact with liquid contamination.	

ACTIVITY HAZARD ANALYSIS FOR EQUIPMENT DECONTAMINATION				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Equipment Decontamination (continued)	Burns	• Wear proper gloves, face shield/safety goggles, shin and toe guards, and splash suits to protect workers from skin burns and injury when operating laser (high pressure washers) • Tape gloves to PPE sleeves to lessen the possibility of hat water entering gloves • Use hand tools to loosen connections and position body to avoid pressure discharge • Wear shin and toe guards to protect from burns, lacerations and similar injuries	Goggles and face shield, shin and toe guards	
	Handling Heavy Objects	• Observe proper lifting techniques • Obey sensible lifting limits (60 lb. maximum per person manual lifting) • Use mechanical lifting equipment (hand carts, trucks) to move large, awkward loads • Avoid actions/activities that contribute to overexertion • Warm up muscles before engaging in manual lifting activities • Review lifting posture/techniques regularly at safety meetings		

ACTIVITY HAZARD ANALYSIS FOR EQUIPMENT DECONTAMINATION				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Equipment Decontamination (continued)	Sharp Objects/ Cuts and Punctures	• Wear cut resistant work gloves when the possibility of lacerations or other injury may be caused by sharp edges/objects or working with hand tools • Maintain all hand and power tools in a safe condition • Keep guards in place during use • Guard or pad metal edges of objects frequently used (access panels, etc.) or manipulated/bypassed during maintenance • Position heavy objects to avoid manipulation while cleaning • Get assistance and dry glove surfaces to improve grip during object manipulation while cleaning	Leather gloves	
	High Noise Levels	• Use hearing protection when exposed to excessive noise levels (greater than 85 dBA over an 8-hour work period)	Hearing Protection	
	Repetitive Strains	• Rotate job tasks on high vibration equipment • Report equipment that produces high vibration for inspection and maintenance • Wear vibration reducing gloves		
	Strains and Sprains	• Maintain a safe stance and body position operating pressurized equipment • Avoid rushing		
	High/Low Ambient Temperature	• Monitor for heat/cold stress in accordance with Health and Safety Procedure HS400 and HS401 • Provide fluids to prevent worker dehydration		Meteorological Equipment

ACTIVITY HAZARD ANALYSIS FOR EQUIPMENT DECONTAMINATION				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
EQUIPMENT REQUIRED		**INSPECTION REQUIREMENTS**	**TRAINING REQUIREMENTS**	
• Pressure washer		• Daily equipment inspections as per manufacturers requirements • Inspection of all emergency equipment (i.e.: first aid kits, fire extinguishers)	• Review AHA with all task personnel • Review Site Specific Health and Safety Plan • Review operations/safety manuals for all equipment utilized	

ACTIVITY HAZARD ANALYSIS FOR SITE RESTORATION				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
Site Restoration	Struck by/ Against Heavy Equipment, Protruding Objects	• Wear reflective warning vests when exposed to vehicular traffic • Avoid equipment swing areas • Make eye contact with operators before approaching equipment • Wear hard hats, safety glasses with side shields, or splash/face shields and goggles, and steel-toe safety boots at all times • Understand and review hand signals	Warning vests, hard hat, safety glasses, steel toe work boots	
	Slips, Trips, Falls	• Clear, walkways of equipment, tools, debris, other materials • Mark, identify, or barricade other obstructions		
	High Noise Levels	• Use hearing protection when exposed to excessive noise levels (greater than 85 dBA over an 8-hour work period)	Hearing Protection	
	Handling Heavy Objects	• Observe proper lifting techniques • Obey sensible lifting limits (60 lb. per person for manual lifting) • Use mechanical lifting equipment (hand carts, trucks) to move large, awkward loads		
	High/Low Ambient Temperature	• Monitor for heat/cold stress in accordance with Health and Safety Procedure HS400 and HS401 • Provide fluids to prevent worker dehydration		Meteorological Equipment

ACTIVITY HAZARD ANALYSIS FOR SITE RESTORATION				
Task Breakdown	**Potential Hazards**	**Critical Safety Practices**	**Personal Protective Clothing and Equipment**	**Monitoring Devices**
EQUIPMENT TO BE USED		**INSPECTION REQUIREMENTS**	**TRAINING REQUIREMENTS**	
• Bob Cat or Forklift for moving bulky loads		• Equipment inspections • Inspection of all emergency equipment (i.e.: first aid kits, fire extinguishers)	• Review AHA with all task personnel • Review the Site-specific Health and Safety Plan • Review operation manuals for equipment • Review all applicable Shaw safety procedures.	

APPENDIX D
DIRECTIONS TO THE HOSPITAL
TO BE INSERTED BY THE SITE SAFETY OFFICER

APPENDIX E
HEALTH AND SAFETY PLAN
AMENDMENT DOCUMENTATION FORM

Site Specific Health & Safety Plan
Amendment Documentation

Project Name: **Project No.**

Amendment No. **Date:**

The Amendment Addresses the Following Sections:

Task(s) Amendment Affects:.

Reason For Amendment:

Amendment:

Completed by: **Approved by:**

APPENDIX F
HURRICANE PREPAREDNESS PLAN

(HPP)

HURRICANE PREPAREDNESS PLAN
VIEQUES ISLAND, PUERTO RICO

Prepared for:

DEPARTMENT OF THE NAVY
Atlantic Division
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Reviewed:
October 2005
Revised:
March 2007

Project No. 116701

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ATTACHMENT B EMERGENCY PHONE NUMBERS

ATTACHMENT C HURRICANE TRACKING MAP

LIST OF ACRONYMS

COR	Condition of Readiness
FEMA	Federal Emergency Management Administration
HPP	Hurricane Preparedness Plan
mph	Miles per Hour
Vieques	Vieques Island
NOAA	National Oceanic and Atmospheric Administration
PPE	Personal Protective Equipment
ROICC	Resident Officer In Charge Of Construction
SSO	Site Safety Officer

1.0 INTRODUCTION

PURPOSE

This Hurricane Preparedness Plan (HPP) outlines the general responsibilities and actions to be taken in preparation for and response to a hurricane or hurricane warnings at the Vieques Island Project locations. All personnel should understand that predicting the occurrence and path of a hurricane is difficult, however the risk can be minimized and controlled by following the procedures in this plan.

SCOPE

This procedure is applicable to all site personnel, including subcontractors; temporary construction facilities; and remediation equipment present at Vieques.

DISCUSSION

This procedure provides information on how to protect personnel and property in the event of a hurricane. In the Vieques area, attention must be paid to all hurricanes, since there is no way to determine with 100 percent accuracy whether a hurricane will actually hit the area until a few hours before landfall.

The following table demonstrates that the accuracy of forecasting where a hurricane landfall will occur is very low more than 24 hours in advance of a storm.

Hours Before Landfall	Maximum Probability Values
72 Hours	10 Percent
48 Hours	13-18 Percent
36 Hours	20-25 Percent
24 Hours	35-45 Percent
12 Hours	60-70 Percent

2.0 DEFINITIONS

The following definitions apply to various terms used in this document.

Conditions of Readiness (COR):

Condition V - Destructive winds are possible at the Vieques site within 96 hours. Normal daily job-site cleanup and good housekeeping practices.

Condition IV - Destructive winds are possible at the Vieques site within 72 hours. Normal daily job-site cleanup and good housekeeping practices. Collect and store in piles or containers, scrap lumber, waste material, and rubbish for removal and disposal at the end of each work day. Maintain the construction site, including storage areas, free of accumulation of debris. Stack form lumber in neat piles less than 4 feet high. Remove all trash debris and other objects which could become missile hazards. Contact Resident Officer In Charge of Construction (ROICC) for Condition requirements, updates, and completion of required actions.

Condition III - Destructive winds are possible at the Vieques site within 48 hours. Maintain Condition IV requirements. Begin securing the job-site for and taking those actions necessary for Condition I that cannot be completed within 18 hours. Cease all routine activities that might interfere with securing operations. Begin collecting and stowing all gear and portable equipment. Make preparations for securing buildings. Review requirements pertaining to Condition II and continue action as necessary to attain Condition III readiness.

Condition II - Destructive winds are possible at the Vieques site within 36 hours. Curtail or cease routine activities until securing operations are complete. Reinforce or remove form work and scaffolding. Secure machinery, tools, equipment and materials, or remove from job site. Expend every effort to clear all missile hazards and loose equipment from the job-site. Contact ROICC for weather and COR updates and completion of required actions.

Condition I - Destructive winds are possible at the Vieques site within 24 hours. Perform and complete all remaining actions required for lower conditions of readiness. Secure the job-site and leave the premises.

Destructive Winds - Generally winds reaching or exceeding the force of a tropical storm ($\geq$ 39 miles per hour [mph] or 34 knots). Winds from any storm system (tropical or otherwise) that are determined to have the potential to cause property damage or personal injury that would warrant the a Condition IV alert.

Gale - Non-tropical windstorm with winds 38 to 63 mph (33 to 55 knots).

Hurricane Watch - An announcement for specific areas where a hurricane or an incipient hurricane poses a possible threat to a coastal area, generally within 36 hours.

Hurricane Warning - A warning that sustained winds of 74 mph (64 knots) or higher, associated with a hurricane are expected in a specified coastal area in 24 hours or less.

Hurricane - A tropical cyclone in which the maximum sustained surface wind is 74 mph (64 knots) or greater.

Missile Hazard - Any object that may become airborne during high winds.

Severe Weather - Any storm of tropical or non-tropical origin that has the capacity to produce destructive winds

Small Area Storms – Thunderstorms or tornadoes.

Small Area Storms Condition I - Destructive winds, heavy rain, lightening and hail are imminent within 1-hour.

Small Area Storms Condition II - Destructive winds, heavy rain, lightening and hail are expected within 6-hours.

Storm - Non-tropical windstorm with winds 38 to 63 mph (33 to 55 knots).

Storm Surge - An abnormal rise in sea level accompanying a hurricane or other intense storm, and whose height is the difference between the observed level of the sea surface and the level that would have occurred in the absence of the storm.

Storm Tide - The actual sea level resulting from the astronomical tide combined with the storm surge. This term is used interchangeably with "Hurricane Tide."

Tornado - Violent rotating columns of air with winds 115 to 288 mph (100 to 250 knots).

Tropical Depression - A tropical low pressure system in which the maximum sustained surface wind is 38 mph (33 knots) or less.

Tropical Storm - A tropical low pressure system in which the maximum surface wind ranges from 39 to 73 mph (34 to 63 knots) inclusive. This is the strength at which the National Hurricane Center applies a name to the storm.

Tropical Storm Watch - Tropical storm conditions pose a threat to a coastal area generally within 36 hours.

Tropical Storm Warning - A warning for tropical storm conditions with sustained winds within the range of 39 to 73 mph (34 to 63 knots) that are expected in a specified coastal area within 24 hours or less.

3.0 RESPONSIBILITIES

Project Manager – Bill Hughes

The Project Manager is responsible for ensuring that all adequate measures have been taken to prepare for hurricanes and to protect site personnel and property in the event of a hurricane. The Project Manager will ensure that ample resources are available to implement this plan and that all personnel are aware of this plan and their responsibilities.

Site Superintendent – Cano Hernandez

The Site Superintendent will communicate all hurricane information to site personnel and keep the site personnel continually informed of the measures to be taken. The Site Superintendent is responsible for the coordination and direction of site equipment shut-down and will oversee the preparation of site facilities for any imminent storm. The Site Superintendent will oversee the coordination of both pre- and post-storm operations and will ensure that the proper material, equipment, and supplies are utilized to implement this procedure.

Site Safety Officer – Cano Hernandez

The Site Safety Officer (SSO) will monitor weather information, including the National Weather Service probability values for landfall. The SSO will maintain the necessary emergency supplies, and will periodically tour the site to ensure that proper steps are being taken to protect site personnel and property. The SSO will develop the emergency contact list will be maintained in a site dedicated vehicle.

Note: When personnel identified in Section 3.0 leave the site, they are responsible for notifying the Site Superintendent or a designated back-up person. The back-up person will be instructed in their responsibilities in the event of a hurricane.

4.0 EMERGENCY OPERATING PROCEDURES

Condition V - Early Preparedness

The Site Superintendent will notify the Project Manager when a tropical storm has been named and/or any severe weather has the potential to produce destructive winds at the Vieques within 96 hours. This will initiate COR Condition V. This phase will continue until:

- The storm or condition is downgraded
- The storm track poses no threat to the site
- Condition IV begins.

During Condition V, the progress of the storm will be monitored and tracked. The Program Manager will be contacted at least twice daily for Condition Requirements updates and to inform him of completion of required actions for Condition V.

See Attachment A for the Hurricane Preparedness Responsibility Punch List - Condition V.

Condition IV - (Destructive winds are possible within 72 Hours)

This COR starts when severe weather is within 72 hours of posing a threat to the project location. The SSO will ensure that the following steps are taken:

- Monitor the storm and inform the Project Manager of its progress
- Check Personal Protective Equipment (PPE) supplies and equipment to determine if any shipments are required or if pending shipments should be advanced or postponed

During Condition IV, the progress of the storm will be continuously monitored and tracked. The Site Superintendent will instruct site personnel to begin general cleanup of all loose materials that may pose a hazard during high winds or rain. This will include removal of all debris, trash, and other items that may become missile hazards. All lumber will be stacked in neat piles less than 4 feet high. The Program Manager will be contacted at least twice daily for Condition Requirements updates and to inform him of completion of required actions for Condition IV.

The Site Superintendent will keep all site personnel advised of the status of the storm and site preparation activities. Due to the urgency and amount of work involved in preparing for a threatening storm, all construction operations that might interfere with securing operations, such as starting a major excavation, will cease.

The Site Superintendent will ensure that the following steps are taken:

- Fill fuel tanks in all equipment on-site
- Secure stockpiled material on-site
- Review requirements for Condition II with all site personnel
- Maintain condition IV requirements.

See Attachment A for the Hurricane Preparedness Responsibility Checklist - Condition IV.

Condition III - Tropical Storm Warning (Destructive winds are possible Within 48 Hours)

This COR starts when severe weather places the project site under a tropical storm warning. Condition III activities will also start if a threatening tropical storm is upgraded to a hurricane, or a severe storm approaching the Vieques site has generated destructive winds in other locations. The Project Manager, Site Superintendent and SSO will determine when to cease all operations based upon current weather conditions and/or as directed by the ROICC. If the storm or Condition is downgraded, the Project Manager and Site Superintendent will meet with the Program Manager and ROICC to decide if a downgrade of the COR is appropriate. Actions for Condition III will be maintained and the following shall also be completed:

- Machinery, tools, equipment, and materials will be secured or removed form the site
- Take actions to secure job-site necessary for Condition II that cannot be completed within 18 hours.

See Attachment A for the Hurricane Preparedness Responsibility Checklist - Condition III.

Condition II - Destructive Winds are anticipated <u>within 36 hours</u> or a Small Area Storm is anticipated <u>within 12 hours</u>.

Condition II begins when destructive winds are anticipated within 36 hours, a small area storm within 6 hours, and/or as directed by the ROICC. The Project Manager and Site Superintendent will determine when to demobilize from the site based upon weather conditions. During this phase the Site Superintendent will direct the following actions:

- Secure machinery, tools, equipment and materials or remove them from the job-site
- Conduct a roll call of personnel on-site and inform the SSO
- Notify personnel on leave of schedule changes
- Personnel needing to leave the project to attend to personal matters will notify their Site Superintendent immediately
- Heavy equipment will be secured according to the manufacturer's recommendations
- All small field equipment will be secured
- All visitors from the site are evacuated
- Make a final site walk-through to determine that the site is secure and clear all missile hazards from the job-site
- Inform the Project Manager that all personnel are being released from the site.

If the storm or Condition is downgraded, the Project Manager and Site Superintendent will meet to decide if a downgrade of the phase is necessary.

See Attachment A for the Hurricane Preparedness Responsibility Checklist - Condition II.

Condition I - Destructive winds are anticipated <u>within 24 hours</u> or a Small Area Storm is imminent <u>within 8 hours</u>.

- Complete all remaining actions required for lower conditions of readiness
- Secure job-site access and evacuate to safe refuge.

See Attachment A for the Hurricane Preparedness Responsibility Checklist - Condition I.

Resume Site Operations

The Project Manager will contact the Program Manager to determine when site operations will resume. Although the hurricane/severe weather has passed, hazards may still exist because of water damage, other hazardous conditions, dangers from electric shock, poisonous snakes, etc.

The Site Superintendent and SSO will conduct a damage survey with the Project Manager. Photographs of the storm damage at the site will be taken by the Site Superintendent or SSO. They will develop a prioritized recovery plan from the survey findings. Subsequently, all site personnel will be notified when it is safe to return to work. Required personnel and subcontractor expertise will be mobilized to the site to repair any damaged equipment.

See Attachment A for the Hurricane Preparedness Responsibility Checklist - Resume Site Operations.

5.0 DEBRIEFING

Following the return to work of site personnel, the Site Superintendent will conduct a debriefing with site personnel. The debriefing will accomplish the following objectives:

- Finalize a recovery plan
- Review the Hurricane Plan for effectiveness
- Suggest and agree on improvements to the plan
- Incorporate plan changes

When completed, the Project Manager and Site Superintendent will meet with site personnel to discuss any corrective actions or changes in this plan.

6.0 REFERENCES

The list of emergency telephone numbers is included as Attachment B. In addition, an example of a Hurricane Tracking Chart is presented in Attachment C.

The following references and sources of information may be consulted for additional guidance on hurricane preparedness and response.

- Disaster Planning Guide for Business and Industry, Federal Emergency Management Administration (FEMA).

- U.S. Department of Commerce; National Oceanic and Atmospheric Administration (NOAA)

ATTACHMENT A

HURRICANE PREPAREDNESS RESPONSIBILITY CHECKLISTS

HURRICANE PREPAREDNESS CHECKLIST

Condition V

Date/Time Entered Condition V:_____

Severe Weather/Tropical Storm: _____

Action Items

☐ Project Manager Notified

☐ Track of Storm Poses No Threat

☐ Storm or Condition is Downgraded

☐ Upgrade to Condition IV

Storm Location

Date/Time:_____ Date/Time:_____

Location/Coordinates:_____ Location/Coordinates:_____

Date/Time:_____ Date/Time:_____

Location/Coordinates:_____ Location/Coordinates:_____

Condition V Action Items Complete:_____ Date:_____

HURRICANE PREPAREDNESS CHECKLIST

Condition IV (Landfall within 72 hours)

Date/Time Entered Condition IV:_____

Action Items:

☐ Notify Project Manager

☐ Notify Project Superintendent

☐ Notify Site Personnel

☐ Assemble shift personnel to begin preparation

☐ Track storm on hurricane tracking map (Attachment C) (if applicable)

☐ Secure all heavy equipment located at the site in accordance with manufacturer's specifications. All equipment will be moved to a secured site location.

☐ All equipment fuel tanks will be filled.

☐ All subcontractors with equipment or supplies on-site will be notified to begin removal procedures

Condition IV Action Items Complete:_____ Date:_____

HURRICANE PREPAREDNESS CHECKLIST

Condition III (Landfall within 48 hours)

Date/Time Entered Condition III:_____

Action Items:

☐ Provide the status of the storm to site personnel on an hourly basis

☐ Take actions to secure job-site necessary for Condition I that cannot be accomplished in 18 hours

☐ Recheck all items on checklist IV to ensure they are complete (i.e. gas tanks are still filled)

See itemized equipment checklist (itemized list of equipment to be secured/removed and COR for action)

Condition III Action Items Complete:_____ Date:_____

HURRICANE PREPAREDNESS CHECKLIST

Itemized Equipment Checklist, Condition III.

Equipment	Secure in Place	Remove from Project Site

HURRICANE PREPAREDNESS CHECKLIST

Condition II

Date/Time Entered Condition II:_____

Action Items:

☐ Evacuate all visitors from the site

☐ Conduct a role call of site personnel and inform the Project Manager

☐ Check the status all incoming shipments of supplies and equipment

☐ Remove all unnecessary vehicles from the site

☐ Secure heavy equipment in accordance with manufacturer's specification

☐ Secure all valuable records and equipment

☐ Release personnel from the site

☐ Recheck all items on checklist IV and III to ensure they are complete (ie: gas tanks are still filled)

Condition II Action Items Complete:_____ Date:_____

HURRICANE PREPAREDNESS CHECKLIST

Condition I

Date/Time Entered Condition I:_____

Action Items:

☐ Complete all action items for lower conditions of readiness

☐ Secure job-site access and evacuate to safe refuge

Condition I Action Items Complete:_____ Date:_____

HURRICANE PREPAREDNESS CHECKLIST

Resume Site Operations

Date/Time Resume Site Operations:_____

Action Items:

- ☐ Conduct a damage survey
- ☐ Notify all site personnel when to return to work
- ☐ Develop a prioritized recovery plan
- ☐ Inspect electrical equipment before re-energizing to detect and repair damage
- ☐ Provide bottled water for drinking until normal drinking water is deemed safe to drink
- ☐ Remove storm debris from site
- ☐ Notify Program Manager of the resumption of site activities

Resume Site Operations Action Items Complete:_____ Date:_____

ATTACHMENT B

EMERGENCY PHONE NUMBERS

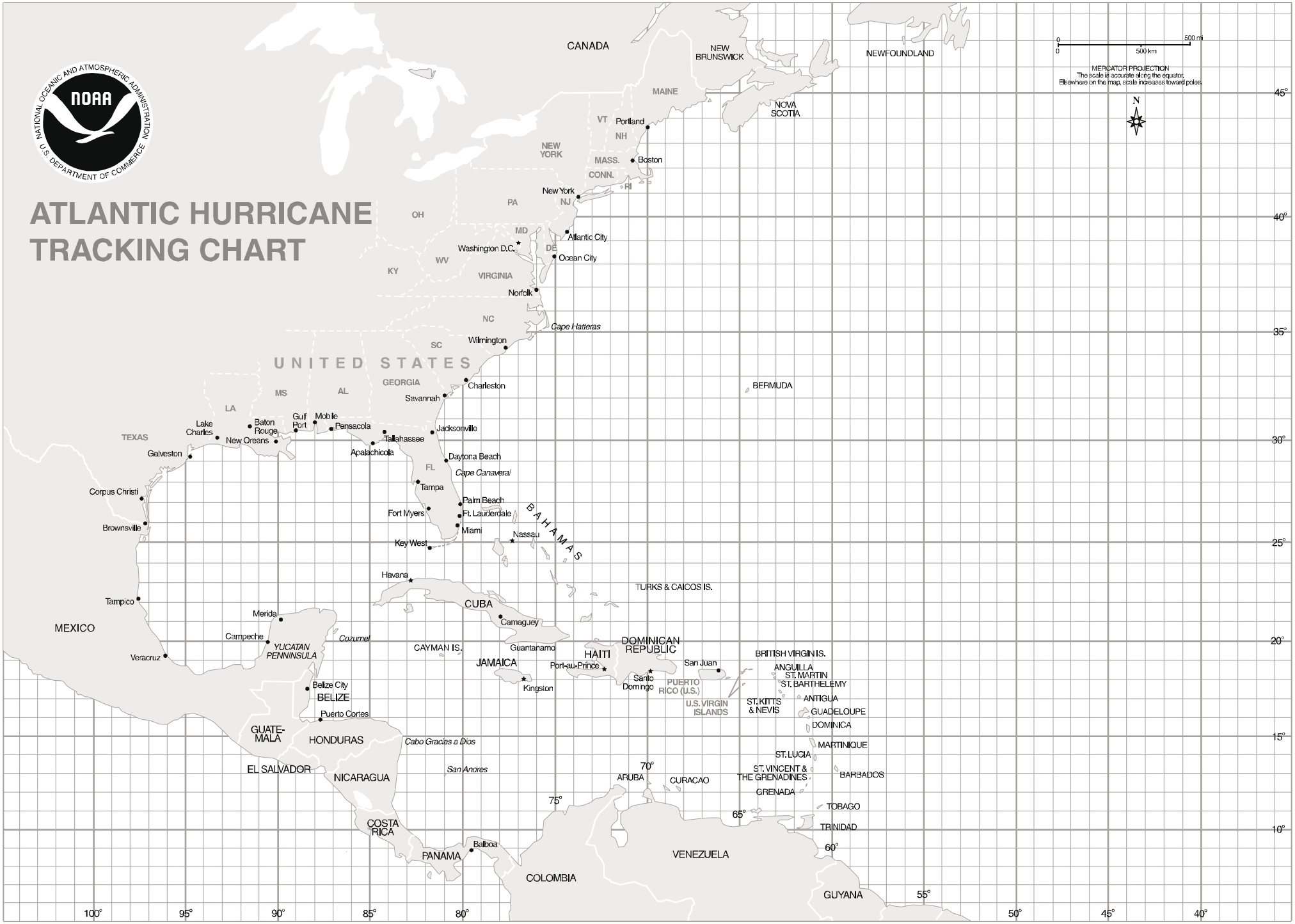
Emergency Telephone Numbers

Local Agencies – Ambulance Fire Police	 787-741-2111 787-741-2111 787-741-2020
Hospital Susanna Centro Hospital **Directions to the hospital are to be inserted into Appendix D by the SS/SSO**	 787-741-3282
Regional Poison Control Center	800-552-6337
Federal Agencies Agency for Toxic Substances and Disease Registry National Response Center	 (404) 639-0615 (24 hr.) 800-424-8802
ROICC Hector Nazario	 787-865-4066 ext 225
Shaw Personnel Project Manager – Bill Hughes Site Superintendent/Site Safety Officer – Cano Hernandez Program CIH – David Mummert, CIH Program Health & Safety Manger – Kym Edelman	 (757) 318-5140 (office) (757) 438-8498 (cellular) (757)544-7687 (cellular) (419) 425-6129 (office) (419) 348-1544 (cellular) 757-435-5384 (cellular)
Shaw Corporation (24 hour)	800-537-9540

ATTACHMENT C

HURRICANE TRACKING MAP

ATLANTIC HURRICANE TRACKING CHART



APPENDIX G
ACCIDENT PREVENTION PLAN

(APP)

ACCIDENT PREVENTION PLAN FOR
INTERIM REMOVAL ACTIONS AT SMWU-6, SWMU-7, AOC-J, AND AOC-R
WEST VIEQUES, PUERTO RICO

Prepared for:

DEPARTMENT OF THE NAVY
Atlantic Division
Naval Facilities Engineering Command
6506 Hampton Boulevard
Building A (South East Wing) 3rd Floor
Norfolk, VA 23508

Contract No. N62470-03-D-4402
Task Order 0015

Prepared by:

SHAW ENVIRONMENTAL, Inc.
500 E. Main Street
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Project No. 116701
August 2005
Revised March 2007

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LIST OF ACRONYMS

AHA	Activity Hazard Analysis
ANSI	American National Standards Institute
APP	Accident Prevention Plan
ASTM	American Society for Testing and Materials
CD	Compact Disc
CFR	Code of Federal Regulations
DoD	Department of Defense
EH&S	Environmental Health and Safety
EMR	Experience Modification Rate
EMS	Emergency Medical Services
EZ	Exclusion Zone
FSSI	Field Support Services, Inc.
HPP	Hurricane Preparedness Plan
HS	Health and Safety
HSM	Health and Safety Manager
MSDS	Material Safety Data Sheet
NIOSH	National Institute for Safety and Health
OSHA	Occupational Safety and Health Administration
PPE	Personal Protective Equipment
Shaw	Shaw Environmental, Inc.
SSHSP	Site Specific Health and Safety Plan
SS/SSO	Site Supervisor/Site Safety Officer
USACE	United States Army Corps of Engineers

1.0 BACKGROUND INFORMATION

The primary site tasks include the following:

- Site Mobilization/Demobilization;
- UXO Clearance;
- Clearing and Grubbing;
- Soil and Debris Segregation and Removal;
- Collection of Soil Samples;
- Material Soil Load-out and Disposal;
- Equipment Decontamination;
- Site Restoration.

Activity Hazard Analyses (AHA) have been prepared for the above activities and are included in Attachment C of the Site Specific Health and Safety Plan (SSHSP).

Shaw Accident Experience

Shaw Accident Experience YEAR	Experience Modification Rate EMR (Interstate)*	OSHA Recordable Incident Rate*
2004	0.50	1.07
2003	0.57	1.4
2002	0.62	1.7

CORPORATE POLICY

Subject: SAFETY

1.0 PURPOSE AND SUMMARY

It is the policy of Shaw to provide a safe and healthful workplace for all employees, subcontractors, and consultants in compliance with governmental requirements. Additionally, the requirements of our clients shall take precedence provided that their requirements exceed those of Shaw and governmental regulations.

We believe in two fundamental principles of safety: all accidents, injuries and occupational illnesses are preventable; and if an operation cannot be done safely, we will not do it. To put these principles into practice, every associate will receive the appropriate training, equipment, and other resources necessary to complete assigned tasks in a safe and efficient manner.

Safety, industrial hygiene and loss prevention are the direct responsibility of all members of management, who must create an environment in which everyone shares a concern for their own safety and the safety of their associates. Safety shall take precedence over expediency or short cuts. It is a condition of employment that all employees work safely and follow established safety rules and procedures. No individual(s) may pose a direct threat to the health and safety of other individuals in the workplace.

Managers must conduct their businesses in compliance with governmental safety regulations and company procedures. All Shaw health and safety procedures shall be implemented for all Shaw employees on all projects where Shaw is the subcontractor, or a joint venture partner. If Shaw is the prime contractor, Shaw procedures shall be applied to all Shaw and subcontractor personnel.

The implementation of effective safety and health practices is a key measure of managerial performance. Management, with the assistance of the internal health and safety professional staff, will conduct audits to assess the effectiveness of the safety program(s) in place, and to identify areas for improvement. All deficiencies shall be corrected promptly.

All injuries, occupational illnesses, vehicle accidents, and incidents with potential for injury or loss will be investigated. Appropriate corrective measures will be taken to prevent recurrence, and to continually improve the safety of our workplace.

3.0 RESPONSIBILITIES AND LINES OF AUTHORITY

Safety responsibilities, accountability and lines of authority are discussed in Section 2.0 of the SSHSP. The Project Manager (PM), Site Superintendent/Site Safety Officer (SSO/SS), and Health and Safety Manager (HSM) are responsible for formulating and enforcing health and safety requirements, and implementing the SSHSP.

4.0 SUBCONTRACTORS AND SUPPLIERS

Each subcontractor working on the project site will be required to adhere to the SSHSP and the requirements presented below.

4.1 SUBCONTRACTOR/SUPPLIER COORDINATION AND CONTROL

All subcontractors will be screened for safety performance and compliance with Federal Alcohol and Drug testing requirements prior to being issued any contract for site work. Subcontractors will comply with the requirements for site safety as outlined in Shaw's Health and Safety (HS) Procedure HS011 (Attachment B of the SSHSP). The SS will be responsible for the conduct and control of Shaw subcontractors.

4.2 SUBCONTRACTOR/SUPPLIER SAFETY RESPONSIBILITIES

All subcontractor employees are subject to the same training and medical surveillance requirements as Shaw personnel depending on job activity. All activities involving the potential for exposure to hazardous waste materials will require medical and training certification as mandated by 29 Code of Federal Regulations (CFR) 1910.120 and 29 CFR 1926.65. All subcontractor personnel will be required to sign in daily and be required to attend a daily meeting discussing operations and safety issues. All subcontractors involved in construction/remedial activities will complete a Subcontractor Pre-Job Safety Checklist prior to the start of work at the site. Subcontractors will submit AHAs for their work activities to the SS. The subcontractor reports directly to the Project Manager. All incidents involving subcontractor employees shall be reported to the SS and a copy of the subcontractor's injury/illness report shall be submitted to the SS within 24 hours.

Subcontractors are required to read and sign the SSHSP and comply with all requirements of this Accident Prevention Plan (APP). Contractors not in compliance will be immediately dismissed from the site.

Suppliers delivering various materials to the project site or providing equipment/ equipment maintenance will comply with all Naval Facility rules and regulations. Supplier personnel will not be permitted into contaminated areas unless training and medical surveillance is in accordance with 29 CFR 1910.120/1926.65. Contractors will not ride on tractors, forklifts or similar vehicles unless specific seats are provided. They will follow facility hot work rules if hot work is required for vehicle or equipment maintenance. Trucks will be loaded and unloaded in a safe and effective manner and materials will be stored safely in designated locations only. Associated packaging will be properly disposed of and litter will not permitted to be scattered or blown from truck beds. Operators of mobile equipment on site must observe all traffic rules such as speed limits and right-of-ways of pedestrians.

5.0 TRAINING

Outlines of the site safety orientation and training for site personnel, subcontractors and visitors are provided in Section 10.0 of the SSHSP.

5.1 MANDATORY TRAINING AND CERTIFICATIONS

Mandatory training and certifications are discussed in Section 10.0 of the SSHSP.

5.2 EMERGENCY RESPONSE TRAINING

All Shaw personnel who have completed the Shaw 40-hour HAZWOPER Training are qualified as emergency responders per 29 CFR 1910.120/1926.65 (e)(3)(iv). Site Specific Emergency Response Procedures will be reviewed with all site personnel as a part of site indoctrination.

5.3 SUPERVISORY AND EMPLOYEE SAFETY MEETINGS

The Shaw SS or SSO will conduct daily safety meetings at the start of each work shift for on-site personnel and will require subcontractors to follow similar meeting procedures or participate in the Shaw daily safety meetings. Daily safety meetings will comply with HS051 (Attachment B of the SSHSP).

6.0 HEALTH AND SAFETY INSPECTIONS

6.1 INSPECTIONS

The Shaw Project Manager and SS/SSO are required to conduct bi-monthly inspections of the sites using the Project Safety Inspection Report. The SS/SSO is responsible for conducting and preparing reports of daily safety inspections of work processes, site conditions, and equipment conditions and submitting them to HSM. The SS/SSO will discuss any necessary corrective actions with the SS and review new procedures. Copies of these reports are maintained on file at the project locations.

The Shaw HSM representative will periodically conduct site visits and perform Site Safety Assessments. These reports are kept on file at the Virginia Beach, Virginia, office and are tracked in a database for each Shaw Project Manager and SS/SSO, including the number of action items noted during the visit and written confirmation of the corrective actions for each item. These responses are compiled and provided to program management for review.

6.2 EXTERNAL INSPECTIONS/CERTIFICATIONS

Shaw does not anticipate, but may consider the use of outside sources, to provide safety inspections on an as necessary basis.

As required, safety equipment will comply with appropriate regulations of OSHA (Occupational Safety and Health Administration), NIOSH (National Institute for Occupational Safety and Health), ANSI (American National Standards Institute), ASTM (American Society for Testing and Materials), United States Coast Guard (USCG), or other recognized certification organizations.

7.0 SAFETY AND HEALTH EXPECTATIONS, INCENTIVE PROGRAMS, AND COMPLIANCE

Shaw considers safety the highest priority during work at a site containing potentially hazardous materials and has established a goal of **zero incidents** for all projects. All projects will be conducted in a manner that minimizes the probability of near misses, equipment/property damage or injury. Shaw will establish programs to recognize people and projects that demonstrate excellence in safety performance. Shaw will use safety observation programs to identify and correct unsafe acts and conditions. Safety awareness programs will be used to provide continuous training and development of good safety practices. Shaw site supervision will investigate all incidents to determine root causes and institute corrective actions to prevent recurrence. Shaw will provide and enforce safety rules to protect employees, subcontractors, clients and the public.

7.1 SHAW SAFETY INCENTIVE PROGRAMS:

A copy of the Shaw Safety Incentive Award Program will be available at each project. The Shaw Project Manager will develop a site-specific program for approval by the HSM and the LANTDIV program Manager within 10 days of project mobilization.

7.2 SHAW EMPLOYEE SAFETY RESPONSIBILITY REQUIREMENTS

Each employee is responsible for personal safety as well as the safety of others in the area and is expected to participate fully in the ***Safety Improvement Process***, particularly the Safety Observation Program. The employee will use all equipment provided in a safe and responsible manner as directed by the SS/SSO. All Shaw personnel will follow the policies set forth in the Shaw Health and Safety Procedures HS001-999 (Available on site on compact disc [CD]). Site personnel concerned with any aspect of health and safety shall bring it to the attention of the SS/SSO. If not satisfied, they should contact the HSM. All project personnel have the authority to stop work if in their judgment serious injury could result from continued activity. The SS shall be notified immediately if this becomes necessary. To protect the health and safety of all personnel, employees that knowingly disregard safety policies/procedures may be subject to disciplinary actions up to and including termination. Shaw Employee Safety Responsibility is fully detailed in HS010 Employee Safety and Health Work Rules (Attachment B of the SSHSP).

7.3 MANAGERS AND SUPERVISORS SAFETY ACCOUNTABILITY

It is the duty of the first line supervisor to motivate employees to adhere to Shaw's safety policy in each work situation. A first line supervisor for these purposes is defined as that person designated to give immediate on-site supervision to personnel involved in a task.

All supervisors shall have complete knowledge of the safe procedure for all jobs and tasks under their supervision, or when in doubt, shall seek assistance prior to initiating a task. This is the only acceptable manner in which to perform the task. If the task cannot be accomplished safely, it will not be attempted.

Supervisors will:

- Explain the safety procedure involved with a task to each employee and check frequently to see that the employee understands and works as instructed.
- Allocate sufficient time for the training and coaching of all employees to insure that everyone knows the correct procedure for safely accomplishing required tasks.
- Prevent new employees from performing any tasks until required training is completed.
- Immediately correct unsafe conditions that involved site employees or contractors.
- Ensure that the employees are outfitted with and wear personal protective equipment (PPE) as specified by this APP, SSHSP, other Shaw procedures or as directed by the HSM.
- Set a good safety example.
- Obtain the cooperation of employees and contractors.
- Provide a safe work environment for employees and contractors.
- Confirm contractor safety performance records have been verified prior to contract award and monitor contractor performance during operations.
- Report all accidents, near misses and property damage in accordance with the Incident Management and Reporting Procedure.
- Establish a safety culture, using the elements of the Shaw Safety Improvement process, which promotes awareness, encourages participation and recognizes excellence.

8.0 ACCIDENT REPORTING

8.1 EXPOSURE DATA (MAN-HOURS WORKED)

The Shaw's Environmental Health and Safety (EH&S) Manager tracks and maintains incident records as to Federal reporting requirement. Incident rates are reported monthly to the Shaw's EH&S Manager. Incident Rates and Workers Compensation losses are tracked for each business line. Program incident rates are reported monthly by the Program HSM.

8.2 ACCIDENT INVESTIGATIONS, REPORTS AND LOGS

The SS/SSO conducts accident/incident investigations. A report is completed by the SS/SSO must be submitted to the Shaw Baton Rouge, Louisiana Safety Department within 24 hours. All incident reporting forms are provided in HS020.

8.3 IMMEDIATE NOTIFICATION OF MAJOR INCIDENTS

Shaw will immediately notify the client of any major incident, including injury, fire, equipment/ property damage, and environmental incident. A full report will be provided within 24 hours. The following procedure will be followed in response to any major personal injury.

8.4 ACCIDENT RESPONSE

The nearest workers will immediately assist a person who shows signs of medical distress or who is involved in an accident. The work crew supervisor will be summoned.

The work crew supervisor will immediately make radio contact with the SS and/or SSO to alert him of a medical emergency situation. The work crew supervisor will advise the following information:

- Location of the victim at the work site
- Nature of the emergency
- Whether the victim is conscious
- Specific conditions contributing to the injury, if known.

9.0 MEDICAL SUPPORT

On-site Medical Support/Off-site Medical Arrangements will be outlined in Section 9.0 of the SSHSP.

10.0 PERSONAL PROTECTIVE EQUIPMENT

Protection levels provided in the SSHSP will be established for the site work activities based on the levels of site contaminants and the scope of work. Once on-site, results of air monitoring and visual inspection of the work activities may indicate the need for changes in these PPE level(s). Any significant change in the PPE level will be approved by the SS/SSO in consultation with the HSM. PPE selection criteria are outlined in HS600 and HS601 (Attachment B of the SSHSP).

All personnel using respiratory protection will be cleared by a physician for use of a respirator and will be fit-tested to assure they can achieve an acceptable fit. Physician clearance and results of fit testing will be documented as required by HS100 (Attachment B of the SSHSP).

11.0 PLANS REQUIRED BY THE SAFETY MANUAL

11.1 HAZARD COMMUNICATION PROGRAM

The Site-Specific Hazard Communication Program is included Section 4.2 of the SSHSP. Shaw Hazard Communication Program complies with 29 CFR 1926.59/1910.1200 and is outlined in HS060 (Attachment B of the SSHSP).

11.2 EMERGENCY RESPONSE PLANS

The Emergency Response and Contingency Plan is included in Section 9.0 of the SSHSP.

11.3 LAYOUT PLANS

Work zones are defined in Section 5.0 of the SSHSP.

11.4 RESPIRATORY PROTECTION PLAN

The primary objective of respiratory protection is to prevent employee exposure to atmospheric contamination. When engineering measures to control contamination are not feasible, or while they are being implemented, personal respiratory protective devices will be used.

The criteria for determining respirator need have been evaluated based on the site contaminants. Air monitoring will be conducted to confirm that respiratory protection levels are adequate. All respirator users will be OSHA trained in proper respirator use and maintenance. The SS and SSO will observe workers during respirator use for signs of stress. The SS, HSM, and SSO will also evaluate the implementation of the SSHSP, periodically, to determine its continued effectiveness with regard to respiratory protection. All persons assigned to use respirators will have medical clearance to do so.

11.5 CONTINGENCY PLAN FOR SEVERE WEATHER

Contingency plans for severe weather are included in Section 9.0 of SSHSP. A Hurricane Preparedness Plan (HPP) is located in Appendix F of the SSHSP.

11.6 ALCOHOL AND DRUG ABUSE PREVENTION PLAN

Shaw substance abuse procedures are outlined in Shaw HS101 - Drug, and Alcohol Testing.

12.0 CONTRACTOR INFORMATION TO MEET THE REQUIREMENTS OF THE MAJOR SECTIONS OF EM 385-1-1

In addition to this APP, Shaw has prepared a SSHSP to meet the major requirements of United States Army Corps of Engineers (USACE) Manual 385-1-1. Additional procedures for major requirements are provided in the Shaw Health and Safety Procedures Manual HS001-999 (Available on site on CD).

APPENDIX H
SAFETY PLAN ACKNOWLEDGEMENT

WORKER ACKNOWLEDGEMENT TO HEALTH-AND-SAFETY PLAN

.

I have been informed of, and will abide by, the procedures set forth in the SSHSP developed for SWMU-6, SWMU-7 and AOC-J Project. I have also been provided with an opportunity to read this SSHSP and the hazard communication program. I also have been properly trained, medically monitored, and fit tested for the work that I am to perform.

Name	**Date**

WORKER ACKNOWLEDGEMENT TO HEALTH-AND-SAFETY PLAN

.

I have been informed of, and will abide by, the procedures set forth in the SSHSP developed for SWMU-6, SWMU-7 and AOC-J Project. I have also been provided with an opportunity to read this SSHSP and the hazard communication program. I also have been properly trained, medically monitored, and fit tested for the work that I am to perform.

Name	**Date**

WORKER ACKNOWLEDGEMENT TO HEALTH-AND-SAFETY PLAN

.

I have been informed of, and will abide by, the procedures set forth in the SSHSP developed for SWMU-6, SWMU-7 and AOC-J Project. I have also been provided with an opportunity to read this SSHSP and the hazard communication program. I also have been properly trained, medically monitored, and fit tested for the work that I am to perform.

Name	**Date**

APPENDIX I
OSHA 300 LOG

OSHA's Form 300 (Rev. 01/2004)

Log of Work-Related Injuries and Illnesses

Attention: This form contains information relating to employee health and must be used in a manner that protects the confidentiality of employees to the extent possible while the information is being used for occupational safety and health purposes.

Year 2005

U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

You must record information about every work-related injury or illness that involves loss of consciousness, restricted work activity or job transfer, days away from work, or medical treatment beyond first aid. You must also record significant work-related injuries and illnesses that are diagnosed by a physician or licensed health care professional. You must also record work-related injuries and illnesses that meet any of the specific recording criteria listed in 29 CFR 1904.8 through 1904.12. Feel free to use two lines for a single case if you need to. You must complete an injury and illness incident report (OSHA Form 301) or equivalent form for each injury or illness recorded on this form. If you're not sure whether a case is recordable, call your local OSHA office for help.

Establishment name _____

City _____ State _____

Identify the person				Describe the case			Classify the case											
(A) Case No.	(B) Employee's Name	(C) Job Title (e.g., Welder)	(D) Date of injury or onset of illness (mo./day)	Buisness Line - Project/Site	(E) Where the event occurred (e.g. Loading dock north end)	(F) Describe injury or illness, parts of body affected, and object/substance that directly injured or made person ill (e.g. Second degree burns on right forearm from acetylene torch)	**CHECK ONLY ONE** box for each case based on the most serious outcome for that case:				Enter the number of days the injured or ill worker was:		Check the "injury" column or choose one type of illness: (M)					
							Death	Days away from work	Remained at work		Away From Work (days)	On job transfer or restriction (days)	Injury	Skin Disorder	Respiratory Condition	Poisoning	Hearing Loss	All other illnesses
									Job transfer or restriction	Other record- able cases								
							(G)	(H)	(I)	(J)	(K)	(L)	(1)	(2)	(3)	(4)	(5)	(6)
						Page totals	*0*	*0*	*0*	*0*	*0*	*0*	*0*	*0*	*0*	*0*	*0*	*0*

Be sure to transfer these totals to the Summary page (Form 300A) before you post it.

Injury | Skin Disorder | Respiratory Condition | Poisoning | Hearing Loss | All other illnesses

(1) (2) (3) (4) (5) (6)

Public

OSHA's Form 300A (Rev. 01/2004)

Summary of Work-Related Injuries and Illnesses

Year _____

U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

All establishments covered by Part 1904 must complete this Summary page, even if no injuries or illnesses occurred during the year. Remember to review the Log to verify that the entries are complete

Using the Log, count the individual entries you made for each category. Then write the totals below, making sure you've added the entries from every page of the log. If you had no cases write "0."

Employees former employees, and their representatives have the right to review the OSHA Form 300 in its entirety. They also have limited access to the OSHA Form 301 or its equivalent. See 29 CFR 1904.35, in OSHA's Recordkeeping rule, for further details on the access provisions for these forms.

Number of Cases

Total number of deaths	Total number of cases with days away from work	Total number of cases with job transfer or restriction	Total number of other recordable cases
_____	_____	_____	_____
(G)	(H)	(I)	(J)

Number of Days

Total number of days away from work	Total number of days of job transfer or restriction
_____	_____
(K)	(L)

Injury and Illness Types

Total number of…
(M)

(1) Injury	_____	(4) Poisoning	_____
(2) Skin Disorder	_____	(5) Hearing Loss	_____
(3) Respiratory Condition	_____	(6) All Other Illnesses	_____

Establishment information

Your establishment name _____

Street _____

City _____ State _____ Zip _____

Industry description (e.g., Manufacture of motor truck trailers)

_____

Standard Industrial Classification (SIC), if known (e.g., SIC 3715)

___ ___ ___ ___

OR North American Industrial Classification (NAICS), if known (e.g., 336212)

___ ___ ___ ___ ___ ___

Employment information

Annual average number of employees _____

Total hours worked by all employees last year _____

Sign here

Knowingly falsifying this document may result in a fine.

I certify that I have examined this document and that to the best of my knowledge the entries are true, accurate, and complete.

_____ Company executive

_____ Title

_____ Phone

_____ Date

Post this Summary page from February 1 to April 30 of the year following the year covered by the form

Public reporting burden for this collection of information is estimated to average 50 minutes per response, including time to review the instruction, search and gather the data needed, and complete and review the collection of information. Persons are not required to respond to the collection of information unless it displays a currently valid OMB control number. If you have any comments about these estimates or any aspects of this data collection, contact: US Department of Labor, OSHA Office of Statistics, Room N-3644, 200 Constitution Ave. NW, Washington, DC 20210. Do not send the completed forms to this office.

OSHA's Form 301
Injuries and Illnesses Incident Report

Attention: This form contains information relating to employee health and must be used in a manner that protects the confidentiality of employees to the extent possible while the information is being used for occupational safety and health purposes.

U.S. Department of Labor

Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

This *Injury and Illness Incident Repor* t is one of the first forms you must fill out when a recordable work-related injury or illness has occurred. Together with the *Log of Work-Related injuries and Illnesses* and the accompanying *Summary*, these forms help the employer and OSHA develop a picture of the extent and severity of work-related incidents.

Within 7 calendar days after you receive information that a recordable work-related injury or illness has occurred, you must fill out this form or an equivalent. Some state workers' compensation, insurance, or other reports may be acceptable substitutes. To be considered an equivalent form, any substitute must contain all the information asked for on this form.

According to Public Law 91-596 and 29 CFR 1904, OSHA's recordkeeping rule, you must keep this form on file for 5 years following the year to which it pertains

If you need additional copies of this form, you may photocopy and use as many as you need.

Completed by _____

Title _____

Phone _____ Date _____

Information about the employee

1) Full Name _____

2) Street _____

City _____ State _____ Zip _____

3) Date of birth _____

4) Date hired _____

5) ☐ Male
☐ Female

Information about the physician or other health care professional

6) Name of physician or other health care professional

_____

_____

7) If treatment was given away from the worksite, where was it given?

Facility _____

Street _____

City _____ State _____ Zip _____

8) Was employee treated in an emergency room?
☐ Yes
☐ No

9) Was employee hospitalized overnight as an in-patient?
☐ Yes
☐ No

Information about the case

10) Case number from the Log _____ *(Transfer the case number from the Log after you record the case.)*

11) Date of injury or illness _____

12) Time employee began work _____ AM/PM

13) Time of event _____ AM/PM ☐ Check if time cannot be determined

14) **What was the employee doing just before the incident occurred?** Describe the activity, as well as the tools, equipment or material the employee was using. Be specific. Examples: "climbing a ladder while carrying roofing materials"; "spraying chlorine from hand sprayer"; "daily computer key-entry."

15) **What happened?** Tell us how the injury occurred. Examples: "When ladder slipped on wet floor, worker fell 20 feet"; "Worker was spayed with chlorine when gasket broke during replacement"; "Worker developed soreness in wrist over time."

16) **What was the injury or illness?** Tell us the part of the body that was affected and how it was affected; be more specific than "hurt", "pain", or "sore." Examples: "strained back"; "chemical burn, hand"; "carpal tunnel syndrome."

17) **What object or substance directly harmed the employee?** Examples: "concrete floor"; "chlorine"; "radial arm saw." If this question does not apply to the incident, leave it blank.

18) **If the employee died, when did death occur?** Date of death

Public reporting burden for this collection of information is estimated to average 22 minutes per response, including time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Persons are not required to respond to the collection of information unless it displays a current valid OMB control number. If you have any comments about this estimate or any other aspects of this data collection, including suggestions for reducing this burden, contact: US Department of Labor, OSHA Office of Statistics, Room N-3644, 200 Constitution Ave, NW, Washington, DC 20210. Do not send the completed forms to this office.

APPENDIX J
BLOOD BORNE PATHOGEN
EXPOSURE CONTROL PLAN

12.0 BLOOD-BORNE PATHOGEN EXPOSURE CONTROL PLAN

Blood-borne pathogens are microorganisms (i.e., bacteria, virus) sometimes present in blood and certain body fluids, which are capable of causing human disease or death. These pathogens can also be present on objects and surfaces that have had contact with infected blood or certain body fluids. Blood-borne pathogens are also capable of causing human disease or death to unprotected people who come into contact with infected blood or body fluids. Diseases caused by blood-borne pathogens include, but are not limited to, hepatitis A, hepatitis B, hepatitis C, malaria, acquired immunodeficiency syndrome (AIDS), and other sexually transmitted diseases. The most significant of these and of greatest concern are hepatitis B and AIDS.

Hepatitis B is a serious disease caused by hepatitis B virus (HBV), which attacks the liver. The virus can cause lifelong infection, cirrhosis (scarring) of the liver, liver cancer, liver failure, and death. Exposure symptoms include fever, fatigue, nausea, vomiting, muscle aches, loss of appetite, and jaundice (yellowing of the eyes or skin). Hepatitis diagnosis is difficult because some symptoms are similar to the flu and may remain mild for an extended period of time. The HBV can remain infectious for up to 10-days, even in dried blood. Hepatitis B vaccine is available for all age groups to prevent hepatitis B virus infection.

Human immunodeficiency virus (HIV) is the virus that causes AIDS. People with HIV have what is called HIV infection. Some of these people will develop AIDS as a result of their HIV infection. Humans may be infected with HIV for many years without experiencing any symptoms. Upon development of AIDS, symptoms may include weight loss, skin lesions, dry cough, fever, fatigue, diarrhea, swelling of the lymph glands, and death. Presently, no cure exists for HIV or AIDS, and no vaccination is currently available.

A hazard exists for blood and other bodily fluids to be infected with dangerous, infectious pathogens. Employees could become infected if they are exposed to these blood-borne pathogens.

The purpose of this Blood-borne Pathogen Exposure Control Plan is to provide the information, procedures, and requirements necessary to prevent employee exposure to blood-borne pathogens.

12.1 REGULATORY, REQUIREMENT, AND POLICY COMPLIANCE

This Blood-borne Pathogen Exposure Control Plan has been prepared in compliance with:

- 29 CFR 1910.1030, Blood-borne Pathogens
- Safety and Health Requirements Manual, EM 385-1-1 (USACE, 2003), Section A.03.06
- Shaw HS 512, Handling of Blood or Other Potentially Infectious Material.

12.2 EXPOSURE DETERMINATION

Occupational Safety and Health Administration (OSHA) requires employers to perform an exposure determination, identifying employees who may incur occupational exposure to blood or other potentially infectious materials. The exposure determination is made without regard to the use of personal protective equipment. For exposure determination purposes, employees are considered to be exposed, even if they wear PPE.

Activities at this project do not present a high risk of employee exposure to blood or other body fluids. An exception to this would be under circumstances when personnel administer first aid care or CPR to injured workers and when personnel clean-up areas and equipment that may have come in contact with blood as a result of the incident. In these cases, there is reasonable potential for employee skin, eye, mucous membrane, or potential contact with blood or other bodily fluids.

OSHA requires a listing of job classifications with identification of tasks performed in which some employees may have potential for occupational exposure. This requirement is for employees to clearly understand the tasks that they may perform have a potential for occupational exposure to infectious materials. The job classifications and associated tasks with an exposure potential are as follows:

- Field Superintendent—Administer first aid or CPR; decontaminate or disinfect surfaces and articles that have contacted infectious materials, and prepare biohazard waste for temporary storage and subsequent disposal.
- Site Safety Officer—Administer first aid or CPR; decontaminate or disinfect surfaces and articles that have contacted infectious materials, and prepare biohazard waste for temporary storage and subsequent disposal.
- Laborer/Equipment Operator—Administer first aid or CPR; decontaminate or disinfect surfaces and articles that have contacted infectious materials, and prepare biohazard waste for temporary storage and subsequent disposal.

These employees have potential for exposure to blood-borne pathogens when administering first aid or CPR and when performing post-accident clean-up operations due to:

- Contact or absorption of blood or blood-contaminated objects through open or broken skin (i.e., cuts, scratches, rashes)
- Blood splashes to their eyes, nose, or mouth, or other mucous membranes
- Punctures through the skin with a contaminated sharp object (i.e., scissors)

Workers can reduce their risk of contacting blood-borne pathogens by implementing the recommended work practices (outlined in this plan) before, during, and after responding to emergency medical incidents primarily involving personal injuries.

12.3 SCHEDULE OF IMPLEMENTATION

The procedures in this Blood-borne Pathogen Exposure Control Plan are to be implemented immediately.

Implementation includes:

- Verifying personnel, who are available to voluntarily provide first aid care and CPR hold a valid training completion certificate from a reputable training provider (American Red Cross or American Heart Association).
- The SSO is responsible for verifying that an appropriate number of personnel have been trained in and hold valid certification to perform first aid and CPR.
- Verifying that personnel voluntarily providing first aid care, CPR, post-accident clean-up operations, and biohazard waste handling have received the specialized training meeting the requirements of 29 CFR 1910.1030, Blood-borne Pathogens; Safety and Health Requirements Manual, EM 385-1-1 (USACE, 2003), Section A.03.06; and Shaw HS512, Handling of Blood or Other Potentially Infectious Material. This training is required for applicable personnel prior to the commencement of work and at least annually thereafter. This training shall cover the following elements:
    - A copy of 29 CFR 1910.1030 and this procedure including an explanation of the contents
    - A general explanation of the epidemiology and symptoms of blood-borne diseases
    - An explanation of the modes of transmission of blood-borne pathogens
    - An explanation of the appropriate methods for recognizing tasks and other activities that may involve exposure to blood and other potentially infectious materials
    - An explanation of the use and limitations of practices that will prevent or reduce exposure including appropriate engineering controls, work practices, and personal protective equipment
    - Information of the types, proper use, location, removal, handling, decontamination, and/or disposal of personal protective equipment
    - An explanation of the basis for selection of personal protective equipment
    - Information on the hepatitis B vaccine, including information on its efficacy, safety, and the benefits of being vaccinated
    - Information on the appropriate actions to take and persons to contact in an emergency

- An explanation of the procedure to follow if an exposure incident occurs including the method of reporting the incident and the medical follow-up that will be made available
- Also information on the medical counseling that is provided for exposed individuals
- An explanation of required signs and labels

The SSO is responsible for verifying that this blood-borne pathogen training has occurred.

- Verifying that engineering controls are readily available at the project for use in an emergency. Engineering controls for this project include:
  - (Three) Red-bags for temporary storage of contaminated PPE and cleaning materials
  - (One) Appropriately labeled, 30-gallon hard-plastic container for the temporary storage of red-bagged waste
  - (One) Whisk-broom and dust pan for cleaning up contaminated broken glass
  - (One) Gallon container of Clorox household bleach
  - (One) Large utility sponge
  - (Three) Rolls paper towels
  - (One) Container of liquid disinfectant hand soap
  - (Three) "Biohazard" warning labels
  - (Ten) Individually packaged disinfectant towelettes
  - (Two) CPR barriers

The SSO is responsible for verifying that this inventory of engineering controls is readily available at the project site for emergency use.

Verifying that the appropriate PPE for use in an emergency is readily available at the project site. Personal protective equipment is necessary to prevent employee exposures to infectious materials. The necessary PPE, which shall be maintained separately for use in an emergency include:

- (Two) Face-shields with ratcheting head-suspension
- (Two-pair) Safety glasses with clear lens
- (Box of 100) Disposable Nitrile examination gloves

- (Two pair) Poly-coated or Saran-coated disposable Tyvek® coveralls with attached hood
- (Two-pair) Vinyl or latex disposable boot covers

The SSO is responsible for verifying that the above inventory of PPE is readily available at the project site for emergency use.

12.4 WORK PRACTICE CONTROLS

Work practice controls reduce the likelihood of exposure by altering the manner in which a task is performed. The work practice controls outlined in this section are applicable to the administration of first aid and the subsequent clean-up operations.

Work practice controls shall be instituted whenever there is potential for employee contact with blood and bodily fluid. Situational examples where these controls are to be implemented include, but are not limited to:

- The voluntary administration of first aid care, such as application of bandages to minor or major cuts and abrasions of another person. This care may allow for contact with sores, wounds, broken skin, blood, or other bodily fluids.
- The voluntary administration of first aid care, such as providing CPR.
- Clean-up activities involving handling soiled articles (e.g., gauze, bandages, compresses, etc.) and the decontamination or disinfecting of surfaces and articles that have contacted potentially infectious materials, such as blood or other bodily fluids.
- Prepare biohazard waste for temporary storage and subsequent disposal.

Based upon professional judgment, an employee may choose to temporarily forego the use of PPE if the employee determines that the use of the PPE will further jeopardize his well being or that of the injured worker. This limited application must be carefully evaluated and considered by the employee. If this situation does occur, Shaw will investigate and document the circumstances in an effort to provide alternative means to avoid further occurrence.

The following are specific work practice controls that shall be implemented in the above noted situations or whenever an employee determines that the implementation of these work practices is prudent or necessary:

- The appropriate PPE shall be donned prior to engaging in any activities that have the potential for employee contact with potentially infectious materials, such as blood or other bodily fluids.
- Hands and face will be washed as soon as possible after engaging in any activities that have the potential for employee contact with potentially infectious materials, such as

blood or other bodily fluids. If wash facilities are not readily available, individually packaged disinfectant towelettes may be used in the interim.

- Eating, drinking, or smoking is not allowed in any work area where a potential exists for occupational exposure to blood borne pathogens.
- Open wounds or cuts shall be promptly bandaged.
- Work surfaces and areas shall be cleaned/disinfected immediately after being contacted by potentially infectious materials. A 10% bleach solution (one part bleach added to nine parts water) shall be applied and allowed to have a contact time of 15-minutes. Non-disposable articles, equipment, or materials contaminated with potentially infectious materials shall be similarly cleaned/disinfected prior to reuse.
- All bins, pails, cans, and similar receptacles intended for reuse, which have become contaminated with blood or other potentially infectious materials shall be cleaned/disinfected immediately after use.
- Broken glassware, which may be contaminated, shall not be picked up directly by hand. Broken glass shall be picked-up using mechanical means, such as by using a whiskbroom and dustpan.
- All personal protective equipment shall be immediately removed upon leaving the potentially contaminated work area, or as soon as possible if visibly contaminated. The contaminated PPE shall be placed in a labeled "red-bag" and then placed in the 30-gallon container for temporary storage and subsequent disposal.
- Any clothing that has contacted blood or other potentially infectious fluids shall be removed as soon as possible.
- Any clothing that has contacted blood or infectious fluids shall be placed in a labeled "red-bag" and then placed in the 30-gallon container for temporary storage and subsequent disposal.

12.4.1 Universal Precautions

Universal precautions is a method of infection control, which operates on the assumption that all human blood and bodily fluids are to be treated as if they are known to be infectious for HIV, HBV, or other blood-borne pathogens. Universal precautions shall be observed to prevent contact with blood or other potentially infectious materials. Universal precautions consist of the following practices:

- All workers shall routinely use appropriate barrier precautions to prevent skin and mucous-membrane exposure when contact with blood or other body fluids is anticipated. Gloves should be worn for touching blood and body fluids, mucous membranes, or non-intact skin and for handling items or surfaces contaminated with blood or body fluids. Masks and protective eye wear or face shields shall be worn during procedures that are likely to generate droplets of blood or other body fluids to prevent exposure of mucous membranes of the mouth, nose, and eyes. Protective suits

shall be worn during procedures that are likely to generate splashes of blood or other body fluids.

- Hands and other skin surfaces shall be washed immediately and thoroughly if contaminated with blood or other body fluids. Hands shall be washed immediately after gloves are removed, using a disinfectant soap.
- CPR barriers or other ventilation devices should be available for use in areas in which the need for resuscitation is foreseeable.
- Workers who have exudative lesions or weeping dermatitis shall be excluded from handling potentially infectious materials until the condition resolves.
- Pregnant workers should be especially familiar with and strictly adhere to precautions to minimize the risk of transmission.

12.4.2 Personal Protective Equipment

The proper use of PPE is an effective work practice control. The following requirements for PPE are mandatory whenever there is potential for employee contact with blood and bodily fluid:

- Inspect PPE prior to use to verify it is in good working order and without defects.
- Blood or other potentially infectious materials.
- Disposable (single use) gloves, such as surgical or examination gloves, shall be replaced when visibly soiled, torn, punctured, or when their ability to function as a barrier is compromised. Gloves should be changed as soon as possible after contact with blood or body fluids. After use, remove gloves from top to bottom inside out, not allowing unprotected skin to contact the exterior of the gloves. Hands and other skin surfaces shall be washed with disinfectant soap immediately after care has been rendered or clean up has been completed. Gloves reduce the incidence of blood contamination of hands, but they cannot prevent penetrating injuries caused by sharp objects. Do not reuse gloves once removed. A CPR barrier shall be used when administering CPR.
- Protection for the eyes, face, hands, body, feet, and against inhalation hazards shall be provided as appropriate for each job.
- Gloves shall be worn when employee has the potential for the hands to have direct skin contact with blood, other potentially infectious materials, mucous membranes, non-intact skin, and when handling items or surfaces soiled with
- Masks and eye protection or chin-length face shields shall be worn whenever splashes, spray, splatter, droplets, or aerosols of blood or other potentially infectious materials may be generated and there is a potential for eye, nose, or mouth contamination.
- Fluid-resistant clothing (e.g. coated Tyvek suits) shall be worn if there is a potential for splashing or spraying of blood or potentially infectious materials. Coated Tyvek coveralls shall also be worn during clean-up activities involving decontamination or

disinfecting of surfaces and articles that have contacted potentially infectious materials, and when preparing biohazard waste for temporary storage and subsequent disposal.

- Fluid-resistant clothing (e.g. coated Tyvek suits) shall be worn if there is a potential for clothing becoming soaked with blood or other potentially infectious materials.
- Fluid-proof coverings shall be worn if there is a potential for shoes or boots to contact blood or other potentially infectious materials.
- Disposable nitrile or vinyl gloves shall be worn for touching blood and body fluids requiring universal precautions, mucous membranes, or non-intact skin and for handling items or surfaces soiled with blood or body fluids to which universal precautions apply.

12.4.3 Waste Handling

All wastes generated as a result of administering emergency first aid care and the subsequent clean-up activities shall be placed in red-bags, labeled as a biohazard, and kept separately from other trash. Wastes used in medical emergency treatment (i.e., gloves, towels, gauze) shall also be bagged and stored in an identical manner. Red-bagged, biohazard waste shall be placed in the 30-gallon collection container, labeled, and secured for temporary storage and disposal. Additional containers shall be obtained as needed and containers shall not be overfilled.

12.5 BIOHAZARD WASTE DISPOSAL

A Shaw Transportation and Disposal Coordinator shall be contacted to arrange for proper disposal of biohazard wastes. The waste shall remain secured on-site in labeled container(s) until disposal arrangements have been made at an approved disposal facility. Disposal of the infectious waste container(s) shall be in accordance with applicable local, state, and federal regulations.

12.6 MEDICAL REQUIREMENTS

Employees receive medical evaluations in accordance with Shaw Procedure HS100 *Medical Policies and Procedures*. The medical requirements of this exposure control plan include provisions for vaccinations to all exposed employees as well as for post-exposure procedures and evaluation. All employees with potential for occupational exposure to blood-borne pathogens shall receive the hepatitis B vaccination and tetanus vaccination prior to workplace exposure, unless they read and sign the Hepatitis B and Tetanus Vaccination Declination Form (see attached).

12.6.1 Hepatitis B Vaccination

All potentially exposed employees will have made available to them at no cost a hepatitis B vaccination. Recombivax or Accelerated Recombivax vaccines shall be utilized. If the employee has previously received the hepatitis B vaccination and/or antibody testing reveals that the employee is immune, a new vaccination is not required. Employees may be subjected to occupational exposure immediately after receiving the first shot in the hepatitis B vaccination series. Antibody testing shall be performed 30-days after completing the hepatitis B vaccination series. Employees unable to develop immunity shall be precluded from further occupational exposure. If a physician recommends a booster dose(s), the doses shall be provided according to standard recommendations for medical practice. The employee will also receive training as to the vaccine's efficacy, safety, benefits, and consequences prior to administration. The vaccination series may also be initiated within 24-hours of an incident with exposure potential.

12.6.2 Tetanus Vaccination

All employees subject to this policy shall maintain current status documentation of tetanus vaccination (current status for tetanus vaccination is within 5 years.) All potentially exposed employees shall be offered a tetanus vaccination at no cost.

12.6.3 Post-Exposure Procedures and Evaluation

All exposure incidents shall be reported as required by Shaw HS020, Accident Prevention Program: Reporting, Investigation, and Review. The Corporate Medical Director shall be advised in addition to standard notification procedures.

Following a report of an exposure incident, each involved employee shall be offered a confidential medical evaluation and follow-up, which includes at least the following elements:

- Documentation of the route(s) of exposure
- HBV and HIV antibody status of the source patient(s) (if known), and how the exposure occurred.
- The medical confidentiality rights of the source patient shall be preserved at all times.
- If the source patient can be determined and permission is obtained, collection of and testing of the source patient's blood to determine the presence of HIV or HBV infection shall be conducted under the direction of the attending physician.
- Collection of blood from the exposed employee as soon as possible after the exposure incident for the determination of HIV and/or HBV status. Actual core antibody and surface antigen testing of the blood or serum sample may be done at that time or at a later date if the employee so requests. If the test is deferred, arrangements shall be made through the attending physician to properly archive the specimen.

- Follow-up of the exposed employee including antibody and antigen testing, counseling, illness reporting, and safe and effective post-exposure prophylaxis, according to standard recommendations for medical practice as defined by the Corporate Medical Director.

Where applicable laws require employee consent, documented consent shall be obtained prior to testing. If an employee refuses the blood test, documentation of the refusal will be made. Documentation of the test results shall be made available to the exposed employee(s). All test results shall be kept confidential.

12.6.4 Physician Information

The following information shall be provided to the evaluating physician:

- A copy of 29 CFR 1910.1030 and its appendices
- A description of the affected employee's duties as they relate to the employee's occupational exposure.

12.6.5 Physician Opinion

For each potentially exposed employee evaluation, the employee shall receive a copy of the evaluating physician's written opinion within 15 working days of the completion of the evaluation. The written opinion shall be limited to the following information:

- The physician's recommended limitations upon the employee's ability to receive hepatitis B vaccination.
- A statement that the employee has been informed of the results of the medical evaluation and that the employee has been told about any medical conditions resulting from exposure to blood or other potentially infectious materials, which require further evaluation or treatment.
- Specific findings or diagnoses, which are related to the employee's ability to receive HBV vaccination. Any other findings and diagnoses shall remain confidential.

12.7 HAZARD COMMUNICATION

There are regulatory requirements for labels, signs, and training. The provisions and exceptions for these are contained in the subsections below.

12.7.1 Warning Labels

Containers used for disposal of blood-contaminated supplies and waste will be labeled in accordance with the word "biohazard." The following symbol shall be an integral part of the label:

12.7.2 Warning Signs

There are no designated areas for medical treatment on-site, because first aid is provided on an emergency basis only; therefore, warning signs are not applicable. In cases of potential exposure, observers and nonessential personnel should be verbally warned to keep a safe distance from injured personnel.

12.7.3 Employee Training Program

All employees who are first aid/CPR trained and may provide assistance shall be trained in the requirements for voluntary providers as described in HS512, *Handling Blood and Other Potentially Infectious Materials* this SHERP and its addenda, and the general provisions of this procedure.

12.8 RECORDKEEPING

There are federal record-keeping requirements for training, medical, and incident reporting documentation. The provisions for keeping these records are contained in the subsections below.

12.8.1 Training Records

All employees covered under this exposure plan shall be trained as required. A record of the training shall be appropriately generated. The training record will contain the date of the training session(s), the contents or a summary of the training session(s), the names of persons conducting the training, and the names of all persons attending the training sessions.

The training records will be maintained by the Shaw Training Department for at least five (5) years from the training date.

12.8.2 Medical Records

Medical records necessary for Shaw employees will include documentation of HBV vaccination status, medical follow-up, post-exposure testing, and a medical professional's written evaluation.

The employee medical records will be forwarded to and maintained by Health Resources, 600 West Cumming Park, Suite 3400, Woburn, Massachusetts 01801-6350 for inclusion in the employee's medical file. Confidentiality of all medical records shall be maintained.

Shaw maintains employee medical records for the duration of the employee's employment plus 30 years thereafter. If, for whatever reason, Shaw no longer does business and no successor exists, Shaw will notify the director of NIOSH in writing three months prior to the disposal of records. If so directed, the records shall be transferred to the director of NIOSH.

12.8.3 Incident Recording

An incident that occurs as a result of rendering emergency medical care will be recorded on the OSHA 300 log as OSHA defines work-related injuries and illnesses. All injuries involving the release of blood or bodily fluids must be immediately reported to the Project CIH for proper reporting and follow-up.

12.9 PLAN REVIEW AND UPDATE

This Blood-borne Pathogen Exposure Control Plan shall be reviewed and updated on an annual basis.

13.0 HEPATITIS B AND TETANUS VACCINATION DECLINATION

Due to the potential for you to have occupational exposure to potentially infectious materials in your work, the company will provide and encourages you to accept, vaccination for Hepatitis B and Tetanus. Information to assist you in this decision is provided below.

13.1.1 Tetanus

- A bacterial disease causing muscle spasms, seizures, and "lockjaw".
- This single injection vaccination has few side effects.
- There is minimal loss in protection if the vaccination is given at the time of an exposure/injury rather than in advance.

13.1.2 Hepatitis B

- A viral infection of the liver.
- About 9,500 occupational cases occur annually, mostly in health care workers, with about 200 deaths.
- This three injection vaccination has few side effects.
- Vaccination is 90% effective for at least seven years when given prior to exposure.
- Vaccination is 70-88% effective when given within one week of exposure.
- Hepatitis B can survive in the environment for 24-48 hours after drying.
- Risk of infection from one cut or puncture wound from a contaminated object:
    - Hepatitis B Virus 6-30%
    - Human Immunodeficiency Virus (AIDS) 0.5%.

If you wish to talk to a company doctor before making your decision, please ask the Health and Safety Manager to make arrangements for you. *If you choose to decline vaccination at this time, you must print and sign your name, and date the bottom of this form.*

I understand that due to my occupational exposure to blood or other potentially infectious materials I may be at risk of acquiring Hepatitis B Virus (HBV) infection.

I have been given the opportunity to be vaccinated by hepatitis B vaccine, at no charge to myself. However, I decline Hepatitis B vaccination at this time. I understand that by declining this vaccine, I continue to be at risk of acquiring Hepatitis B, a serious disease.

If in the future I continue to have occupational exposure to blood or other potentially infectious materials and I want to be vaccinated with Hepatitis B vaccine, I can receive this vaccination series at no charge to me.

Name (print) _____

Signature _____ Date _____

FIELD SAMPLING AND ANALYSIS PLAN
FOR
LABORATORY ACTIVITIES
REMOVAL ACTIONS
SWMU 6, SWMU 7, AOC J, and AOC R
FORMER NAVAL AMMUNITIONS SUPPORT DETACHMENT
VIEQUES, PUERTO RICO

Prepared for:

Department of the Navy
Contract N62470-03-D-4402
Task Order 0015
NAVFAC Atlantic
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August 2004
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List of Acronyms

AOC	Area of Concern
ASTM	American Society for Testing and Materials
°C	degrees Celsius
CAA	Clean Air Act
CAR	corrective action report
CAX	Cheatham Annex
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-term Environmental Action Navy
CLP	Contract Laboratory Program
CNO	Chief of Naval Operations
COC	Chain of Custody Form
CRM	certified reference material
CWA	Clean Water Act
DI	de-ionized
DOD	U.S. Department of Defense
DQO	data quality objectives
DRO	diesel range organics
EDD	electronic data deliverables
EPA	U.S. Environmental Protection Agency
ER,N	Environmental Restoration, Navy
FFA	Federal Facilities Agreement
FID	flame ionization detector
FS	feasibility study
FSP	Field Sampling Plan
FSSI	Field Support Services, Inc.
G	glass
GC	gas chromatography
GC/MS	gas chromatography/mass spectroscopy
GRO	gasoline range organics
GW	groundwater
H_2SO_4	sulfuric acid
HASP	Health and Safety Plan
HCL	hydrochloric acid
HDPE	high density polyethylene
HNO_3	nitric acid
HPLC	high-performance liquid chromatography
ICB	initial calibration blank
ICP	inductively coupled plasma
ICS	interference check standard
ID	identification

IDW	investigation-derived waste
IR	Installation Restoration
IS	internal standards
LANTDIV	Department of the Navy, Atlantic Division
LCS	laboratory control sample
LIMS	Laboratory Information Management System
MB	method blank
MDL	method detection limit
mg/Kg	milligram per kilogram
mg/L	milligram per liter
MS/MSD	matrix spike/matrix spike duplicate
N/A	not applicable
NaOH	sodium hydroxide
$Na_2S_2O_3$	sodium thiosulfate
NELAP	National Environmental Laboratory Accreditation Program
NEPA	National Environmental Policy Act
NFESC	Naval Facilities Engineering Service Center
NIOSH	National Institute of Occupational Safety and Health
NIST	National Institute of Standards and Technology
NPDES	National Pollutant Discharge Elimination System
OSHA	Occupational Safety and Health Administration
OVM	organic vapor monitor
P	polyethylene
PA	preliminary assessment
PAH	polynuclear aromatic hydrocarbon
PARCC	precision, accuracy, representativeness, comparability, and completeness
PC	Shaw project chemist
PCB	polychlorinated biphenyls
PDS	post digestion spike
PE	performance evaluation
PM	Shaw project manager
ppb	parts per billion
ppm	parts per million
QA	quality assurance
QAM	laboratory quality assurance manual
QAO	quality assurance objectives
QAPP	quality assurance project plan
QC	quality control
QMS	quality management system
R	recovery
RA	remedial action
RCRA	Resource Conservation and Recovery Act
RD	remedial design
RI	remedial investigation
RL	reporting limits

RPD	relative percent difference
SAP	sampling and analysis plan
SARA	Superfund Amendments and Reauthorization Act
SCS	Soil Conservation Survey
SI	site inspection
SOP	standard operating procedure
SOW	statement of work
SRM	standard reference material
SVOC	semi-volatile organic compound
SW-846	Test Methods for Evaluating Solid Waste, Physical/Chemical Methods
SWMU	solid waste management unit
TCLP	toxicity characteristic leaching procedure
TIC	tentatively identified compounds
ug/Kg	microgram per kilogram
ug/L	microgram per liter
USGS	U.S. Geological Survey
USACE	U.S. Army Corps of Engineers
UST	underground storage tank
VOC	volatile organic compound

Preface

FSSI/Shaw has been tasked by the Department of the Navy, NAVFAC Atlantic to perform various removal actions at the former Naval Ammunitions Support Detachment, Vieques Island, Puerto Rico. This Field Sampling and Analysis Plan (SAP) has been prepared as a deliverable under Contract Number N62470-03-D-4402, Task Order 0015. This SAP will apply to all site and laboratory activities performed under contract for SWMU 6, SWMU 7, AOC J and AOC R.

Included with the SAP is the Quality Assurance Project Plan (QAPP) which provides the guidelines for the systematic data collection and analysis associated with project **(Appendix A)**. In accordance with the Uniform Federal Policy for Quality Assurance Project Plans (UFPQAPP, March 2005), the Master QAPP includes 37 worksheets that detail various aspects of the environmental investigation process It establishes protocols to allow for comparability and defensibility of sampling and analytical data.

1.0 INTRODUCTION

This Field Sampling and Analysis Plan (SAP) presents the procedures designed to achieve comparability and defensibility regarding the reporting and use of analytical data from pre- and post-excavation soil samples to be collected at SWMU 6, SWMU 7, AOC J and AOC R at former NASD, Vieques, Puerto Rico. This SAP provides assurance that data are collected, analyzed, reviewed, and reported in a consistent and representative manner. This SAP is required reading for representative field and laboratory personnel involved in sample handling and data reporting for the samples collected as a part of these removal actions.

2.0 PROJECT DESCRIPTION

This Field Sampling and Analysis Plan (SAP) for the Removal Actions at SWMU 6, SWMU 7, AOC J and AOC R at former Naval Ammunitions Support Detachment, Vieques, Puerto Rico, has been prepared by FSSI/Shaw for the Department of the Navy, NAVFAC Atlantic under Contract Number N62470-03-D-4402, Task Order 0015. The following sections of this SAP describe the general scope of the project, current remedial and monitoring activities, and procedures involved in sample collection. The associated QAPP addresses policies to be used by the fixed-base laboratory for delivering usable and legally-defensible data.

2.1 SCOPE OF WORK

FSSI/Shaw will excavate and remove an estimated 11,050 tons of debris and soil from these four sites in a manner consistent with the types of contaminate encountered at each site. The excavations may be one to three-feet below ground surface (bgs) and irregular in shape. The actual tonnage may be more or less, depending upon the site conditions encountered. The overall goal of the actions is to eliminate the uncertainty of the debris being a potential future source of contamination.

Waste Characterization Samples SWMU 7, SWMU 6, AOC J and AOC R

It will be necessary to collect waste characterization samples to establish proper waste disposal methods. Criteria for the Waste Characterization Sampling are listed within Appendix A – QAPP Worksheet #17. The samples shall be analyzed for the parameters listed in **Appendix A – QAPP Worksheet #19**. All results will be evaluated against parameters listed in **Appendix A – QAPP Worksheet #15** as part of the waste profiling to ensure proper disposal to a Navy approved off-site disposal facility.

Confirmation Sample Laboratory Analyses

Confirmation Samples shall be taken to confirm the completion of the removal activities. Confirmation sampling protocol shall be established once the results of the waste characterization sampling and associated residual risk assessment are available, and shall be based upon the criteria detailed within the QAPP Worksheet # 10 and 17, located within Appendix A of this Sampling Plan. All confirmation sampling locations shall be concurred upon by all the regulatory agencies prior to any confirmatory sampling events.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

All project personnel, including field, laboratory, and subcontractor personnel are subject to the requirements of this SAP. Following is an overview of responsibilities provided for this scope of work.

3.1 PRIMARY CONTRACTOR TASKS

The FSSI/Shaw Project Manager (PM) serves as the focal point for control of all project activities. The PM will be supported by the project team which will provide reviews, guidance, and technical advice on project execution issues. The project team will consist of supervisory, health and safety, technical, and QA/QC staff members to ensure safe and compliant progress of the removal actions. Individuals of the project team each have specific responsibilities involving adherence with the Statement of Work (SOW), QAPP, Health and Safety Plan (HASP), and the SAP.

3.2 SUBCONTRACTOR ACTIVITIES

The selection of qualified subcontractors will be in accordance with FSSI/Shaw procurement and QA procedures. The bulk of the work will be accomplished by FSSI/Shaw personnel, but a selection of environmental laboratories for Navy work have been procured and coordinated based on capacity and capability. FSSI/Shaw performs quality checks to determine that the subcontractors have fulfilled the procurement requirements necessary to perform site or off-site activities. All subcontractors will be required to follow the procedures of the SAP.

3.3 QUALIFICATIONS AND TRAINING OF PERSONNEL

Personnel assigned to the project, including field personnel and subcontractors, will be qualified to perform the tasks to which they are assigned. Said personnel will meet requirements set forth in OPNAVINST 5090.1B 25-5.8. This includes but is not limited to basic sampling techniques; field testing methodology, task-specific sampling methods, maintenance of environmental paperwork, and how to avoid cross contamination. In addition to education and experience, specific training may be required to qualify individuals to perform certain activities. Training will be documented appropriately and the forms placed in the project file as a record. Project personnel will receive an orientation to the full SAP, QAPP, and the HASP as appropriate to their responsibilities before participation in project activities. Training of field personnel will be provided by the Site Supervisor, the QA Officer, or by a qualified designee. (Refer to **Appendix**

A - QAPP Worksheets #7 and #8 for Personnel Responsibilities and Qualifications Table and Special Personnel Training Requirements Table, respectively.)

4.0 SAMPLING PROCEDURES OVERVIEW

4.1 PURPOSE

The sampling events for this project may include, but are not limited, to the following:

- Waste Characterization Sampling
- Post-excavation Confirmation Sampling

Sampling procedures will be compliant with EPA, state, and applicable DOD specifications. This SAP meets or exceeds guidelines set forth by Naval Environmental compliance documentation OPNAVINST 5090.1B Chapter 25, NAVSEA T0300-AZ-PRO-010, and National Environmental Laboratory Accreditation Program (NELAP) quality system requirements. The matrix and required analytical will determine which sample collection procedure(s) is appropriate. Section 5.0 provides specific techniques to be used when collecting characterization/confirmation soil samples. The remainder of Section 4.0 of this SAP addresses information that is consistent for any type of field sampling. An example sampling and analysis summary table is included in **Appendix A – QAPP Worksheet # 19**.

4.2 PREPARATIONS FOR FIELD SAMPLING

The Site Supervisor will oversee procurement of all required sampling equipment (**Table 4-1**). Where feasible, single-use disposable equipment will be used to minimize decontamination time and effort. For events requiring bailers and tubing, Teflon® and/or silicone will be used. Stainless steel will be used where metal equipment is to be used for soil sample collection.

TABLE 4-1
Equipment and Supplies Checklist
Former Naval Ammunitions Support Detachment –Vieques, Puerto Rico

Equipment	Required	Calibration	Comments
Materials			
Paper towels	✓		
Trash bags	✓		
Aluminum foil	✓		
De-ionized water	✓		
Alconox® detergent	✓		
Plastic sheet	✓		
Nylon rope	✓		
Teflon® bailers	✓		
Teflon® or silicone tubing	✓		
Cooler	✓		
Ice	✓		
Baggies	✓		
Bubble wrap	✓		
Stainless steel bowl	✓		
Forceps	✓		
1:1 HNO_3	✓		
Appropriate PPE	✓		PPE - personal protective equipment
Instruments			
Dual interface probe	✓		
PID or FID	✓	✓	PID – photo ionization detector FID - flame ionization detector

The Site Supervisor will ensure that all permits and authorizations have been acquired prior to the start of excavation sampling at the four sites at former NASD. The Project Chemist or appropriate representative will notify the chosen laboratory to provide EPA-approved sample bottles, preservative, and shipping containers. The laboratory may also be requested to provide deionized water for decontamination of equipment, trip blanks, chains of custody, custody seals, and approved packing material to protect sample bottles during transit.

4.3 HEALTH AND SAFETY PROGRAM

A Health and Safety Officer will be assigned to oversee compliance of the remedial plans with OSHA regulations. This Health and Safety Officer may or may not be assigned solely to work at former NASD and may or may not handle tasks for all sites undergoing remediation or

monitoring. The Health and Safety Officer(s) is responsible for writing and maintaining a Health and Safety Plan (HASP). He/she will ensure that the HASP is available to all site personnel prior to beginning work. A sign-off form will be maintained with the site copy of the HASP to indicate that personnel have read the document and are aware of its implications.

4.4 SAMPLE CUSTODY AND DOCUMENTATION

Sample custody can be defined as physical possession of samples, having samples within visual range, or having samples located in a restricted access area. Sample possession during all sampling efforts must be traceable from the time of collection until the results are verified and reported. The sample custody procedures provide a mechanism for documentation of all information related to sample collection and handling. The primary piece of documentation to ensure sample custody is the Chain of Custody Form (COC). FSSI/Shaw personnel are responsible for providing evidence of sample custody from the time of collection until the laboratory receives the samples. Specific field sample custody procedures are outlined in **Appendix A – QAPP Worksheet # 27**. The laboratory will be able to provide documentation of sample custody from that point to sample disposal. In **Appendix A – QAPP Worksheet 26** the procedures for laboratory sample disposal are defined. In **Appendix B** the custody procedures used by the laboratory are defined.

As part of appropriate documentation, all sample bottles will be adequately labeled. The label will present sample identification and collection information. It will be pre-printed from the sample tracking system or completed with indelible ink. At a minimum, all sample labels will include the following sample information:

- Field sample location and unique sample identifier
- Project name and number
- Analysis requested for each bottle
- Method of preservation for each bottle
- Date and time of collection
- Initials of sample technician.

A sample numbering system will be utilized in the field to uniquely identify each sample collected at former NASD (**Appendix A – QAPP Worksheets #18 and #27** contains the proper sampling nomenclature). The sample number will be traceable to the site, location, and depth (where applicable). The sample identification and description will be recorded by the Site Supervisor or representative in the sample collection logs.

4.4.1 FIELD CUSTODY PROCEDURES

The sampling team with the Site Supervisor will maintain overall responsibility for the care and custody of the collected samples until they are transferred or properly dispatched to the fixed-base laboratory. All shipping or sample transfer activities will be documented (**Appendix A – QAPP Worksheet # 27**).

Transfer of custody and shipping procedures will include:

- The Site Supervisor instructing sampling team personnel in the proper COC procedures before sampling begins,
- A COC entry made in the field for each sample. This document will accompany the samples in shipment, and a copy will be maintained at the site for placement in the project files at the conclusion of field activities. The custody of individual sample containers will be documented by recording each sample identification and the number of bottles on the appropriate COC form,
- COC records initiated in the field will be placed in a plastic bag and taped to the underside of the top of the shipping cooler used for sample transport,
- Each time responsibility for custody of the sample changes, the new custodian will sign and date the record,
- All coolers must be secured at the site with two custody seals prior to transport. Custody seals should be signed and dated by the person relinquishing custody of the samples being shipped. They should be placed over the opening of each cooler so that the cooler cannot be opened without breaking the seal.

4.4.2 LABORATORY CUSTODY PROCEDURES

All samples to be analyzed by the fixed-base laboratory will be shipped via overnight courier service. Upon receipt, a representative of the laboratory shall check the integrity of the custody seals, then locate, sign, and date the COC. The laboratory is responsible for verifying that the COC and containers are in agreement. The COC, a Cooler Receipt Form, and information regarding any discrepancies between the COC and bottle labels will be faxed to the Project Chemist prior to preparation for analysis. The Laboratory Information Management System will provide evidence of sample custody from receipt by the laboratory until appropriate disposal (**Appendix A – QAPP Worksheet #27**).

4.5 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Samples must be placed in sample containers certified by the manufacturer to be pre-cleaned to EPA specifications. Containers provided by the fixed-base laboratory will be compatible with the matrix and intended analysis, and properly prepared and preserved to maintain sample integrity. **Appendix A – QAPP Worksheet #19** specifies the types of containers needed for each analytical method and matrix.

Physical and chemical preservation includes techniques designed to stabilize the concentrations of analytes in the sample matrix during the time from sample collection through preparation and analysis. All samples shall be preserved by cooling to 4°C $\pm$ 2°C immediately after sample collection. Whenever possible, chemical preservatives will come prepared from the laboratory. The final pH of the chemically-preserved samples will be checked using pH paper and the result recorded. The pH paper will not be dipped into the sample container to measure pH, but an aliquot of sample shall be removed for pH testing and then properly disposed. If the pH check indicates pH adjustment is necessary, additional preservative will be added to attain the correct sample pH.

A sample holding time establishes the recommended maximum time that a sample may be held under preservation before extraction and analysis without compromising sample representativeness. Preservation techniques and holding times are standardized by the EPA according to analytical method. **Appendix A – QAPP Worksheet #19** summarizes the latest information on preservation techniques and holding times for the requested analyses.

Chemical preservation is not usually recommended for solid matrix samples. Unpreserved samples that require preservation by the laboratory should be indicated on the sample label and should be flagged on the COC to identify these samples to the laboratory receiving personnel as requiring special handling.

4.6 SAMPLE PACKAGING AND SHIPMENT

Samples that are collected for off-site laboratory analysis that require overnight shipment will be generally prepared by:

- Sealing the container in an outer Ziploc storage bag,
- Securely wrapping and taping each collected bottle in bubble wrap (or other similar shock-adsorbing material).
- A temperature blank will be included in each cooler. The temperature will be recorded upon receipt at the laboratory to verify sample temperatures during transport.

- When collecting volatile samples, including GRO samples, the field personnel will include trip blanks in each of the coolers containing volatile samples.

At least three sides of the container must be wrapped or surrounded with material when placing the samples into the shipping cooler. Adequate ice will be placed in doubled zip lock bags and added to the cooler around and over the top of the sample containers to form a cooling layer to help ensure proper preservation during shipment. In addition, most samples will have been pre-cooled to the desired temperature prior to packing for shipment.

Trip blanks are volatile bottles that have been prepped by the laboratory using laboratory grade water and are shipped with the field sample containers, and returned unopened to the laboratory for volatile analysis. Trip blanks are used to indicate if any contamination during shipment of containers to the field, storage of containers at the site, or shipment from the site to the laboratory has occurred. Trip Blanks will be included in each cooler used for shipping volatile samples. If more than one cooler will be sent on a given day, each cooler should contain a set of Trip Blank samples. To minimize the number of Trip Blanks being analyzed, care should be taken to place all volatile samples in one cooler.

Temperature blanks are Nalgene bottles containing water that will be included in each sample cooler. Upon receipt at the laboratory, the temperature will be recorded to verify the sample temperatures during transport. Completed and signed COCs will be placed into the cooler in a protective Ziploc bag and taped to the underside of the cooler lid. A minimum of 2 custody seals will be applied across the opening of the cooler and the lid secured by wrapping the cooler with clear plastic packing tape. The cooler will then be ready for shipment according to the methods required by the overnight delivery service. At a minimum, the laboratory address, telephone number, and contact name should be included on the original air bill and, if multiple packages are sent, on each sample cooler.

At all times from the point of sample collection in the field through storage, inventory, preparation, and shipment, the samples must remain sealed, protected from sources of contamination, and adequately preserved by chilling.

As shipping regulations are subject to frequent updates without notification, the sample technician should, at least quarterly, petition a set of applicable shipping regulations from the overnight shipper to be assured of regulatory compliance.

4.7 DECONTAMINATION

All reusable equipment that may directly or indirectly contact samples shall be cleaned in a designated decontamination area. In addition, the contractor shall take care to prevent the sample from coming into contact with potentially contaminating substances, such as tape, oil, engine exhaust, corroded surfaces, or dirt. To minimize the possible contribution of even trace levels of contamination from sampling equipment, adequate decontamination must be completed prior to each use of the equipment. Sampling and drilling devices shall be scrubbed with a solution of potable water and Alconox®, or equivalent laboratory-grade detergent. The equipment will then be rinsed with copious quantities of potable water followed by a deionized (DI) water rinse. If equipment has come in contact with oil or grease, it will be rinsed with pesticide-grade methanol followed by pesticide-grade hexane. The equipment will be air-dried on a clean surface. If the sampling device shall not be used immediately after decontamination, it shall be wrapped in oil-free aluminum foil. Decontamination fluids shall be collected in appropriate storage containers. These will be sampled for characterization and disposal by an approved facility.

5.0 SOIL/SEDIMENT SAMPLING

5.1 PURPOSE

This section identifies the steps and technical areas that will be encountered in conducting a soil/sediment sampling program. The role of a sampling run is identified along with procedures that will guide the sample technician in conducting a compliant program. Refer to **Appendix A - QAPP Worksheets #17 and #18**.

5.2 GENERAL SAMPLING PROCEDURES

All sampling events should be well planned and coordinated with the Project Manager, the Site Supervisor, and the Project Chemist as required by this SAP. All target analyte lists should be reviewed with both the laboratory that will perform the analysis and the sample technician(s). Refer to **Appendix A – QAPP Worksheets #15 and #19.**

Sampling personnel are critically important to sampling program success, since the sample technician is often in the best position to detect areas of suspicion. Even sophisticated sampling procedures should not be relied upon to replace good judgment and common sense on the part of sampling personnel in discerning the difference between routine and extreme case scenarios. Sampling personnel must be alert to unusual conditions in their surroundings such as dead animal or plant life, odors not normally associated with an area, and changes in the visual appearance of the location to be sampled.

Samples will be collected using a stainless steel trowel, a split-spoon sampler, hand auger, or Encore® Sampler. All sampling equipment that may come into contact with samples or sampling surfaces will be constructed of stainless steel, borosilicate glass, or Teflon®. All equipment used for collection, transfer, and homogenization will be properly decontaminated before collecting samples and between sampling locations.

Samples collected for the analysis of Target Compound List (TCL), semi-volatile organic compounds (SVOCs), pesticides, and polychlorinated biphenyls (PCBs), explosives, and Target Analyte List (TAL) metals shall be collected using a steel trowel, split spoon sampler, or a hand auger. After these samples are collected they will be homogenized as thoroughly as possible in a stainless-steel bowl. Samples for the analysis of TCL volatile organic compounds (VOCs), TCLP VOCs and GRO shall be collected using the Encore® Sampler or equal. Samples shall be collected according to the SOPs listed in Appendix A, QAPP Worksheet 21, and shall be placed

in the appropriate sample containers, labeled with proper identification, and packed in a cooler with ice pending shipment to the laboratory. To maintain integrity, samples collected in the field must be placed in a dedicated sample ice chest, on ice, and chilled to 4ºC ± 2ºC from the time of collection until receipt by the laboratory for analysis. (See **Appendix A – QAPP Worksheet #21** for Project Sampling SOP Reference Table.)

5.3 SAMPLE COLLECTION OF CONFIRMATION, FILL, AND WASTE DISPOSAL SAMPLES

All waste soil samples will be visually classified and documented on a sample collection log. The Site Supervisor will identify the method for sampling based on the task-specific SAP. The following sample procedure is applicable:

Samples shall be collected in accordance with the SOP's detailed in Worksheet #21, Appendix A, and placed into the appropriate number of jars as noted in **Appendix A – QAPP Worksheet #19**, taking care to minimize headspace as much as possible, and then labeled and stored in a cooler at 4°C. No duplicate samples or equipment blank samples are required for waste disposal samples. Refer to **Appendix A – QAPP Worksheet #20** for required blanks, duplicates, and MS/MSD pairs for confirmation sampling.

All sample documentation and chain of custody procedures outlined in Section 4.4 should then be followed. After proper documentation has been performed, sample packaging and shipping as outlined in Section 4.6 of this document should be completed.

6.0 LABORATORY AND DATA PERFORMANCE CRITERIA

6.1 MEASUREMENT PERFORMANCE CRITERIA

All investigative remediation samples and soil collected for waste disposal characterization will be evaluated against **Appendix A – QAPP Worksheet #15**.

Laboratory established precision and accuracy criteria are listed in the laboratory SOPs in **Appendix A – QAPP Worksheets #12, #15, and #28** and **Appendix B.**

6.2 ANALYTICAL LABORATORY AND LABORATORY CERTIFICATION

Accutest Laboratories Southeast, Inc. located in Orlando, Florida has been chosen to complete the analytical requirements. Accutest Southeast has current National Environmental Laboratories Accreditation Conference (NELAC), Army Corp validation and a Navy approval letter. Accutest will provide to Shaw copies of all current certifications prior to project start up.

6.3 LABORATORY QUALITY SYSTEMS MANUAL

Accutest has a detailed Quality Systems Manual, Volume VI, Revision II, dated March 2005 that is designed to meet the quality program requirements of NELAC and ISO Guide 25. This Quality Systems Manual is included in **Appendix C**.

6.4 LABORATORY STANDARD OPERATING PROCEDURES

The laboratory has established Standard Operating Procedures (SOPs), and each SOP has a specific SOP Number with revisions dates. An analytical SOP reference table is provided in **Appendix A – QAPP Worksheet #23**. Distributions of updated SOPs are documented on an SOP cover page. Superseded copies of the SOPs are collected according to the distribution list contained within the SOP cover page. As changes occur, these documents are updated and the staff is trained to apply the changes. The complete list of laboratory SOPs is included in **Appendix B**. These SOPs provide specific operational instructions on the execution of each quality element and assure compliance with the requirements of the Quality Systems Manual. Analytical method SOPs are based upon USEPA-approved methods or other recognized standard methods.

6.5 LABORATORY TRAINING PROCEDURES

Laboratory senior management staff retains oversight responsibility for the data integrity program and retains the ultimate responsibility for execution of the data integrity program elements. Senior laboratory management staff is responsible for providing the resources required to conduct SOPs, ethics training, and operate data integrity evaluation procedures.

Laboratory employees receive technical ethics training during new employee orientation. All employees are required to attend ethics refresher training and to sign an ethical conduct agreement annually, which verifies their understanding of the laboratories ethics policy and the analyst's ethical responsibilities. Training on data integrity procedures and SOPs are conducted by the individual departments' group leaders within the laboratory. All records of training are retained at the laboratory in the individual staff training folders and are maintained by the laboratory quality assurance officer. All information related to staff qualifications, experience, external training courses, and education are placed into the individuals training file. Verification documentation for laboratory orientation, health and safety, and quality assurance training is also maintained with the training file. Additional training documentation is added to the files as it occurs. This includes data for initial and continuing demonstrations of proficiency, performance evaluations, study data and notes, and attendance lists from individual and group training sessions.

6.6 LABORATORY CORRECTIVE ACTION AND REPORTING PROCEDURE

Routine corrective action is defined as procedures used to return out of control analytical systems back to control. This level of corrective action applies to all analytical quality control parameters and analytical system specification as defined in the laboratory SOPs. Bench analysts have full responsibility and authority for performing routine corrective action. Routine corrective actions are documented as part of the analytical record. Defective processes, holding time violations, systematic errors and quality defects that occur are to be reported by the bench chemist immediately to the section supervisor and a non-conformance record initiated. The section supervisor will notify the designated Shaw Laboratory Project Manager (Heather Wandrey) who will then notify the Shaw Lantdiv Project Chemist (Natasha Sullivan). All notifications must be made in a timely manner. The non-conformance record should become part of the analytical record.

6.7 LABORATORY INSTRUMENT CALIBRATION PROCEDURES (WORKSHEET 24)

Calibrations use a series of reference standards to establish the concentration range for performing quantitative analyses. Method specific procedures for calibration are followed prior to any analysis and are established in the laboratory SOPs provided in **Appendix B** and defined in **Appendix A – QAPP Worksheet #24**. It is the laboratories responsibility to maintain logbooks of these calibrations.

6.7.1 Initial Calibrations

Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run, and of producing a linear calibration curve. Initial Calibrations are verified using a calibration standard, and continuing validity of an existing calibration must be verified prior to sample analysis. The response to the standard must meet the criteria as stated in the laboratory SOPs and are defined in **Appendix A - QAPP Worksheet #28** and indicate that the initial calibration curve remains valid. If the criterion has not achieved corrective action, re-calibration is performed before any samples may be analyzed, see **Appendix A - QAPP Worksheet #28**.

6.7.2 Continuing Calibration Verifications

Continuing Calibration Verification is used on a daily basis to verify whether instrument performance and quantitation models are relatively unchanged from the day the Initial Calibration was performed. This process requires testing a known amount of target analyte(s), usually around mid-to-low point of Initial Calibration and calculating its value using quantitation model obtained during the process of initial calibration.

Continuing Calibration Verification is performed at the beginning of the analytical sequence, then used to verify calibration every 10 samples and at the end of the analytical sequence. Such frequency is necessary since the sample matrix can interfere with instrument performance. This is not required for GC/MS methods. The analytical sequence is restricted to certain period of time.

In the event of a failing Continuing Calibration Verification refer to the laboratory method SOP for corrective action and **Appendix A - QAPP Worksheet #28**. Corrective action may include re-analysis of the samples.

6.8 LABORATORY QUALITY CONTROL SAMPLES

Prior to the analysis of the field sample, the laboratory analyst must determine that the method is functioning properly. Specific control parameters indicate whether critical processes meet specified requirements before continuing with the analysis. Method specific control parameters must meet criteria before sample analysis can be conducted. Each of the following parameters is related to processes that are under the control of the laboratory and can be adjusted if out of control:

6.8.1 Method Blanks (MB)

A method blank is analyzed during the analysis of any field sample (one per analytical batch). The method blank is defined as a sample. It contains the same standards (internal standard, surrogates) and reagents that are added to the field sample during analysis. If the method blank contains target analytes at concentrations that exceed the method criteria as defined in the laboratory SOPs and in **Appendix A - QAPP Worksheet #28**, the source of the contamination is eliminated before proceeding with analysis. Corrective action may include re-extraction of sample aliquot and or re-analysis.

6.8.2 Laboratory Control Samples (LCS)

Laboratory control samples (LCS) are used to monitor laboratory accuracy by calculating the percent recoveries of known spiked compounds. All LCS percent recoveries must be within the specified performance limits. These performance limits are derived from published method specifications or from statistical controls generated from the laboratory method performance data. If the spike accuracy exceeds the performance limits, corrective action as specified in the laboratory SOP in **Appendix B** for the method is performed and verified before continuing with a field sample analysis.

The analysis of samples can be initiated following a successful demonstration that the method is operating within established controls. Additional controls are incorporated into the analysis of each sample to determine if the method is functioning with established specifications for each individual sample. Sample QC data is evaluated and compared to established laboratory performance criteria as defined within the laboratory SOPs and in **Appendix A - QAPP Worksheet #28**. If the criteria is not achieved the method or the SOP specifies the corrective action required to continue sample analysis.

The following are sample control parameters (All parameters may be found in **Appendix A - QAPP Worksheet #28**):

6.8.3 Matrix Spike/Matrix Spike Duplicates (MS/MSD)

Matrix Spike (MS)/Matrix Spike Duplicates (MSD) are analyzed one per twenty samples or one for each analytical batch. Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard are added. The matrix spike and spike duplicate are then extracted and taken though all cleanup procedures along with the other samples within the analytical batch to monitor the analytical precision and accuracy for the sample and matrix. Precision and accuracy are method dependent. If precision and accuracy criteria are not achieved, corrective action is required as established in the laboratory SOPs. If the recoveries indicate that the problem is procedure related, re-extraction and re-analysis is required. If the recoveries indicate that the failures are sample related, the Shaw Chemist will be contacted and a decision will be made to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed.

6.8.4 Surrogate Spikes (Organic Parameters)

Surrogate spikes are organic compounds that are similar in behavior to the target analytes but unlikely to be found in nature. They are added to quality control and field samples to measure the method performance for each individual sample. Surrogate accuracy limits are derived form published method specifications or by statistical evaluation of laboratory generated surrogate accuracy data. Accuracy data is compared to the applicable performance limits. If the surrogate accuracy exceeds the performance limits, corrective action, as specified in the method or laboratory SOP, is performed before sample data can be reported. These corrective actions may include re-extraction and or re-analyzing sample.

6.8.5 Internal Standards (Organic Methods)

Internal standards are retention time and instrument response markers added to every sample to be used as references for quantitation. Their response is compared to reference standards and used to evaluate instrument sensitivity on a sample specific basis. Internal standard retention time is also compared to reference standards to assure that target analytes can be located by their individual relative retention time. If the internal standard retention time criteria as described in the laboratory SOP and **Appendix A - QAPP Worksheet #28** is not met, corrective action is required. If failure is due to instrument performance issues, the problem must be identified, corrected, and the sample must be re-analyzed. If no instrument problem is found the sample

must be re-analyzed. If upon re-analysis the responses are still not within limits, the problem may be considered sample matrix interference.

6.8.6 Serial Dilutions (Metals Analysis)

Serial dilutions of metal samples are analyzed to determine if the analytical matrix effects may have impacted the data being reported. If the value of the serially diluted samples does not agree with the undiluted value with a method specified range, the sample matrix may be causing interference, which may lead to either a high or low bias. If the serial dilution criterion is not achieved as defined with the laboratory SOP and **Appendix A - QAPP Worksheet #28**, the data should be flagged to indicate possible bias matrix effect.

6.9 FIELD DUPLICATE SAMPLE EVLAUATION

For chemical data, sampling precision will be evaluated by obtaining one duplicate sample for every 10 samples collected for each type of media (10% frequency). Precision will be evaluated by calculating the RPD as follows:

$$RPD\ (\%) = [(XA-XB)/\ XM] * 100$$

Where:
XA and XB are the concentration in the sample and its duplicate, and
XM is the mean value of the concentrations in the sample and its duplicate, (XA + XB)/2

The RPD will be calculated for the field duplicates for each analytical parameter that was detected in both samples. The RPD is calculated only for those analytes that are detected at levels exceeding the method detection limits in both samples of the duplicate pair. If one of the duplicate pair is detected above the method reporting limit (MRL) and the remaining is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2MRL). It is expected that the field duplicates for solid matrices will have a RPD less than 50% for inorganic and organic parameters. If the criteria are not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data.

6.10 LABORATORY REPORTING DELIVERABLES

All data produced by the laboratory will be required to undergo several levels of review, which will include two levels of management review at the laboratory. For investigative, fill and waste

characterization samples, the laboratory will be providing a summary report in PDF format which will include the following:

- Signed cover page by Laboratory Director, Harry Behzadi.
- Laboratory Analytical Case Narrative
- Sample Summary which will include Client Identification, Laboratory Accession Number, Sampling date and Sampling Time
- Copy of the Chain-of-Custody Record
- Copy of Sample Receipt Confirmation Form
- Sample Result Forms I's
- Method Blank Summary Forms, Laboratory Control Sample Summary Forms, Matrix Spike/Matrix Spike Duplicate Forms, and Surrogate Recovery Results will be included

For fill samples and sample sets that have results below the Site specific PRG's, and no further excavation is required, CLP equivalent data packages suitable for validation will be generated by the laboratory. The laboratory will also submit a Microsoft Excel file containing the data in an electronic deliverable.

6.11 LABORATORY SAMPLE DISPOSAL

All samples will be retained under proper storage conditions for a minimum of 30 days following completion of the analytical report. The laboratory disposes of all laboratory wastes following the requirements of the Resource Conservation and Recovery Act (RCRA). Accutest has obtained and maintains a waste generator identification number, FLR00001263309002 (FLR designates the State of Florida). The Sampling Handling System is represented in **Appendix A – QAPP Worksheet #26**.

Accutest's sample management generates a sample disposal list from their LIMS (Laboratory Information System), which lists all samples whose holding period has expired. Data from each sample is compared to the hazardous waste criteria established by the Florida Department of Environmental Protection (FDEP). Samples containing constituents at concentrations above the criteria are labeled as hazardous, segregated, scheduled for removal from the facility, and disposed of as hazardous waste.

6.12 DATA VERIFICATION

The Shaw Lantdiv Chemist will verify that data has been received for all samples that have been sent to the laboratory. An evaluation of this data as defined in **Worksheet 35 and 36** will be performed to determine whether the laboratory met the QC requirements for the analytical as

stated in the analytical methods and laboratory SOPs. Analytical results will be compared to site specific PRGs for investigative remediation samples, and TCLP Maximum Contaminant Concentrations (40 CFR 261 6/96) for disposal samples as identified in **Worksheet 15**; if any results exceed these limits the Shaw Project Manager will be notified. Worksheets contained in **Appendix A (QAPP Worksheets 30, 31, 34, 35 and 36)** will be utilized to complete the data verification.

Data sets for samples that are below site specific PRGs will be validated as per the project criteria enclosed within Appendix A and using USEPA Region II functional guidelines modified SW-846 analysis. For investigative soil samples, associated field/equipment blanks, and disposal samples data validation will not be required since the use of the analytical data is to determine further excavation, and characterizing soil for disposal.

APPENDIX A
QUALITY ASSURANCE PROJECT PLAN

QAPP Worksheet #1
Title and Approval Page

Removal Action, SWMU-6, SWMU-7, AOC-J, and AOC-R Vieques, Puerto Rico
Document Title

U.S. Navy NAVFAC Atlantic
Lead Organization

Natasha Sullivan, Shaw Environmental, Inc.
Preparer's Name and Organizational Affiliation

500 E. Main Street, Suite 1630, Norfolk, VA 23510, (410) 529-7598, Natasha.Sullivan@Shawgrp.com
Preparer's Address, Telephone Number, and E-mail Address

23-March-2007
Preparation Date (Day/Month/Year)

Investigative Organization's Project Manager: _____
Signature
William L. Hughes/Shaw Environmental, Inc./
Printed Name/Organization/Date

Investigative Organization's Project QA Officer: _____
Signature
Archie Vega/Shaw Environmental, Inc./
Printed Name/Organization/Date

Lead Organization's Project Manager: _____
Signature
Kevin Cloe / U.S. Navy NAVFAC Atlantic /
Printed Name/Organization/Date

Approval Signatures: _____
Signature
Daniel Rodriquez / U.S. EPA Project Manager
Printed Name/Title/Date
U.S. EPA Region 2
Approval Authority

_____
Signature
Josefina Gonzalez/Puerto Rico Environmental Quality Board /
Printed Name/Title/Date

Other Approval Signatures: _____
Signature

_____
Printed Name/Title/Organization/Date

QAPP Worksheet #2 (UFP-QAPP Manual Section 2.2.4) -- QAPP Identifying Information

Site Number/Code: N/A
Operable Unit: N/A
Contractor Name: Shaw Environmental, Inc.
Contractor Number: N/A
Contract Title: Removal Actions – SWMU-6, SWMU-7, AOC J, and AOC R
Work Assignment Number: Task Order No. 15

1. Identify guidance used to prepare QAPP:
Uniform Federal Policy for Quality Assurance Project Plans

2. Identify regulatory program: U.S. EPA Region 2 - CERCLA

3. Identify approval entity: U.S. Environmental Protection Agency Region 2

4. The QAPP is a *project-specific* QAPP.

5. List dates of scoping sessions that were held:
April 24, 2007 and June 26, 2007

General scoping sessions:

6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Received Date
N/A	

7. List organizational partners (stakeholders) and connection with lead organization:
U.S Navy (NAVFAC Atlantic), U.S. EPA Region 2, Puerto Rico Environmental Quality Board; Department of Interior U.S. Fish and Wildlife, Municipality of Vieques.

8. List data users:
U.S Navy (NAVFAC Atlantic), U.S. EPA Region 2, Puerto Rico Environmental Quality Board; Department of Interior U.S. Fish and Wildlife, Municipality of Vieques.

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below:

Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Crosswalk to Required Documents	Optional QAPP Worksheet # in QAPP Workbook	Required Information
Project Management and Objectives			
2.1 Title and Approval Page		1	- Title and Approval Page
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information		2	- Table of Contents - QAPP Identifying Information
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet		3 4	- Distribution List - Project Personnel Sign-Off Sheet
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	SAP 3.0	5 6 7 8	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	SAP 2.1 W.P. 1.0	9 10	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria		11 12	- Site-Specific PQOs - Measurement Performance Criteria Table
			- Sources of Secondary Data

Required QAPP Element(s) and Corresponding QAPP Section(s)	Crosswalk to Required Documents	Optional QAPP Worksheet # in QAPP Workbook	Required Information
2.7 Secondary Data Evaluation		13	and Information - Secondary Data Criteria and Limitations Table
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule		14 15 16	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table
Measurement/Data Acquisition			
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	WP 2.3 WP 3.0 SAP 4.4 SAP 4.5	17 18 19 20 21 22	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/ SOP Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	SAP 6.4 SAP 6.7	23 24 25	- Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
3.3 Sample Collection Documentation, Handling, Tracking, and Custody	WP 3.0 SAP 4.4.1 SAP 5.0	26	- Sample Collection Documentation Handling, Tracking, and Custody

Required QAPP Element(s) and Corresponding QAPP Section(s)	Crosswalk to Required Documents	Optional QAPP Worksheet # in QAPP Workbook	Required Information
Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	WP 3.0 SAP 4.4.1 SAP 5.0		SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	SAP 6.8	27	- QC Samples Table - Screening/Confirmatory Analysis Decision Tree
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	WP 4.0 SAP 6.10	28 29	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs
Assessment/Oversight			
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses		30 31	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table
4.2 QA Management Reports		32	- QA Management Reports Table
4.3 Final Project Report			
Data Review			
5.1 Overview			
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation	SAP 6.12	33 34 35 36 37	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table

Required QAPP Element(s) and Corresponding QAPP Section(s)	Crosswalk to Required Documents	Optional QAPP Worksheet # in QAPP Workbook	Required Information
Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities			
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining			

QAPP Worksheet #3
Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Kevin Cloe	Navy RPM	U.S. Navy	(757) 322-4736		Kevin.Cloe@navy.mil	UFP-QAPP 116701
Anselmo Martinez	Navy ROICC	U.S. Navy	(787) 865-4066 ext.227		martineza@napr.navy.mil	
William Hughes	Project Manager	Shaw Environmental, Inc.	(757) 318-5140	(757) 363-7222	Bill.Hughes@Shawgrp.com	
Cano Hernández	Project Superintendent	Shaw Environmental, Inc.	(757) 544-7687		Cano.Hernandez@Shawgrp.com	
Archie Vega	QC Representative	Shaw Environmental, Inc.			Archie.Vega@Shawgrp.com	
Natasha Sullivan	Program Chemist	Shaw Environmental, Inc.	(410) 529-7598	(410) 529-7599	Natasha.Sullivan@Shawgrp.com	
Diana Cutt	Geologist	U.S. Environmental Protection Agency	(212) 637-4311		Cutt.Diana@epamail.epa.gov	
Oscar Díaz	Refuge Manager	U.S. Fish & Wildlife Service	(787) 741-2138 ext. 0659		Oscar.Diaz@fws.gov	
Sergio López	Investigation QC Specialist	U.S. Environmental Protection Agency	(732) 321-6778		Lopez.Sergio@epamail.epa.gov	
Felix López Arroyo	Environmental Contaminants Specialist	U.S. Fish & Wildlife Service	(787) 851-7297 ext. 226		Felix.lopez@fws.gov	
Josefina Gonzalez	Project Manager	Puerto Rico Environmental Quality Board	(787) 529-5956		Josefinagonzalez@jca.gobierno.pr	
Bud Oliveira	Refuge Supervisor	U.S. Fish & Wildlife Service	(404) 679-7155		Bud.Oliveira@fws.gov	
Chris Penny	Eastern Vieques Project Coordinator	U.S. Navy	(757)322-4815		Christopher.Penny@navy.mil	
Mindy Pensak	Natural Resource Specialist	U.S. Environmental Protection Agency	(732) 321-6705		Pensak.Mindy@epamail.epa.gov	
Madeline Rivera	Vieques Navy Environmental Representative	U.S. Navy	(787) 865-4152		Riveramad@napr.navy.mil	

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Daniel Rodríguez	Project Manager	U.S. Environmental Protection Agency	(787) 741-5201		Rodriguez.daniel@epa.gov	
Katarina Rutkowski	Technical Support Consultant for EQB	TRC Environmental Corporation	(860) 298-6202		krutkowski@trcsolutions.com	
Susan Silander		U.S. Fish & Wildlife	(787) 851-7258 Ext. 38		Susan.Silander@fws.gov	
Michael Sivak	Risk Assessor	U.S. Environmental Protection Agency	(212) 637-4310		Sivak.Michael@epamail.epa.gov	
Andrew Smyth	Technical Support Consultant for EQB	TRC Environmental	(978) 656-3568		ASmyth@trcsolutions.com	
Carl Soderberg	Puerto Rico Manager	U.S. Environmental Protection Agency	(787) 977-5814		Soderberg.Carl@epa.gov	
Diane Wehner	Natural Resource Manager	National Oceanic & Atmospheric Administration	(732) 872-3030		Diane.wehner@noaa.gov	
Erik Malarek	Project Data Validator/Chemist	Shaw Environmental, Inc.	(410) 612-6322		emalarek@shawgrp.com	
Harry Behazadi	Laboratory Manager	Accutest Orlando	(407) 425-6700	(407) 425-0707	harryb@accutest.com	
Svetlana Izosimova	Laboratory QAO	Accutest Orlando	(407) 425-6700	(407) 425-0707	svetlanl@accutest.com	

QAPP Worksheet #4 (UFP-QAPP Manual Section 2.3.2) -- Project Personnel Sign-Off Sheet

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

Organization: Shaw Environmental, Inc

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
William Hughes	Project Manager	757.640.6937 bill.hughes@shawgrp.com		
Cano Hernandez	Project Superintendent	787.630.9883 cano.hernandez@shawgrp.com		
Archie Vega	QC Manager	479.651.1010 Archie.vega@shawgrp.com		
William Deane	Project Engineer	757.640.6956 William.deane@shawgrp.com		
Natasha Sullivan	Program Chemist	410.529.7598 Natasha.sullivan@shawgrp.com		
Eric Malarek	Project Data Validator	410.612.6322 eric.malarek@shawgrp.com		

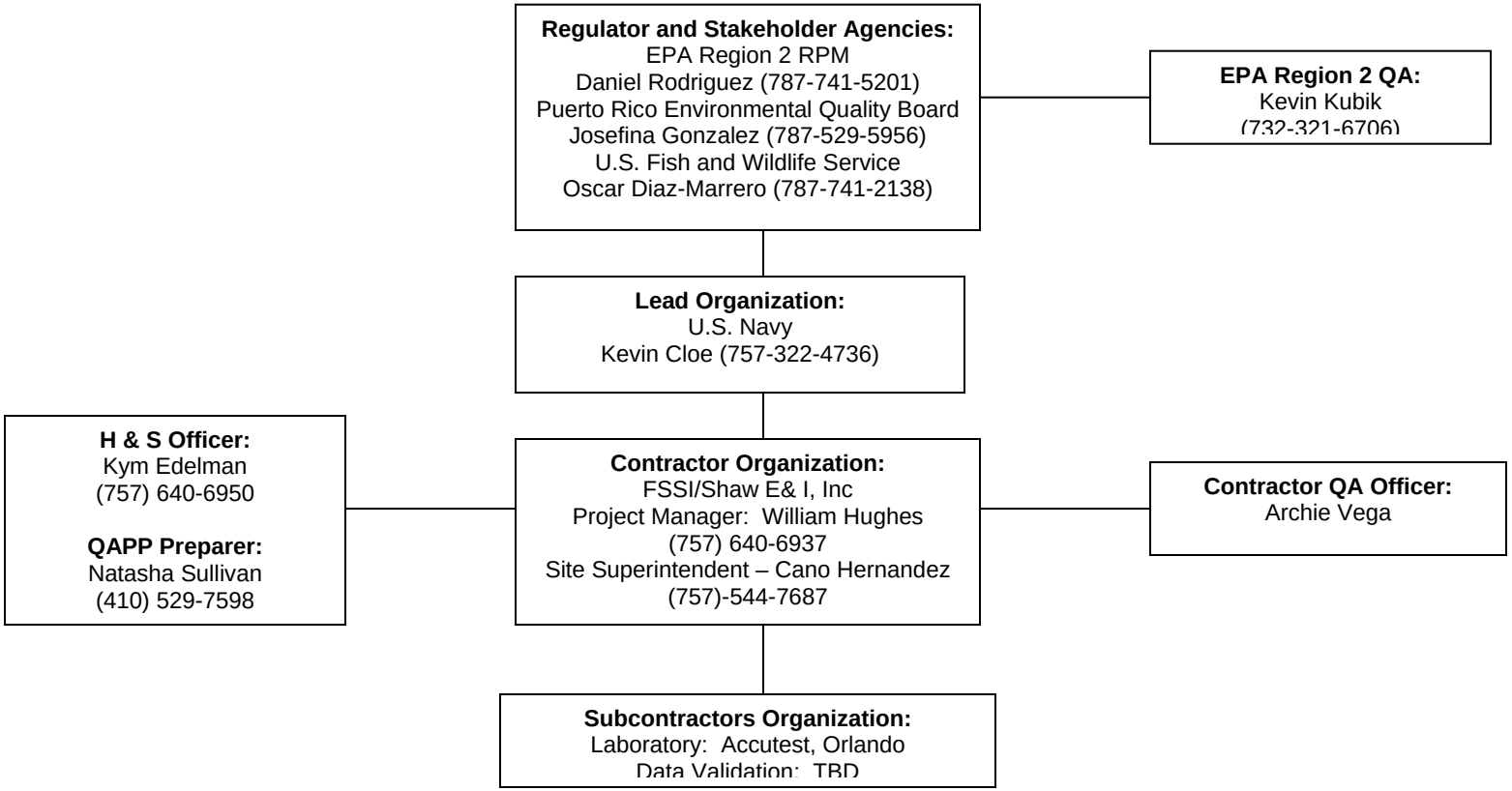
Organization: Navy/Puerto Rico EQB/EPA/USFWS

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Kevin Cloe	RPM –Navy	757.322.4736 Kevin.cloe@navy.mil		
Josefina Gonzalez	Project Manager - PREQB	787.529.5956 Josefinagonzalez@jca.gobierno.pr		
Daniel Rodriguez	Project Manager – EPA	787.741.5201 Rodriguez.daniel@epa.gov		
Richard Henry	Project Manager –USFWS	Richard.henry@fws.gov		

Organization: Accutest Laboratories - Orlando

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Harry Behazadi	Laboratory Manager	407.425.6200		
Svetlana Izosimova	Laboratory QC Specialist	407.425.6200		
Heather Wandry	Laboratory Project Manager	407.425.6200		

QAPP Worksheet #5
Project Organizational Chart



QAPP Worksheet #6
Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Point of Contact with EPA RPM	NAVFAC Atlantic RPM	Kevin Cloe	(757) 444-3826	All materials and information pertaining to the project will be forwarded to Daniel Rodríguez by Kevin Cloe.
Manage all project phases	Contractor Project Manager	William Hughes	(757) 640-6937	Will be the Shaw Environmental liaison to Kevin Cloe.
QAPP Changes in the field	Project Superintendent	Cano Hernández	(757) 544-7687	Notify Bill Hughes by phone and e-mail of changes to the QAPP made in the field and the reasons within 24 hours. Changes will be documented in a technical directive.
Daily Field Progress Reports	Project Superintendent	Cano Hernández	(757) 544-7687	Cano will provide daily reports to Bill Hughes via fax or e-mail.
Reporting Lab Data Quality Issues	Laboratory Project Manager	Heather Wandrey	(407) 425-6700	All QA/QC issues with laboratory analyses will be reported to Natasha Sullivan within 2 business days.
Field Corrective Actions	QC Representative	Archie Vega	(757) 393-4807	The need for corrective action for field issues will be reported by Archie and documented in a technical directive.
Lab Analytical Corrective Actions	Shaw Program Chemist	Natasha Sullivan	(410) 529-7598	The need for corrective actions for analytical issues will be determined by Natasha Sullivan
Release of Analytical Data	Shaw Program Chemist	Natasha Sullivan	(410) 529-7598	No analytical data will be released until validation is completed and Natasha Sullivan has approved the release.
QAPP Amendments	EPA Remedial Project Manager	Daniel Rodríguez	(787) 741-5201	Any major changes to the QAPP must be approved by Daniel Rodríguez before the changes can be implemented. Mr. Rodríguez may need solicit consensus from the Partnering Group.

QAPP Worksheet #7
Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Kevin Cloe	Remedial Project Manager	Navy	Oversees the project and serves as the liaison between the Navy and U.S. EPA and Puerto Rico EQB.	
Madeline Rivera	Vieques Navy Environmental Representative	Navy	Oversees construction activities in the field and responds to RPM.	
William Hughes	Project Manager	Shaw Environmental, Inc.	Manages project and coordinates between Navy and Shaw Environmental, Inc.	M.S. Geology, Certified Professional Geologist Virginia, 16 yrs exp.
Cano Hernández	Project Superintendent	Shaw Environmental, Inc.	Responsible of supervising all field activities and supervises field sampling.	Construction professional, 20 years of experience. Procurement of equipment and materials, project health and safety compliance, budgeting, scheduling, and client liaison.
Archie Vega	QC Representative	Shaw Environmental, Inc.	QA oversight on the project.	
Natasha Sullivan	Shaw Program Chemist	Shaw Environmental, Inc.	Serves as liaison between Shaw Environmental and laboratory.	17 years of experience working in all department of an analytical laboratory including sample management office. data reporting, and project management
Kym Edelman	Health & Safety Manager	Shaw Environmental, Inc.	Oversees the H&S for field activities.	B.S. Environmental Health, 17 years of experience.

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Harry Behazadi	Laboratory Manager	Accutest Orlando	Manages the generation of analytical data.	Ph.D. Chemistry, 24 years of experience
Svetlana Izosimova	Laboratory QA Officer	Accutest Orlando	Performs lab QA oversight.	M.S. Chemical Engineering Ph.D. Colloid Chemistry, 16 years experience
Heather Wandrey	Laboratory Project Manager	Accutest Orlando	Serves as laboratory project manager, coordinates incoming work with staff, and communicates with Shaw.	B.A. Environmental Studies, 12 years experience

QAPP Worksheet #8
Special Personnel Training Requirements Table

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates[1]
Site Superintendent	• 40-Hour Hazardous Waste Site Worker • 24-Hour Initial On-site Field Experience • 8-Hour Hazardous Waste Site Supervisor Training • 10-Hour OSHA Construction Site Worker Safety Training	Varies[2]	Varies[2]	All	Cano Hernandez, William Hughes / Shaw Environmental	Certificates available upon request and documentation files are maintained on site.
Field Technicians and Equipment Operators	• 40-Hour Hazardous Waste Site Worker • 24-Hour Initial On-site Field Experience	Varies[2]	Varies[2]	All	Field Technicians, all levels; Equipment Operators, all levels	Shaw Environmental: Certificates available upon request. Certification files are maintained on site.
Any personnel working in the exclusion area or area of contamination on the project site	• 40-Hour Hazardous Waste Site Worker • 24-Hour Initial On-site Field Experience	Varies[2]	Varies[2]	All	Varies by trade	Shaw Environmental: Certificates available upon request. Certification files are maintained on site.

[1] Training records and/or certificates are available at the Shaw Norfolk Office, Norfolk Virginia

[2] The training provider and date of training may vary from person to person, this is indicated on the individual's certificate.

QAPP Worksheet #9
Project Scoping Session Participants Sheet

Project Name: Removal Action, SWMU-6, SWMU-7, AOC-J, and AOC-R **Projected Date(s) of Sampling:** TBD **Project Manager:** Bill Hughes	**Site Name:** Removal Action, SWMU-6, SWMU-7, AOC-J, and AOC-R **Site Location**: Vieques, Puerto Rico

Date of Session: 4/27/07
Scoping Session Purpose: ERP Tech Meeting Work Plan Analysis

Name	Organization	Title	Phone #	E-mail Address	Project Role
Richard Henry	USFWS				
Oscar Diaz	USFWS				
James Oland	USFWS				
Bill Tucker	MACTEC				
Wilmarie Rivera	PREQB				
Yarissa Martinez	PREQB				
Katarina Rutkowski	TRC				
Danny Rodriguez	EPA				
Diana Cutt	EPA				
Mindy Pensak	EPA				
Michael Sivak	EPA				
Michael P. Connelly	MOV				
Kevin Cloe	NAVFAC				
Madeline Rivera	NAVFAC				
Diane Wehner	NOAA				
Gayle Waldron	Management Edge				
Barrie Selcoe	CH2M Hill				
Brett Doerr	CH2H Hill				

Comments/Decisions:

Action Items:

Consensus Decisions:

Discussing the Waste Characterization and Confirmatory Sampling Protocols for AOC J, AOC R, SWMU 6, and SWMU 7

The revised removal action waste characterization and confirmatory sampling protocol for AOC J, AOC R, SWMU 6, and SWMU 7 submitted to the agencies on November 3, 2006 was discussed during the December 7, 2006 ERP Technical Subcommittee meeting, as summarized below.

Discussion was held regarding how the waste would be characterized and a determination of how much removal would take place. According to the revised draft protocol submitted to the agencies, the removal would be based on:

1. Vertical and horizontal extent of debris (to ensure debris is removed), and

2. Calculated cleanup levels calculated based on human health and ecological risk assessments performed on the waste profiling data (to ensure site is protective of human health and the environment)

One of the primary concerns expressed by the regulatory agencies was that the number and types of analyses of confirmatory samples were proposed to be contingent upon the risk assessment performed on the waste profiling data. This concern is based on the probable heterogeneous nature of the debris. In other words, while profiling is necessary, it may not completely characterize the area of removal due to the heterogeneity, so confirmatory samples should be collected regardless of any waste profiling risk assessment findings, the locations of which can specifically target certain locations based on observations made during the removals. The regulatory agencies further stated that again because of the heterogeneity and associated post-removal confirmatory sampling, new risk assessments should be conducted on the post-removal confirmatory soil data. The regulatory agencies proposed that the profiling be done in only those areas suspected to best represent the highest levels of contamination and that confirmatory sampling be done across the area of excavation, with potential bias based on removal action observations (e.g., location of drum or some other suspected release area).

Another concern stated by the regulatory agencies is that the waste profiling is proposed to be done in-situ instead of ex-situ. The Navy stated that it is not practical to characterize the waste ex-situ because of the very large quantity of soil/debris to be removed. It is not practical to manage this quantity of material in a pile or in roll-offs. The pile would be too massive to be able to control from wind, precipitation runoff, thievery, etc. while the profiling is performed. The risk to unintended contaminant transport is too great to justify ex-situ profiling. The Subcommittee concurred that in-situ profiling is acceptable as long as confirmatory sampling is conducted at all sites.

EPA noted that it is possible that some debris is not visible at the ground surface, being either covered during the disposal activities or sinking into the sediment (at SWMU 6). USFWS recommended that within the general disposal area, test excavations be made at various locations where waste is not visible to confirm or refute its presence beneath the land surface. USFWS also noted that field techniques such as PIDs and XRF could be used to help guide the removal. CH2M HILL noted that a PID will be used, but that there are too many uncertainties associated with XRF that could be misleading, which would hinder its use for real-time guidance of a removal action. EQB noted that geophysical surveys have been conducted at AOC J, SWMU 6, and SWMU 7 to identify the extent of metallic debris.

EPA stated that the profiling needs to be done not only to determine whether the material is hazardous or not (i.e., TCLP analysis), but also to determine whether it does not pose a risk for use as daily cover at the landfill (i.e., TCL/TAL analysis). EQB noted that although the Navy stated that large debris will be separated from the soil, there will be small pieces of debris mixed in with the soil. CH2M HILL noted that this is common for daily cover, and it should be kept in mind that the landfill is currently using gravel, so the use of material from the removal actions, if deemed acceptable, will improve the daily cover on the landfill. It was further noted that the very small debris that is on the order of the size of soil particles will be captured in the sample containers and analyzed as part of the sample matrix.

It was recognized that there is a possibility that something unexpected will be encountered during the removal action. In other words, the visible debris should be a good representation of what will be found upon excavation, but the possibility exists that something unexpected (e.g., drum, evidence of a release) will be encountered. Therefore, contingency language should be added to the removal action that permits additional digging, material segregation and additional characterization of the segregated material, and focused confirmatory sampling in case this is encountered. This will help to identify if there is material that does not meet the original waste profile and to ensure it is properly disposed of. A rolloff(s) should be staged at each site for the contingency segregation.

EQB noted that the removal action work plan needs to provide specifics of how the debris will be segregated from the soil for disposal purposes.

USFWS commented on the concern about road access to the refuge and other parts of the island due to heavy machinery being on the roads during the removal. It was stated that the removal action work plan should include a communication plan regarding notifying USFWS and the public about the heavy machinery on the roads during the removal action. USFW may close access during that time.

It was concurred that to facilitate agency concurrence on the areas to be sampled for waste profiling, a site visit to each of the removal action sites will be made to select mutually acceptable sampling locations. Vegetation clearance will be done prior to this site visit.

NOAA indicated that surface water/sediment sampling may be requested as part of the SWMU 6 confirmatory protocol.

To facilitate conceptualizing the removal process, a series of flip charts were posted on the wall and the various steps of the process enumerated, as follows:

<u>Step 1 – Coordinate w/FWS</u>

1. Vegetation clearance/UXO support (entire debris area)

2. Visual confirmation of debris area
3. Red flag RI sample points to help visualize where data exist
4. Develop waste profiling sample grid
   - bias toward observed debris
   - sampling to guide removal activity and waste disposal
   - team site visit – agree upon sample locations – including metal detector

Step 2 – Sampling

- Conduct sampling in accordance with final removal action work plan

Step 3

1. Waste characterization
   - TCLP
   - TPH
   - TAL/TCL
2. Present validated data to team
3. Perform risk assessments for internal (Navy) use to guide risk-based removal limits
4. Discuss and agree on waste characterization and disposal
5. Incorporate any changes into removal action work plan
6. Agree on confirmatory sample locations
7. Develop preliminary confirmatory sampling plan (location and number of samples)

Step 4 - Coordinate w/FWS and municipality

1. Impacts to refuge access
2. Visitors
3. Infrastructure
4. Access/Logistics
5. Timeline for removal

Step 5 – Commence Removal Action (follow Work Plan)

- Provide contingency plans in Work Plan in case unexpected debris (e.g., drum, evidence of release, UXO, etc.) is encountered
  - Additional segregation
  - Additional sampling
  - Additional excavation
  - UXO handling
  - Decision matrix for when to sample and when to continue excavation
- Collect confirmatory samples

Step 6 – Team Meeting to Review Removal Action Results

It was concurred that the information above will be used to revise the removal action work plan, including its associated UFP-QAPP, which will then be resubmitted to the agencies. Navy will notify the agencies when submittal of the revised removal action work plan is anticipated.

Date of Session: 6/26/07
Scoping Session Purpose: ERP Tech Meeting Work Plan Comment Response

Name	Organization	Title	Phone #	E-mail Address	Project Role
Richard Henry	USFWS				
Oscar Diaz	USFWS				
Pete Jerome	USFWS				
Oscar Diaz	USFWS				
Susan Silander	USFWS				
Bill Tucker	MACTEC				
Wilmarie Rivera	PREQB				
Yarissa Martinez	PREQB				
Enid Y Villegas	PREQB				
Adam Balogh	TRC				
Danny Rodriguez	EPA				
Kevin Cloe	NAVFAC				
Madeline Rivera (Partial)	NAVFAC				
Johnny Noles	NAVFAC				
Tom Brosnan	NOAA				
Diane Wehner	NOAA				
William Deane	Shaw				
Gayle Waldron	Management Edge				
Susana Struve (partial)	CH2M Hill				
Brett Doerr	CH2H Hill				

Comments/Decisions:

Revised Draft Removal Action Work Plan for AOC J, AOC R, SWMU 6, and SWMU 7

William Deane provided an overview of the revisions made to the draft work plan in response to the regulatory agency comments in order to be able to focus the meeting discussion on the larger, main points of the work plan. A general summary of the discussion is as follows:

- Team Consensus Statement – The ERP Technical Subcommittee agreed with the waste profiling sampling and analysis protocol stated in Worksheet 17. This includes the ability to bias the sampling locations toward areas of obvious debris or contamination, as determined during the pre-waste profiling sampling team visit.
- Shaw addressed all comments referencing chemical analyses and SOPs requested by EQB.
- Contingency language was added to each applicable section of the work plan and QAPP to clarify the procedure for unanticipated discovery during removal, including language that says notification shall be made to EPA, EQB, and USFWS upon discovery of any unknown or unanticipated items.

- Revisions were made to the work plan and QAPP, as applicable, to ensure consistency between both.
- Shaw will add text clarifying that groundwater sampling will be conducted, but will indicate that the sampling will be conducted by CH2M HILL in accordance with the Master QAPP.
- Excavation procedure – Shaw will not stage material outside of the contaminated areas and, therefore, there is no need to provide baseline testing or post-excavation sampling under the excavation staging piles, other than the post-excavation confirmatory sampling.
- USFWS voiced concern with the notification to the Municipality of Vieques. The Power and Water districts have planned activities within the anticipated work areas, so coordination should be with these organizations prior to the work. It was concurred that the notification of intent should be distributed within the next month to allow for Municipality interaction
- USFWS noted that Martineau Bay Resort (MBR) will be performing wetlands restoration work near or within the planned excavation areas (east and west of SWMU 6) that will involve construction of swales and dredging of canals. USFWS will provide the MBR contact information to Shaw. Shaw will provide notice of intent to MBR.
- Shaw will add text to the contingency portion of the work plan that describes the process that will be followed for discovery of unanticipated munitions items (i.e., not including the items on the ground surface that are currently known). The text will indicate the need for UXO techs to adequately determine the course of action for the unanticipated items. Notification to the regulatory agencies upon discovery of unanticipated munitions items will follow the contingency process documented in the work plan.
- The current plan is to evaluate the waste profiling data using industrial standards to determine if the soil can be used as daily cover. There is concern by EQB and EPA that soil staged at the landfill for daily cover will be taken by residents and used at their homes. A general discussion of security for the soil staged at the landfill followed, where it was noted that security fencing may be feasible to install at the landfill, but that the operators could still unlock the gate for the residents to take soil.
- The Subcommittee agreed that if use consistent with remaining in place scenario was added and the waste profiling risk assessment demonstrated that this use was acceptable, the soil could be left at the sites once the debris was removed.
- Shaw noted that the landfill has agreed to accept the waste based on receipt of soil for daily cover use. It is possible if the soil meets industrial standards, but the agencies do not permit the use of the soil as daily cover, the landfill will not accept the waste. EQB feels that although the landfill has stated that it will use the soil as daily cover that it will not and that the soil will end up being taken by residents. It was noted that the landfill is currently out of compliance, including daily cover for waste, and that being able to use this soil as daily cover would help the landfill with compliance. A question was asked if the landfill could be forced to secure the soil and use it as daily cover, under the threat of action due to current out-of-compliance status. EQB stated this could not be done. The Municipality should be consulted in this issue to discuss their willingness to ensure the use the soil as daily cover and secure it from being taken by residents. Pedro Tejada/RWEC will contact the

Municipality's landfill point of contact and discuss their willingness to ensure the soil is used as daily cover and potential methods for securing it from the public. Pedro will also determine if the landfill is willing accept the debris if they don't get the soil as daily cover. This information will be documented in a memorandum of understanding, which Shaw will then discuss with the Navy

- The protocol for post-removal confirmatory sampling/analysis as written in the work plan is acceptable to the ERP Technical Subcommittee. Further, the Subcommittee concurred that the concept of interim sampling/analysis not being subject to agency review (other than for informational purposes only) is acceptable because it is to be used as a guide for the contractor to determine when to stop excavating and take post-removal confirmatory samples that will be used in the post-removal risk assessment, which will be subject to agency review.
- EPA stated that it would like to see text regarding installation of wells within the areas of debris once removed. The Navy stated that the existing wells on the downgradient perimeter of the debris areas are sufficient for determining the groundwater quality associated with the debris areas because the waste has been there long enough for the groundwater in these wells to be representative of the groundwater quality associated with any potential leaching from the debris. Further, it was stated that it is common practice to install wells at the downgradient boundary of waste for this type of monitoring (e.g., landfill regulations). It was agreed that some contingency text will be added to the work plan that states if, during removal, visual evidence of a potential point source of contamination that may not have been captured by the existing downgradient wells is observed, a well intended to characterize the area will be installed at a location concurred upon by the Navy and regulatory agencies. The contingency portions of the decision flow charts will be revised to reflect this.
- EPA and EQB questioned the Navy on how staging can be done at SWMU 6 to allow the water to drain from the debris without potentially contaminating previously uncontaminated areas. Shaw stated that they will be using a leap-frog approach starting at the furthest point away from the road and working toward the road, during which if debris needs to be staged to drain, it will be placed on an area that will be excavated as the removal proceeds toward the road. The text does not need to be modified; this was just a question for clarification.
- NOAA stated that the NOAA National Marine Fisheries Service (NMFS) needs to be notified of the pending work and an Essential Fish Habitat assessment done if it hasn't already been done.

Following the discussion above, a tentative schedule for the removal action and associated activities (e.g., work plan finalization, waste profiling, etc.) was developed by the Subcommittee. The tentative schedule is attached to these minutes.

Action Items:

1. **Pedro Tejada/RWEC will distribute notification of intent for removal action to Municipality within 1 month.**

2. **USFWS will provide Shaw with the MBR contact information regarding the planned wetland restoration project.**
3. **Pedro Tejada/RWEC will provide notification of intent to the MBR contact.**
4. **Pedro Tejada/RWEC will contact the Municipality's landfill point of contact and discuss their willingness to ensure the soil is used as daily cover and potential methods for securing it from the public. Pedro will also determine if the landfill is willing accept the debris if they don't get the soil as daily cover. This information will be documented in a memorandum of understanding, which Shaw will then discuss with the Navy.**
5. **Shaw will revise the removal work plan as stated above.**

Consensus Decisions:

QAPP Worksheet #10
Problem Definition

The problem to be addressed by the project:

Trash and debris was dumped at the four sites and must be removed in order to ensure it does not pose a future source of contamination. The overall goal of these removal actions is to eliminate the debris and ensure residual media concentrations are protective of human health and the environment.

The environmental questions being asked:

1. Is any of the impacted soil or debris from the site hazardous?
2. If the material or soil is non-hazardous, can it be used as intermittent cover on the Vieques Municipal Landfill?
3. If the soil is non-hazardous, can it remain onsite?
4. Following the removal of the impacted soil and debris, are residual media concentrations protective of human health and the environment?

Observations from any site reconnaissance reports:

Debris is present at all four sites. Existing reports suggest sites do not pose an unacceptable human health or ecological risk, but there is uncertainty in the conclusion because samples were not collected directly though the debris piles. It is this uncertainty and the potential for the debris to act as a future source of contamination that warrants the removal action.

A synopsis of secondary data or information from site reports:

See above.

The possible classes of contaminants and the affected matrices:

Existing data do not suggest constituent concentrations in site media pose an unacceptable risk to human health of the environment. However because samples were not collected directly through waste pile, all classes of contaminants are potentially present. Therefore, waste characterization and confirmatory samples will be analyzed for Target Compound List (TCL) volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), pesticides, polychlorinated biphenyls (PCBs), Target Analyte List (TAL) metals, and explosives. Waste characterization and confirmatory samples will be collected from media in direct contact with the debris to evaluate affects.

The rationale for inclusion of chemical and non-chemical analyses:

See Above

QAPP Worksheet #10

Problem Definition (continued)

Project decision conditions (If..., then...@ statements):

Waste Characterization Soil Samples

Soil samples will be collected from the material to be excavated prior to mobilization. The samples will be analyzed for TCL, VOCs, TCL, SVOCs, TAL metals, TCL pesticides/PCBs, total petroleum hydrocarbons (TPH), and explosives. The samples will also be analyzed for full toxicity leaching characteristic procedure (TCLP) parameters, and reactivity, corrosivity, and ignitability (RCI).

IF the TCLP/RCI concentrations ***exceed*** regulatory limits[1], THEN the soil and debris will be deemed a hazardous material and will be transported to a permitted hazardous waste facility.

IF the TCLP/RCI concentrations ***do not exceed*** regulatory limits[1], THEN the **debris** will be deemed not to be a hazardous material. The debris will be segregated into trash and recyclable material. IF the debris is deemed recyclable, THEN the debris will be transported to a permitted recycling facility. IF the debris is classified as trash, THEN it will be transported to a permitted non-hazardous landfill.

IF the TCLP/RCI concentrations ***do not exceed*** regulatory limits[1], THEN the **soil** will be deemed not to be hazardous material and the soil data will be used to determine whether the soil can be used as daily cover or remain on site, as follows:

IF no unacceptable hazards or risks are identified for the proposed land use based upon the post waste characterization sampling risk assessment, THEN the soil can remain onsite. IF there are unacceptable risks associated with the proposed land use, THEN the soil will be evaluated for disposal options as follows:

IF no unacceptable hazards or risks are identified via the human health risk assessment conducted with TAL/TCL/explosives soil data (using industrial PRGs as screening criteria and a maintenance worker exposure scenario) over that of background, THEN the soil will be used as daily cover at the Vieques Municipal Landfill. If unacceptable hazards or risks are identified via the above process, THEN the soil will be disposed in the Vieques Municipal Landfill.

[1]Project action limits (regulatory limits) are based upon USEPA TCLP Maximum Concentration of Contaminants 40CFR 261 (June, 1996). Specific limits can be found in Worksheet 15

Project decision conditions (If..., then...@ statements) cont'd:

Confirmation Soil Samples
Following the removal of debris and potentially contaminated soil, confirmatory samples will be collected in accordance with the confirmatory protocol concurred upon by the agencies and included as an addendum to the removal action work plan. The confirmatory samples will be used to evaluate residual risk via the HHRA and ERA protocols in the Master QAPP (CH2M Hill, February 2007).

IF the results of the risk assessments indicate no unacceptable risks or hazards are present (above background), THEN no further excavation will be conducted and the removal action report will be prepared and submitted for regulatory review.

IF the results of the risk assessment(s) indicate unacceptable risks or hazards are present, THEN additional excavation will be conducted in the area where contaminants of concern (COCs) are contributing to unacceptable risk or hazards. For the purposes of determining whether unacceptable risks or hazards are present, the residential scenario (i.e. unrestricted land use) will be initially evaluated. However, the Navy may choose to utilize a more realistic and restrictive land use scenario for making the ultimate determination of risks or hazards for excavation purposes.

Soil Characteristics
It is anticipated that varying soil conditions will be encountered that will effect the excavation and transportation of the soil.

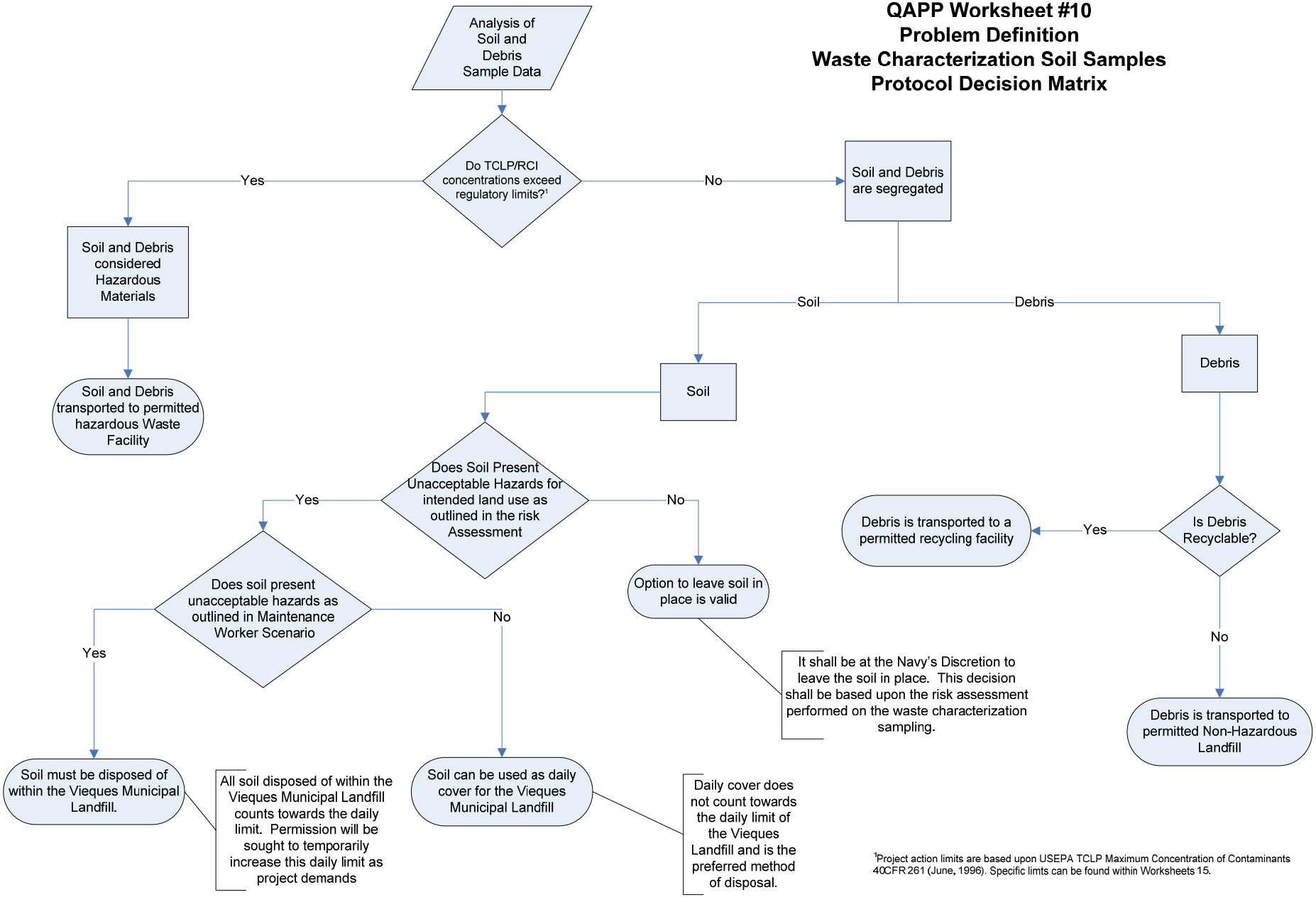
IF the soil is saturated from existing bodies of water, or from a groundwater source, THEN a slotted excavation bucket will be used to allow for the water to flow through the excavated soils out of the bucket. IF soils are not thoroughly dewatered through this process, the soils will be placed atop existing temporary daily piles to allow for the water to drain out of the soils with the aid of gravitational forces.

Contingency Planning
There is a possibility of encountering unexpected material, soils, debris, UXO ordinance, and others that may present additional exposures to human health factors, during the removal actions.

IF unexpected materials are encountered notification shall be made to the NTR, who shall notify the regulatory agencies and the property owners, THEN the areas immediately surrounding the materials will be excavated and segregated from the remaining soils in a roll-off container. The materials shall be characterized and sampled to establish their identity. IF the materials fall within the established waste profile for the removal actions, THEN the materials will be handled as described above. IF the materials do not fall within the established waste profile, THEN the materials will remain segregated and a Navy representative will be consulted to determine the appropriate handling, transportation, and disposal of this material, in consultation with the EPA and Puerto Rico EQB.

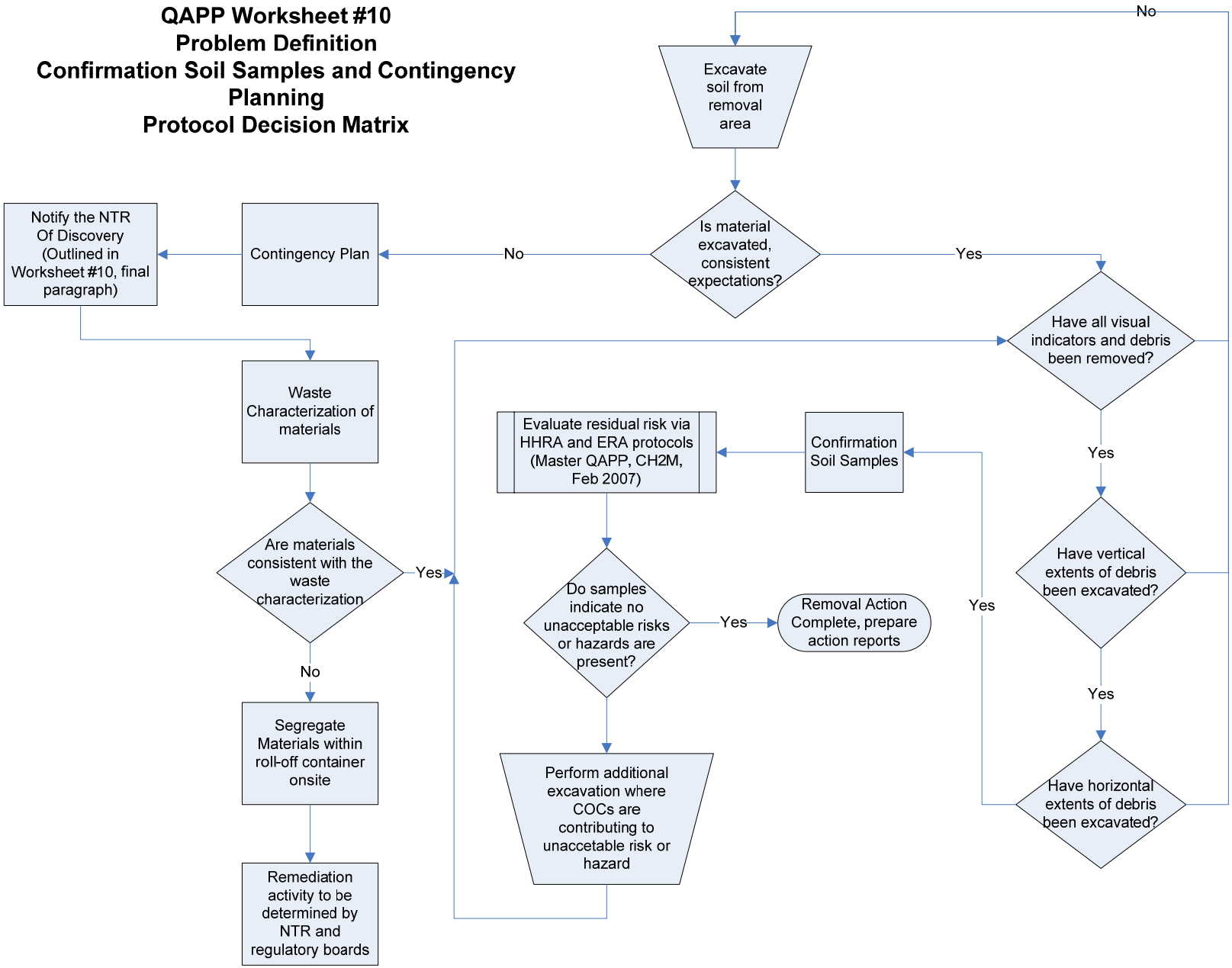
[1]Project action limits (regulatory limits) are based upon USEPA TCLP Maximum Concentration of Contaminants 40CFR 261 (June, 1996). Specific limits can be found in Worksheet 15

QAPP Worksheet #10
Problem Definition
Waste Characterization Soil Samples
Protocol Decision Matrix



[1]Project action limits are based upon USEPA TCLP Maximum Concentration of Contaminants 40CFR 261 (June, 1996). Specific limts can be found within Worksheets 15.

QAPP Worksheet #10
Problem Definition
Confirmation Soil Samples and Contingency Planning
Protocol Decision Matrix



QAPP Worksheet #11 (UFP-QAPP Manual Section 2.6.1) -- Project Quality Objectives/Systematic Planning Process Statements

Use this worksheet to develop project quality objectives (PQOs) in terms of type, quantity, and quality of data determined using a systematic planning process. Provide a detailed discussion of PQOs in the QAPP. List the PQOs in the form of qualitative and quantitative statements. These statements should answer questions such as those listed below. These questions are examples only, however; they are neither inclusive nor appropriate for all projects.

Who will use the data?
Confirmation Samples:
Shaw E & I will use the confirmation analyses to verify that the impacted soils from site have achieved all remedial action goals.
Waste Characterization Samples:
Shaw E&I will use the sample results from the waste profiling soil samples to ensure proper disposal.

What will the data be used for?
Confirmation Samples:
Shaw E & I will use the confirmation analyses to verify that the impacted soil from the site has achieved all remedial goals. Following removal of the soils Shaw will collect confirmation soil samples in accordance with the analytical methods listed in Worksheet 19 for analysis; from the bottom of the excavation to verify that the impacted soils the remediation goals.

The confirmatory data will be used to perform residual risk assessments as described in the protocols in the Master QAPP (CH2M Hill, February 2007)

Waste Characterization Samples:
Shaw will use the data to analyze the soil and debris to determine the appropriate location and method for disposal, at an approved disposal facility, as daily cover for the Vieques Municipal Landfill, or to be left onsite. See Worksheet 17 for disposal criteria.

What types of data are needed (matrix, target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)?
The site sampling at this site will include the following:
- Standard Operating Procedures for sample collection, handling, sample preparation, and analytical methods.

For sample collection and handling refer to worksheet 21 for field sampling SOPs.

All sample analysis will be performed by an off-site laboratory. Refer to Worksheet 19 for sample types, matrices, analytical groups and methods, and laboratory SOPs.

How "good" does the data need to be in order to support the environmental decision?
Waste profiling data needs to be good enough to sufficiently characterize the material (debris and soil) to determine if it will be disposed of as hazardous or non-hazardous and, if non-hazardous, if the soil can be used as daily cover at the Vieques Municipal Landfill or left in place.

Confirmatory data needs to be good enough to ensure the residual media concentrations are protective of human health and the environment.

How much data are needed (number of samples for each analytical group, matrix, and concentration)?
Waste Characterization: Waste characterization soil samples for analysis of Target Compound List (TCL), Semi-Volatile Organic Compounds (SVOCs), pesticides, and polychlorinated biphenyls (PCBs), explosives, and Target Analyte List (TAL) metals will be collected at an approximate frequency of 1 three-point composite sample (from the vertical extent of the waste) per 2000 square feet of the debris area. A photo-ionization detector (PID), or similar, will be used to screen the vertical debris interval and each of the composite components shall be collected from the location of the highest PID reading (or visual staining in the absence of an elevated PID reading) for TCL VOC's, TCLP VOC's, and GRO. Samples will be biased toward areas of observable debris and will include previously collected (e.g. RI) sample data. If appropriate, all parties shall agree if additional samples are needed if additional evidence of a release is observed. This shall be at the discretion of the Navy, and agreed upon by all parties. The Navy and regulatory agencies will perform a site visit to concur upon the number and locations of waste characterization samples.

Confirmation Samples: The number of confirmatory samples will be determined following the waste characterization effort and documented in an addendum to the removal action work plan. However, for planning purposes the confirmation samples to be taken match the number of waste characterizations samples. The actual number of confirmation samples may increase or decrease based upon the removal action findings.

Where, when, and how should the data be collected/generated?

Waste Characterization sampling will be taken prior to excavation activities, before disposal.

Confirmation samples will be collected once the debris and visually contaminated soil have been removed. The data will be collected from the locations concurred upon by the agencies and documented in an addendum to the work plan. Sample collection procedures are documented in Worksheet 21, of this document.

Who will collect and generate the data?
On site Shaw personnel will collect the samples and Accutest Laboratories, Inc. will analyze the samples as per the methods contained in Worksheet 19 for the appropriate sample type.

What is the Completeness Goal?
Completeness is defined as the percentage of measurements that are judge to be useable compared to the total number of measurements made. The objective of the overall project completeness goal is set at 95% usable data. The goal is inclusive of both laboratory analytical data and the project field sampling program. Project completeness is subject to change with future changes in scope of work pending site condition changes and the results of the residual risk assessment to be performed post waste characterization profiling.

How will the data be reported?
Refer to Worksheet 17 of this document.

How will the data be archived?
Upon completion of all field activities, Shaw will prepare a closeout report documenting site activities and reporting all data. The analytical reports will be included in the Final closeout report. All analytical reports and closeout reports will be stored on the Shaw server in PDF format.

QAPP Worksheet #15.5 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil/Sediment
Analytical Group: TCL Pesticides Organic - SW-846 3550B/8081A
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Aldrin	309-00-2	100	1.7	Not Available in Method	Not Available in Method	0.67	1.7	45-140	45-140	30	NA	50
alpha-BHC	319-84-6	360	1.7	Not Available in Method	Not Available in Method	0.67	1.7	60-125	60-125	30	NA	50
beta-BHC	319-85-7	1300	1.7	Not Available in Method	Not Available in Method	0.67	1.7	60-125	60-125	30	NA	50
delta-BHC[1]	319-86-8	1700	1.7	Not Available in Method	Not Available in Method	0.67	1.7	55-130	55-130	30	NA	50
gamma-BHC (Lindane)	58-89-9	1700	1.7	Not Available in Method	Not Available in Method	0.67	1.7	60-125	60-125	30	NA	50
alpha-Chlordane	5103-71-9	6500	1.7	Not Available in Method	Not Available in Method	0.67	1.7	65-120	65-120	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
gamma-Chlordane	5103-74-2	6500	1.7	Not Available in Method	Not Available in Method	0.67	1.7	65-125	65-125	30	NA	50
Dieldrin	60-57-1	110	1.7	Not Available in Method	Not Available in Method	0.67	1.7	65-125	65-125	30	NA	50
4,4'-DDD	72-54-8	10000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	30-135	30-135	30	NA	50
4,4'-DDE	72-55-9	7000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	70-125	70-125	30	NA	50
4,4'-DDT	50-29-3	7000	3.3	Not Available in Method	Not Available in Method	1.00	3.3	45-140	45-140	30	NA	50
Endrin	72-20-8	180000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	60-135	60-135	30	NA	50
Endosulfan sulfate[1]	1031-07-8	3700000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	60-135	60-135	30	NA	50
Endrin aldehyde[1]	7421-93-4	180000	3.3	Not Available in Method	Not Available in Method	1.30	3.3	35-145	35-145	30	NA	50
Endrin ketone[1]	53494-70-5	180000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	65-135	65-135	30	NA	50
Endosulfan-I[1]	959-98-8	3700000	1.7	Not Available in Method	Not Available in Method	0.67	1.7	15-135	15-135	30	NA	50
Endosulfan-II[1]	33213-65-9	3700000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	35-140	35-140	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Heptachlor	76-44-8	380	1.7	Not Available in Method	Not Available in Method	0.67	1.7	50-140	50-140	30	NA	50
Heptachlor epoxide	1024-57-3	190	1.7	Not Available in Method	Not Available in Method	0.67	1.7	65-130	65-130	30	NA	50
Methoxychlor	72-43-5	3100000	3.3	Not Available in Method	Not Available in Method	1.30	3.3	65-130	65-130	30	NA	50
Toxaphene	8001-35-2	1600	170	Not Available in Method	Not Available in Method	83	170	50-150	50-150	30	NA	50
Tetrachloro-m-xylene	877-09-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	70-125	NA
Decachlorobiphenyl	2051-24-3	NA	NA	NA	NA	NA	NA	NA	NA	NA	55-130	NA

NA = Not Applicable.

[1]Project action limits are based upon USEPA Region IX Preliminary Remediation Goals (PRG) – Industrial Scenario (October, 2004) on a dry weight basis. Compound gamma-BHC (Lindane) PRG value was used as surrogate PRG for delta-BHC. Compound endosulfan PRG value was used as surrogate PRG for endosulfan I, endosulfan II, and endosulfan sulfate. Compound endrin PRG value was used as surrogate PRG for endrin aldehyde and endrin keytone. The project quantitation limit goals are based upon a wet weight basis. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.* The listed MDLs and QLs are based upon a wet weight basis.

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #12.1 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid, Aqueous
Analytical Group	Total Petroleum Hydrocarbons GRO
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sampling for VOCs in soil – Sealed-Cap (EnCore®) Sampler SOP TS-FS-104 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8015/ LAB SOP#GC010	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous	Field Blank/ Equipment Blank / Trip Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	<1/2 Lab Reporting Limit Project QLs for all target compounds are specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sampling for VOCs in soil – Sealed-Cap (EnCore®) Sampler SOP TS-FS-104 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8015/ LAB SOP#GC010	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A
		Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A
		Completeness	QC acceptance criteria: ≥95% for TPHGRO Solids ≥95% for TPHGRO Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.2 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid, Aqueous
Analytical Group	VOC
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sampling for VOCs in soil – Sealed-Cap (EnCore®) Sampler SOP TS-FS-104 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8260B/ LAB SOP#MS005	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. For the common laboratory contaminants methylene chloride, acetone, and 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous	Field Blank/ Equipment Blank / Trip Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. For the common laboratory contaminants methylene chloride, acetone, and 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sampling for VOCs in soil – Sealed-Cap (EnCore®) Sampler SOP TS-FS-104 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8260B/ LAB SOP#MS005	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A
		Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A
		Accuracy (Instrument sensitivity control)	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard	Internal Standards	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sampling for VOCs in soil – Sealed-Cap (EnCore®) Sampler SOP TS-FS-104 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8260B/ LAB SOP#MS005	Completeness	QC acceptance criteria: ≥95% for VOCs Solids ≥95% for VOCs Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.3 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid, Aqueous
Analytical Group	BNA SVOC
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8270C/ LAB SOP#MS006	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. For the common laboratory contaminants phthalate esters, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous	Field Blank/ Equipment Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. For the common laboratory contaminants phthalate esters, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous	Method Blank	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8270C/ LAB SOP#MS006	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A
		Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A
		Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8270C/ LAB SOP#MS006	Accuracy (Instrument sensitivity control)	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard	Internal Standards	A
		Completeness	QC acceptance criteria: ≥95% for SVOCs Solids ≥95% for SVOCs Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.4 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid, Aqueous
Analytical Group	Explosives (Nitroaromatics and Nitramines)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8330/ LAB SOP#GC016	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous	Field Blank/ Equipment Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8330/ LAB SOP#GC016	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A
		Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A
		Completeness	QC acceptance criteria: ≥95% for explosives Solids ≥95% for explosives Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.5 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid, Aqueous
Analytical Group	Chlorinated Pesticides
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8081/ LAB SOP#GC015	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous	Field Blank/ Equipment Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8081/ LAB SOP#GC015	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A
		Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A
		Completeness	QC acceptance criteria: ≥95% for pesticides Solids ≥95% for pesticides Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.6 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid, Aqueous
Analytical Group	PCBs
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8082/ LAB SOP#GC014	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous	Field Blank/ Equipment Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8082/ LAB SOP#GC014	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A
		Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A
		Completeness	QC acceptance criteria: ≥95% for PCBs Solids ≥95% for PCBs Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.7 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid, Aqueous
Analytical Group	Total Petroleum Hydrocarbons DRO
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8015/ LAB SOP#GC011	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.7 for TPH DRO Solids Worksheet 15.15 for TPH DRO Aqueous	Field Blank/ Equipment Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7 for TPH DRO Solids Worksheet 15.15 for TPH DRO Aqueous	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.7 for TPH DRO Solids Worksheet 15.15 for TPH DRO Aqueous	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7 for TPH DRO Solids Worksheet 15.15 for TPH DRO Aqueous %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 8015/ LAB SOP#GC011	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7 for TPH DRO Solids Worksheet 15.15 for TPH DRO Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A
		Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7 for TPH DRO Solids Worksheet 15.15 for TPH DRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7 for TPH DRO Solids Worksheet 15.15 for TPH DRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A
		Completeness	QC acceptance criteria: ≥95% for TPH DRO Solids ≥95% for TPH DRO Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.8 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid, Aqueous
Analytical Group	ICP Metals
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 6010/ LAB SOP#MET100	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for ICP metals Solids Worksheet 15.16 for ICP metals Aqueous	Field Blank/ Equipment Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for ICP metals Solids Worksheet 15.16 for ICP metals Aqueous	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for ICP metals Solids Worksheet 15.16 for ICP metals Aqueous	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for ICP metals Solids Worksheet 15.16 for ICP metals Aqueous %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 6010/ LAB SOP#MET100	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for ICP metals Solids Worksheet 15.16 for ICP metals Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A
		Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for ICP metals Solids Worksheet 15.16 for ICP metals Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Accuracy	ICS-A: Absolute value of concentration for all nonspiked analytes <2x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Interference check solutions (ICS)	A
		Precision (field samples)	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >50x MDL for ICP.	Serial Dilution Test	S + A
		Completeness	QC acceptance criteria: ≥95% for ICP metals Solids ≥95% for ICP metals Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.9 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid, Aqueous
Analytical Group	Mercury
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 7470, 7471/ LAB SOP# MET105, MET106	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for mercury Solids Worksheet 15.16 for mercury Aqueous	Field Blank/ Equipment Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for mercury Solids Worksheet 15.16 for mercury Aqueous	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for mercury Solids Worksheet 15.16 for mercury Aqueous	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for mercury Solids Worksheet 15.16 for mercury Aqueous %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101 Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113	SW-846 7470, 7471/ LAB SOP# MET105, MET106	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for mercury Solids Worksheet 15.16 for mercury Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A
		Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for mercury Solids Worksheet 15.16 for mercury Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Precision (field samples)	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >25x MDL for CVAA.	Serial Dilution Test	S + A
		Completeness	QC acceptance criteria: ≥95% for mercury Solids ≥95% for mercury Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.10 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid
Analytical Group	TCLP Volatiles
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	SW-846 1311/8260B LAB SOP#MS005	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. For the common laboratory contaminants 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.17 for TCLP VOCs Solids	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.17 for TCLP VOCs Solids %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.17 for TCLP VOCs Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A
		Accuracy (Instrument sensitivity control)	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard	Internal Standards	A
		Completeness	QC acceptance criteria: ≥95% for TCLP VOCs Solids ≥95% for TCLP VOCs Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.11 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid
Analytical Group	TCLP Semivolatiles
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	SW-846 1311/8270 LAB SOP#MS006	Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.18 for TCLP SVOCs Solids	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.18 for TCLP SVOCs Solids %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.18 for TCLP SVOCs Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A
		Accuracy (Instrument sensitivity control)	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard	Internal Standards	A
		Completeness	QC acceptance criteria: ≥95% for TCLP SVOCs Solids ≥95% for TCLP SVOCs Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.12 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid
Analytical Group	TCLP Pesticides
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	SW-846 1311/8081 LAB SOP#GC015	Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.19 for TCLP pesticides Solids	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.19 for TCLP pesticides Solids %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.19 for TCLP pesticides Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A
		Completeness	QC acceptance criteria: ≥95% for TCLP pesticides Solids ≥95% for TCLP pesticides Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.13 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid
Analytical Group	TCLP Herbicides
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	SW-846 1311/8151 LAB SOP# TGC 011	Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.20 for TCLP herbicides Solids	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.20 for TCLP herbicides Solids %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A
		Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.20 for TCLP herbicides Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Surrogate Spike	A
		Completeness	QC acceptance criteria: ≥95% for TCLP herbicide Solids ≥95% for TCLP herbicide Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.14 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid
Analytical Group	TCLP ICP Metals
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	SW-846 1311/6010B LAB SOP# Met 100, Met 103	Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.21 for TCLP ICP metals Solids	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.21 for TCLP ICP metals Solids %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A
		Accuracy	ICS-A: Absolute value of concentration for all nonspiked analytes <2x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Interference check solutions (ICS)	A
		Precision (field samples)	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >50x MDL for ICP.	Serial Dilution Test	S + A
		Completeness	QC acceptance criteria: ≥95% for TCLP ICP metals Solids ≥95% for TCLP ICP metals Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.15 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid
Analytical Group	TCLP Mercury
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	SW-846 1311/7470, SOP#MET106	Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.21 for TCLP mercury Solids	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.21 for TCLP mercury Solids %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A
		Accuracy	ICS-A: Absolute value of concentration for all nonspiked analytes <2x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Interference check solutions (ICS)	A
		Precision (field samples)	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >25x MDL for CVAA.	Serial Dilution Test	S + A
		Completeness	QC acceptance criteria: ≥95% for TCLP mercury Solids ≥95% for TCLP mercury Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.16 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid
Analytical Group	Total Cyanide
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	SW-846 9010/9014 LAB SOP#GN113, 115	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for total cyanide Solids	Field Blank/ Equipment Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for total cyanide Solids	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for total cyanide Solids	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for total cyanide Solids %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A
		Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for total cyanide Solids Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	SW-846 9010/9014 LAB SOP#GN113, 115	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for total cyanide Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Completeness	QC acceptance criteria: ≥95% for total cyanide Solids ≥95% for total cyanide Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.17 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid
Analytical Group	Total Sulfide
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	EPA 376.1/ LAB SOP#GN140	Field Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for total sulfide Solids	Field Blank/ Equipment Blank	S + A
		Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for total sulfide Solids	Field Duplicate	S + A
		Laboratory Representativeness (Absence of interference/ contamination)	The blank results are evaluated for the analytes of concern to ascertain the efficiency of decontamination and assess the potential for cross-contamination. All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for total sulfide Solids	Method Blank	A
		Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for total sulfide Solids %Recovery = (Calculated Value/True Value) *100%	Laboratory Control Sample	A
		Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for total sulfide Solids Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value - Sample Value/True Value) *100%	Matrix Spike	A

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	EPA 376.1/ LAB SOP#GN140	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for total sulfide Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD (%) = [(XA-XB)/ XM] * 100 Where: XA and XB are the concentration in the MS and MSD, and XM is the average value of the concentrations in the MS and MSD, (XA + XB)/2	Matrix Spike Duplicate or Sample Duplicate	A
		Completeness	QC acceptance criteria: ≥95% for sulfide Solids ≥95% for sulfide Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.18 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid
Analytical Group	Flash Point (Ignitability)
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	SW-846 1010/ LAB SOP#GN121	Laboratory Accuracy	p-Xylene, second source flash point of p-Xylene is 77 ± 4.3°F	Second Source Standard	A
		Completeness	QC acceptance criteria: ≥95% for ignitability Solids ≥95% for ignitability Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12.19 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Solid
Analytical Group	Corrosivity as pH
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Sample Homogenization, 6/05/03, SOP T FS-010 Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101	SW-846 9045/ LAB SOP#GN179	Laboratory Accuracy	Percent recoveries for continuing calibration must be within ±0.05 units of standard true value for pH.	Daily continuing calibration verification	A
		Laboratory Precision	% QC acceptance criteria for all target compounds as: %RPD≤10% RPD = (Difference between S and SD) * 100 / (Average of S and SD)	Sample Duplicate	A
		Completeness	QC acceptance criteria: ≥95% for corrosivity as pH Solids ≥95% for corrosivity as pH Aqueous % Analytical Completeness = 100 * (Number of Useable Data) / (Total Number of Requested Analyses)	Analytical Sample Completeness (Usability)	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #13
Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
	CH2MHILL\ Engineering Evaluation/Cost Analysis for Areas of Concern J and R, and Solid Waste Management Units 6 and 7\Former Naval Ammunitions Support Detachment, Vieques Island, Puerto Rico\June 2005	CH2MHILL	No use planned; however, it will be used for risk assessment if necessary.	None

QAPP Worksheet #14 (UFP-QAPP Manual Section 2.8.1) -- Summary of Project Tasks

Provide a brief overview of the listed project activities. The following table must be completed for each project area.

Confirmation Sampling

Sampling Tasks:

1. Confirmatory soil samples will be collected post excavation. The protocol for the confirmatory sampling shall be established after the waste characterization sampling results have been analyzed in accordance with Worksheet 17.
2. Soil confirmation samples will be collected using a stainless steel trowel, a split-spoon sampler, hand auger, or Encore® Sampler. See Worksheet 18 for field SOP and sampling techniques.
3. All soil/debris sampling equipment that may come into contact with samples or sampling surfaces will be constructed of stainless steel, borosilicate glass, or Teflon™.
4. All equipment used for collection, transfer, and homogenization will be properly decontaminated before collecting samples and between sampling locations.
5. After the sample is collected, the soil/samples will be homogenized as thoroughly as possible in a stainless-steel bowl, if required.
6. Samples collected for chemical analysis will be placed in the appropriate sample containers, labeled with proper identification, and packed in a cooler with ice pending shipment to the laboratory. See Worksheet 19 for sample containers requirements.
7. To maintain integrity, samples collected in the field must be placed in a dedicated sample ice chest, on ice, and chilled to 4ºC +/- 2ºC from the time of collection until receipt by the laboratory for analysis
8. All waste soil samples will be visually classified and documented on a sample collection log. The Site Supervisor will choose the method for sampling, such as split-spoon sampler or auger.
9. The following procedure is applicable to discrete samples, (TCL SVOCs, pesticides, PCBs, explosives, and TAL metals)

Don a fresh pair of sample gloves and place the sample into a stainless steel bowl. The sample is then thoroughly homogenized using a stainless steel spoon. If the sample aliquot is to large to reasonably homogenize in the stainless steel bowl, the sample should be carefully divided into quarters and then each of these quarters further divided into quarters. One of each of the second set of quarters is then recombined into the mixing bowl and then homogenized. The sample is then placed into the appropriate containers as described in Worksheet 19, taking care to minimize headspace as much as possible, and then labeled and stored in a cooler at 4ºC +/- 2ºC.

11. The procedure for collecting samples for TCL VOCs, TCLP VOCs, and GROs is detailed in Worksheets 17 and 21.
10. All sample documentation and chain of custody procedures outlined in Worksheet 27 should then be followed. After proper documentation has been performed, sample packaging and shipping as outlined in Worksheet 27 of this document should be completed.

Analysis Tasks:

1. Accutest Laboratories will process, prepare, and analyze soil and sediment samples. The confirmation samples will be used to evaluate the residual risk via the HHRA and the ERA protocols defined in the Master QAPP (CH2M Hill, February 2007) See Worksheet 19 for analytical method requirements.

Quality Control Tasks:

1. Implement SOPs as defined in Worksheet 18 for field sampling procedures, follow guidelines as described in Worksheet 26 and 27 for sample custody procedures, packaging and transporting of samples.
2. Laboratory to follow preparation and analysis methods as described in Worksheets 15 and 19. Laboratory quality control samples and procedures as defined in Worksheet 28.
3. Field duplicate samples will be collected at a rate of 1 per 10 field samples. Equipment and field blank samples will be collected one per day of field sampling.

Secondary Data:

1. See Worksheet 13.

Other Data:

Data Management Tasks:

1. Analytical data will be placed in an excel spreadsheet with the site remediation goals, analytical reports will be received in PDF format.

Documentation and Records:

1. All samples collected will have sample locations document in field log books. Chains-of-Custody records, air bills and laboratory sample logs will be retained for each sample and will become a part of the analytical PDF data report.
2. Analytical laboratory reports will be stored on the Shaw server.
3. Copy of the finalized QAPP will be retained in the Shaw and Accutest central file area and at the site trailer for review.

Assessment / Audit Tasks:

1. Sampling SOPs and Safety will be reviewed prior to start up of sampling.

Data Review Tasks:

1. The Shaw Program Chemist will verify that data has been received for all samples that have been sent to the laboratory. An evaluation of this data will be performed to determine whether the laboratory met the QC requirements for the analytical as stated in the analytical methods and laboratory SOPs.
2. The final remediation goal will be initially based upon unrestricted land use protocols, however the Navy may choose to utilize a more realistic and restrictive land use scenario for making the ultimate determination of risks or hazards for excavation purposes.
3. Refer to Worksheets 35 and 26 for data validation requirements.

Waste Characterization Samples

Sampling Tasks:

1. Prior to excavation and removal of the soil/sediment one 3-point composite sample (representing near surface, middle, and near bottom vertical extents of the debris) per 2000-square feet will be collected for disposal characterization analysis of Target Compound List (TCL) semi-volitile organic compounds (SVOCs), pesticides, and polychlorinated biphenyls (PCBs), explosives, and Target Analyte List (TAL) metals. The 3-points composite samples will also be analyzed for full toxicity characteristic leaching procedure (TCLP), reactivity, corrosivity, ignitability (RCI), and total petroleum hydrocarbons (TPH) In the event there is visible contamination within the site, the sample will be preferentially relocated to represent the worst condition. A photo-ionization detector (PID), or similar, will be used to screen the vertical debris interval and each component of the composite sample will be collected from the location of the highest PID reading (or visual staining in the absence of an elevated PID reading) for TCL volatile organic compounds (VOCs), TCLP VOCs, and GRO analysis. In the absence of a PID reading above background, and in the absence of any visual soil staining, the soil sample VOC analysis will be collected from the middle of the debris interval.
2. Soil/disposal samples will be collected using a stainless steel trowel, a split-spoon sampler, hand auger, or Encore® Sampler. See Worksheet 19 for field SOP and sampling techniques.
3. All soil/debris sampling equipment that may come into contact with samples or sampling surfaces will be constructed of stainless steel, borosilicate glass, or Teflon™.
4. All equipment used for collection, transfer, and homogenization will be properly decontaminated before collecting samples and between sampling locations.
5. After the sample is collected, the soil/samples to be analyzed for TCL SVOCs, pesticides, PCBs, explosives, and TAL metals will be homogenized as thoroughly as possible in a stainless-steel bowl. Samples collected for the analysis of TCL VOCs, TCLP VOCs, and GRO shall be sampled according to Worksheet 19 SOP.
6. Samples collected for chemical analysis will be placed in the appropriate sample containers, labeled with proper identification, and packed in a cooler with ice pending shipment to the laboratory. See Worksheet 19 for sample containers requirements.
7. To maintain integrity, samples collected in the field must be placed in a dedicated sample ice chest, on ice, and chilled to 4ºC +/- 2ºC from the time of collection until receipt by the laboratory for analysis
8. All waste soil samples will be visually classified and documented on a sample collection log. The Site Supervisor will choose the method for sampling, such as split-spoon sampler or auger.
9. The following procedures are applicable for composite/discrete samples:

Don a fresh pair of sample gloves. For VOC and GRO analysis in soil, a grab sample will be collected directly from the sampling point using an Encore™ sampler or its equivalent and not homogenized prior to sampling. For the remaining analysis, place the sample into a stainless steel bowl. The sample is then thoroughly homogenized using a stainless steel spoon. If the sample aliquot is too large to reasonably homogenize in the stainless steel bowl, the sample should be carefully divided into quarters and then each of these quarters further divided into quarters. One of each of the second set of quarters is then recombined into the mixing bowl and then homogenized. The sample is then placed into the appropriate containers as described in Worksheet 19, taking care to minimize headspace as much as possible, and then labeled and stored in a cooler at 4°C.

10. All sample documentation and chain of custody procedures outlined in Worksheet 27 should then be followed. After proper documentation has been performed, sample packaging and shipping as outlined in Worksheet 27 of this document should be completed.
11. Data validation for waste characterization sampling shall not be required, as the material is being proposed as fill. Please refer to Worksheet 35 for clarification.

Analysis Tasks:

1. Accutest Laboratories will process, prepare, and analyze soil and sediment samples for Target Compound List (TCL), semi-volatile organic compounds (SVOCs), volatile organic compounds (VOCs), pesticides, polychlorinated biphenyls (PCBs), total petroleum hydrocarbons (TPH), explosives, and Target Analyte List (TAL) metals. The samples will also be analyzed for full toxic leaching characteristic procedure (TCLP) parameters, and reactivity, corrosivity, and ignitability (RCI). Based upon these results, the appropriate disposal method shall be chosen (daily cover, non-hazardous landfill, hazardous landfill, or left in place). See Worksheet 10.

Quality Control Tasks:

1. Implement SOPs as defined in Worksheet 18 for field sampling procedures, follow guidelines as described in Worksheet 26 and 27 for sample custody procedures, packaging and transporting of samples.
2. Laboratory to follow preparation and analysis methods as described in Worksheets 15 and 19. Laboratory quality control samples and procedures as defined in Worksheet 28.
3. No field duplicate samples or equipment blank samples are required for waste disposal samples.

Secondary Data:

1. See Worksheet 13.

Other Data:

Data Management Tasks:

1. Analytical data will be placed in an excel spreadsheet with the TCLP Maximum Contaminant Concentrations, analytical reports will be received in PDF format.

Documentation and Records:

1. All samples collected will have sample locations document in field log books. Chains-of-Custody records, air bills and laboratory sample logs will be retained for each sample and will become a part of the analytical PDF data report.
2. Analytical laboratory reports will be stored on the Shaw server.
3. Copy of the finalized QAPP will be retained in the Shaw and Accutest central file area and at a central location on the island, controlled by the Shaw Site Manager.

Assessment / Audit Tasks:

1. Sampling SOPs and Safety will be reviewed prior to start up of sampling.

Data Review Tasks:

1. The Shaw Program Chemist will verify that data has been received for all samples that have been sent to the laboratory. An evaluation of this data will be performed to determine whether the laboratory met the QC requirements for the analytical as stated in the analytical methods and laboratory SOPs.
2. For disposal samples, data validation will not be required since the use of the analytical data is to determine characterization of soil for disposal.

QAPP Worksheet #15.1 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil/Sediment
Analytical Group: Organic – TPH GRO **SW**-846 5035A/8015M
Concentration Level: Low

				Analytical Method[2]		**Achievable Laboratory Limits[3]**		**Precision and Accuracy Method Performance Criteria[4]**				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/kg)**	**MDLs (mg/kg)**	**Method QLs (mg/kg)**	**MDLs (mg/kg)**	**QLs (mg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
TPH-GRO (C6-C10)	NA	100	0.1	Not Available in Method	Not Available in Method	0.05	0.1	66-122	37-142	17	NA	50
4-Bromofluorobenzene	460-00-4	NA	NA	NA	NA	NA	NA	NA	NA	NA	62-135	NA
aaa-Trifluorotoluene	98-08-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	65-118	NA

NA = Not Applicable.

[1]Project action limits for soil/sediment TPH GRO are provided only to evaluate the laboratory limits and are taken from the *Vieques Puerto Rico Environmental Restoration Program Master QAPP* (CHM2Hill, May 2007). The project action limits are based upon a dry weight basis and the project quantitation limit goals are based upon a wet weight basis. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.* The listed MDLs and QLs are based upon a wet weight basis.

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.2 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil/Sediment
Analytical Group: TCL Volatiles Organic - SW-846 5035A/8260B
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Acetone	67-64-1	54000000	50	Not Available in Method	Not Available in Method	25	50	20-160	20-160	30	NA	50
Benzene	71-43-2	1400	5	Not Available in Method	Not Available in Method	2	5	75-125	75-125	30	NA	50
Bromodichloromethane	75-27-4	1800	5	Not Available in Method	Not Available in Method	2	5	70-130	70-130	30	NA	50
Bromoform	75-25-2	220000	5	Not Available in Method	Not Available in Method	2	5	55-135	55-135	30	NA	50
Chlorobenzene	108-90-7	530000	5	Not Available in Method	Not Available in Method	2	5	75-125	75-125	30	NA	50
Chloroethane	75-00-3	6500	5	Not Available in Method	Not Available in Method	3	5	40-155	40-155	30	NA	50
Chloroform	67-66-3	470	5	Not Available in Method	Not Available in Method	2	5	70-125	70-125	30	NA	50
Carbon disulfide	75-15-0	720000	5	Not Available in Method	Not Available in Method	2	5	30-160	30-160	30	NA	50
Carbon tetrachloride	56-23-5	550	5	Not Available in Method	Not Available in Method	2	5	65-135	65-135	30	NA	50
1,1-Dichloroethane	75-34-3	1700000	5	Not Available in Method	Not Available in Method	2	5	75-125	75-125	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
1,1-Dichloroethylene	75-35-4	410000	5	Not Available in Method	Not Available in Method	2	5	65-135	65-135	30	NA	50
1,2-Dichloroethane	107-06-2	600	5	Not Available in Method	Not Available in Method	2	5	70-135	70-135	30	NA	50
1,2-Dichloropropane	78-87-5	740	5	Not Available in Method	Not Available in Method	2	5	70-120	70-120	30	NA	50
Dibromochloromethane	124-48-1	2600	5	Not Available in Method	Not Available in Method	2	5	65-130	65-130	30	NA	50
cis-1,2-Dichloroethylene	156-59-2	150000	5	Not Available in Method	Not Available in Method	2	5	65-125	65-125	30	NA	50
cis-1,3-Dichloropropene	10061-01-5	1800	5	Not Available in Method	Not Available in Method	2	5	70-125	70-125	30	NA	50
trans-1,2-Dichloroethylene	156-60-5	230000	5	Not Available in Method	Not Available in Method	2	5	65-135	65-135	30	NA	50
trans-1,3-Dichloropropene	10061-02-6	1800	5	Not Available in Method	Not Available in Method	2	5	65-125	65-125	30	NA	50
Ethylbenzene	100-41-4	400000	5	Not Available in Method	Not Available in Method	2	5	75-125	75-125	30	NA	50
2-Hexanone[1]	591-78-6	110000000	25	Not Available in Method	Not Available in Method	10	25	45-145	45-145	30	NA	50
4-Methyl-2-pentanone	108-10-1	47000000	25	Not Available in Method	Not Available in Method	10	25	45-145	45-145	30	NA	50
Methyl bromide	74-83-9	13000	5	Not Available in Method	Not Available in Method	2	5	30-160	30-160	30	NA	50
Methyl chloride	74-87-3	160000	5	Not Available in Method	Not Available in Method	2	5	50-130	50-130	30	NA	50
Methylene chloride	75-09-2	21000	10	Not Available in Method	Not Available in Method	5	10	55-140	55-140	30	NA	50
Methyl ethyl ketone	78-93-3	110000000	25	Not Available in Method	Not Available in Method	10	25	30-160	30-160	30	NA	50
Styrene	100-42-5	1700000	5	Not Available in Method	Not Available in Method	2	5	75-125	75-125	30	NA	50
1,1,1-Trichloroethane	71-55-6	1200000	5	Not Available in Method	Not Available in Method	2	5	70-135	70-135	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
1,1,2,2-Tetrachloroethane	79-34-5	930	5	Not Available in Method	Not Available in Method	2	5	55-130	55-130	30	NA	50
1,1,2-Trichloroethane	79-00-5	1600	5	Not Available in Method	Not Available in Method	2	5	60-125	60-125	30	NA	50
Tetrachloroethylene	127-18-4	1300	5	Not Available in Method	Not Available in Method	2	5	65-140	65-140	30	NA	50
Toluene	108-88-3	520000	5	Not Available in Method	Not Available in Method	2	5	70-125	70-125	30	NA	50
Trichloroethylene	79-01-6	110	5	Not Available in Method	Not Available in Method	2	5	75-125	75-125	30	NA	50
Vinyl chloride	75-01-4	750	5	Not Available in Method	Not Available in Method	2	5	60-125	60-125	30	NA	50
Xylene (total)	1330-20-7	420000	15	Not Available in Method	Not Available in Method	5	15	80-125	80-125	30	NA	50
Dibromofluoromethane	1868-53-7	NA	NA	NA	NA	NA	NA	NA	NA	NA	78-123	NA
Toluene-D8	2037-26-5	NA	NA	NA	NA	NA	NA	NA	NA	NA	85-115	NA
4-Bromofluorobenzene	460-00-4	NA	NA	NA	NA	NA	NA	NA	NA	NA	85-120	NA
1,2-Dichloroethane-D4	17060-07-0	NA	NA	NA	NA	NA	NA	NA	NA	NA	74-125	NA

NA = Not Applicable.

[1]Project action limits are based upon USEPA Region IX Preliminary Remediation Goals (PRG) – Industrial Scenario (October, 2004) on a dry weight basis. Compound methyl ethyl ketone PRG value was used as surrogate PRG for 2-hexanone. The project quantitation limit goals are based upon a wet weight basis. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.* The listed MDLs and QLs are based upon a wet weight basis.

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.3 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil/Sediment
Analytical Group: TCL Semivolatiles Organic - SW-846 3550B/8270C
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Benzoic acid	65-85-0	100000000	830	Not Available in Method	Not Available in Method	330	830	10-110	10-110	30	NA	50
2-Chlorophenol	95-57-8	240000	170	Not Available in Method	Not Available in Method	33	170	45-105	45-105	30	NA	50
4-Chloro-3-methyl phenol[1]	59-50-7	240000	170	Not Available in Method	Not Available in Method	33	170	45-115	45-115	30	NA	50
2,4-Dichlorophenol	120-83-2	1800000	170	Not Available in Method	Not Available in Method	33	170	45-110	45-110	30	NA	50
2,4-Dimethylphenol	105-67-9	1200000	170	Not Available in Method	Not Available in Method	33	170	30-105	30-105	30	NA	50
2,4-Dinitrophenol	51-28-5	120000	830	Not Available in Method	Not Available in Method	330	830	15-130	15-130	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
4,6-Dinitro-o-cresol	534-52-1	62000	330	Not Available in Method	Not Available in Method	67	330	30-135	30-135	30	NA	50
2-Methylphenol	95-48-7	31000000	170	Not Available in Method	Not Available in Method	33	170	40-105	40-105	30	NA	50
3&4-Methylphenol	NA	3100000	170	Not Available in Method	Not Available in Method	33	170	40-105	40-105	30	NA	50
2-Nitrophenol[1]	88-75-5	240000	170	Not Available in Method	Not Available in Method	33	170	40-110	40-110	30	NA	50
4-Nitrophenol[1]	100-02-7	240000	830	Not Available in Method	Not Available in Method	830	830	15-140	15-140	30	NA	50
Pentachlorophenol	87-86-5	9000	830	Not Available in Method	Not Available in Method	330	830	25-120	25-120	30	NA	50
Phenol	108-95-2	100000000	170	Not Available in Method	Not Available in Method	33	170	40-100	40-100	30	NA	50
2,4,5-Trichlorophenol	95-95-4	62000000	170	Not Available in Method	Not Available in Method	33	170	50-110	50-110	30	NA	50
2,4,6-Trichlorophenol	88-06-2	62000	170	Not Available in Method	Not Available in Method	33	170	45-110	45-110	30	NA	50
Acenaphthene	83-32-9	29000000	170	Not Available in Method	Not Available in Method	33	170	45-110	45-110	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Acenaphthylene[1]	208-96-8	29000000	170	Not Available in Method	Not Available in Method	33	170	45-105	45-105	30	NA	50
Anthracene	120-12-7	100000000	170	Not Available in Method	Not Available in Method	33	170	55-105	55-105	30	NA	50
Benzo(a)anthracene	56-55-3	2100	170	Not Available in Method	Not Available in Method	33	170	50-110	50-110	30	NA	50
Benzo(a)pyrene	50-32-8	210	170	Not Available in Method	Not Available in Method	33	170	50-110	50-110	30	NA	50
Benzo(b)fluoranthene	205-99-2	2100	170	Not Available in Method	Not Available in Method	33	170	45-115	45-115	30	NA	50
Benzo(g,h,i)perylene[1]	191-24-2	29000000	170	Not Available in Method	Not Available in Method	67	170	40-125	40-125	30	NA	50
Benzo(k)fluoranthene	207-08-9	21000	170	Not Available in Method	Not Available in Method	33	170	45-125	45-125	30	NA	50
4-Bromophenyl phenyl ether[1]	101-55-3	7400	170	Not Available in Method	Not Available in Method	33	170	45-115	45-115	30	NA	50
Butyl benzyl phthalate	85-68-7	100000000	330	Not Available in Method	Not Available in Method	83	330	50-125	50-125	30	NA	50
Benzyl Alcohol	100-51-6	100000000	170	Not Available in Method	Not Available in Method	33	170	20-125	20-125	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
2-Chloronaphthalene	91-58-7	23000000	170	Not Available in Method	Not Available in Method	33	170	45-105	45-105	30	NA	50
4-Chloroaniline	106-47-8	2500000	330	Not Available in Method	Not Available in Method	130	330	10-95	10-95	30	NA	50
Carbazole	86-74-8	86000	170	Not Available in Method	Not Available in Method	33	170	45-115	45-115	30	NA	50
Chrysene	218-01-9	210000	170	Not Available in Method	Not Available in Method	33	170	55-110	55-110	30	NA	50
bis(2-Chloroethoxy)methane[1]	111-91-1	7400	170	Not Available in Method	Not Available in Method	33	170	45-110	45-110	30	NA	50
bis(2-Chloroethyl)ether	111-44-4	580	170	Not Available in Method	Not Available in Method	67	170	40-105	40-105	30	NA	50
bis(2-Chloroisopropyl)ether	108-60-1	7400	170	Not Available in Method	Not Available in Method	33	170	20-115	20-115	30	NA	50
4-Chlorophenyl phenyl ether[1]	7005-72-3	7400	170	Not Available in Method	Not Available in Method	33	170	45-110	45-110	30	NA	50
1,2-Dichlorobenzene	95-50-1	600000	170	Not Available in Method	Not Available in Method	33	170	45-95	45-95	30	NA	50
1,3-Dichlorobenzene	541-73-1	600000	170	Not Available in Method	Not Available in Method	33	170	40-100	40-100	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
1,4-Dichlorobenzene	106-46-7	7900	170	Not Available in Method	Not Available in Method	33	170	35-105	35-105	30	NA	50
2,4-Dinitrotoluene	121-14-2	2500	170	Not Available in Method	Not Available in Method	67	170	50-115	50-115	30	NA	50
2,6-Dinitrotoluene	606-20-2	2500	170	Not Available in Method	Not Available in Method	67	170	50-110	50-110	30	NA	50
3,3'-Dichlorobenzidine	91-94-1	750	330	Not Available in Method	Not Available in Method	170	330	10-130	10-130	30	NA	50
Dibenzo(a,h)anthracene	53-70-3	210	170	Not Available in Method	Not Available in Method	67	170	40-125	40-125	30	NA	50
Dibenzofuran	132-64-9	1600000	170	Not Available in Method	Not Available in Method	33	170	50-105	50-105	30	NA	50
Di-n-butyl phthalate	84-74-2	62000000	330	Not Available in Method	Not Available in Method	83	330	55-110	55-110	30	NA	50
Di-n-octyl phthalate	117-84-0	25000000	330	Not Available in Method	Not Available in Method	170	330	40-130	40-130	30	NA	50
Diethyl phthalate	84-66-2	100000000	330	Not Available in Method	Not Available in Method	83	330	50-115	50-115	30	NA	50
Dimethyl phthalate	131-11-3	100000000	330	Not Available in Method	Not Available in Method	83	330	50-110	50-110	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
bis(2-Ethylhexyl)phthalate	117-81-7	120000	330	Not Available in Method	Not Available in Method	170	330	45-125	45-125	30	NA	50
Fluoranthene	206-44-0	22000000	170	Not Available in Method	Not Available in Method	33	170	55-115	55-115	30	NA	50
Fluorene	86-73-7	26000000	170	Not Available in Method	Not Available in Method	33	170	50-110	50-110	30	NA	50
Hexachlorobenzene	118-74-1	1100	170	Not Available in Method	Not Available in Method	33	170	45-120	45-120	30	NA	50
Hexachlorobutadiene	87-68-3	22000	170	Not Available in Method	Not Available in Method	67	170	40-115	40-115	30	NA	50
Hexachlorocyclopentadiene	77-47-4	3700000	170	Not Available in Method	Not Available in Method	67	170	40-115	40-115	30	NA	50
Hexachloroethane	67-72-1	120000	170	Not Available in Method	Not Available in Method	67	170	35-110	35-110	30	NA	50
Indeno(1,2,3-cd)pyrene	193-39-5	2100	170	Not Available in Method	Not Available in Method	67	170	40-120	40-120	30	NA	50
Isophorone	78-59-1	510000	170	Not Available in Method	Not Available in Method	33	170	45-110	45-110	30	NA	50
2-Methylnaphthalene[1]	91-57-6	29000000	170	Not Available in Method	Not Available in Method	33	170	45-105	45-105	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
2-Nitroaniline	88-74-4	1800000	330	Not Available in Method	Not Available in Method	83	330	45-120	45-120	30	NA	50
3-Nitroaniline	99-09-2	82000	330	Not Available in Method	Not Available in Method	83	330	25-110	25-110	30	NA	50
4-Nitroaniline	100-01-6	82000	330	Not Available in Method	Not Available in Method	120	330	35-115	35-115	30	NA	50
Naphthalene	91-20-3	190000	170	Not Available in Method	Not Available in Method	33	170	40-105	40-105	30	NA	50
Nitrobenzene	98-95-3	100000	170	Not Available in Method	Not Available in Method	33	170	40-115	40-115	30	NA	50
N-Nitroso-di-n-propylamine	621-64-7	250	170	Not Available in Method	Not Available in Method	67	170	40-115	40-115	30	NA	50
N-Nitrosodiphenylamine	86-30-6	350000	170	Not Available in Method	Not Available in Method	33	170	50-115	50-115	30	NA	50
Phenanthrene[1]	85-01-8	29000000	170	Not Available in Method	Not Available in Method	33	170	50-110	50-110	30	NA	50
Pyrene	129-00-0	29000000	170	Not Available in Method	Not Available in Method	67	170	45-125	45-125	30	NA	50
1,2,4-Trichlorobenzene	120-82-1	220000	170	Not Available in Method	Not Available in Method	33	170	45-110	45-110	30	NA	50
2-Fluorophenol	367-12-4	NA	NA	NA	NA	NA	NA	NA	NA	NA	35-105	NA

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Phenol-d5	4165-62-2	NA	NA	NA	NA	NA	NA	NA	NA	NA	40-100	NA
2,4,6-Tribromophenol	118-79-6	NA	NA	NA	NA	NA	NA	NA	NA	NA	35-125	NA
Nitrobenzene-d5	4165-60-0	NA	NA	NA	NA	NA	NA	NA	NA	NA	35-100	NA
2-Fluorobiphenyl	321-60-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	45-105	NA
Terphenyl-d14	1718-51-0	NA	NA	NA	NA	NA	NA	NA	NA	NA	30-125	NA

NA = Not Applicable.

[1]Project action limits are based upon USEPA Region IX Preliminary Remediation Goals (PRG) – Industrial Scenario (October, 2004) on a dry weight basis. Compound 2-chlorophenol PRG value was used as surrogate PRG for 4-chloro-3-methyl phenol, 2-nitrophenol, and 4-nitrophenol. Compound acenaphthene PRG value was used as surrogate PRG for acenaphthylene. Compound bis(2-chloroisopropyl)ether PRG value was used as surrogate PRG for bis(2-Chloroethoxy)methane, 4-chlorophenyl phenyl ether, and 4-bromophenyl phenyl ether. Compound pyrene PRG value was used as surrogate PRG for phenanthrene, benzo(g,h,i)perylene, and 2-methylnaphthalene. The project quantitation limit goals are based upon a wet weight basis. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.* The listed MDLs and QLs are based upon a wet weight basis.

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.4 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil/Sediment
Analytical Group: Explosives Organic - SW-846 8330
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
HMX	2691-41-0	31000000	250	Not Available in Method	Not Available in Method	100	250	75-125	75-125	30	NA	50
RDX	121-82-4	16000	250	Not Available in Method	Not Available in Method	100	250	70-130	70-130	30	NA	50
1,3-Dinitrobenzene	99-65-0	62000	250	Not Available in Method	Not Available in Method	100	250	80-125	80-125	30	NA	50
2,6-Dinitrotoluene	606-20-2	2500	250	Not Available in Method	Not Available in Method	130	250	80-125	80-125	30	NA	50
2,4-Dinitrotoluene	121-14-2	2500	250	Not Available in Method	Not Available in Method	100	250	80-125	80-125	30	NA	50
2-amino-4,6-Dinitrotoluene	35572-78-2	120000	250	Not Available in Method	Not Available in Method	100	250	80-125	80-125	30	NA	50
4-amino-2,6-Dinitrotoluene	19406-51-0	120000	250	Not Available in Method	Not Available in Method	100	250	80-125	80-125	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Nitrobenzene	98-95-3	100000	250	Not Available in Method	Not Available in Method	100	250	75-125	75-125	30	NA	50
o-Nitrotoluene	88-72-2	1000000	250	Not Available in Method	Not Available in Method	100	250	80-125	80-125	30	NA	50
m-Nitrotoluene	99-08-1	2200	250	Not Available in Method	Not Available in Method	100	250	75-125	75-125	30	NA	50
p-Nitrotoluene	99-99-0	30000	250	Not Available in Method	Not Available in Method	100	250	75-125	75-125	30	NA	50
Tetryl	479-45-8	6200000	250	Not Available in Method	Not Available in Method	100	250	10-150	10-150	30	NA	50
1,3,5-Trinitrobenzene	99-35-4	18000000	250	Not Available in Method	Not Available in Method	100	250	75-125	75-125	30	NA	50
2,4,6-Trinitrotoluene	118-96-7	57000	250	Not Available in Method	Not Available in Method	100	250	55-140	55-140	30	NA	50
3,4-Dinitrotoluene	610-39-9	NA	NA	NA	NA	NA	NA	NA	NA	NA	71-130	NA

NA = Not Applicable.

[1]Project action limits are based upon USEPA Region IX Preliminary Remediation Goals (PRG) – Industrial Scenario (October, 2004) on a dry weight basis. The project quantitation limit goals are based upon a wet weight basis. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.* The listed MDLs and QLs are based upon a wet weight basis.

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.5 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil/Sediment
Analytical Group: TCL Pesticides Organic - SW-846 3550B/8081A
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Aldrin	309-00-2	100	1.7	Not Available in Method	Not Available in Method	0.67	1.7	45-140	45-140	30	NA	50
alpha-BHC	319-84-6	360	1.7	Not Available in Method	Not Available in Method	0.67	1.7	60-125	60-125	30	NA	50
beta-BHC	319-85-7	1300	1.7	Not Available in Method	Not Available in Method	0.67	1.7	60-125	60-125	30	NA	50
delta-BHC[1]	319-86-8	1700	1.7	Not Available in Method	Not Available in Method	0.67	1.7	55-130	55-130	30	NA	50
gamma-BHC (Lindane)	58-89-9	1700	1.7	Not Available in Method	Not Available in Method	0.67	1.7	60-125	60-125	30	NA	50
alpha-Chlordane	5103-71-9	6500	1.7	Not Available in Method	Not Available in Method	0.67	1.7	65-120	65-120	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
gamma-Chlordane	5103-74-2	6500	1.7	Not Available in Method	Not Available in Method	0.67	1.7	65-125	65-125	30	NA	50
Dieldrin	60-57-1	110	1.7	Not Available in Method	Not Available in Method	0.67	1.7	65-125	65-125	30	NA	50
4,4'-DDD	72-54-8	10000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	30-135	30-135	30	NA	50
4,4'-DDE	72-55-9	7000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	70-125	70-125	30	NA	50
4,4'-DDT	50-29-3	7000	3.3	Not Available in Method	Not Available in Method	1.00	3.3	45-140	45-140	30	NA	50
Endrin	72-20-8	180000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	60-135	60-135	30	NA	50
Endosulfan sulfate[1]	1031-07-8	3700000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	60-135	60-135	30	NA	50
Endrin aldehyde[1]	7421-93-4	180000	3.3	Not Available in Method	Not Available in Method	1.30	3.3	35-145	35-145	30	NA	50
Endrin ketone[1]	53494-70-5	180000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	65-135	65-135	30	NA	50
Endosulfan-I[1]	959-98-8	3700000	1.7	Not Available in Method	Not Available in Method	0.67	1.7	15-135	15-135	30	NA	50
Endosulfan-II[1]	33213-65-9	3700000	3.3	Not Available in Method	Not Available in Method	0.67	3.3	35-140	35-140	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Heptachlor	76-44-8	380	1.7	Not Available in Method	Not Available in Method	0.67	1.7	50-140	50-140	30	NA	50
Heptachlor epoxide	1024-57-3	190	1.7	Not Available in Method	Not Available in Method	0.67	1.7	65-130	65-130	30	NA	50
Methoxychlor	72-43-5	3100000	3.3	Not Available in Method	Not Available in Method	1.30	3.3	65-130	65-130	30	NA	50
Toxaphene	8001-35-2	1600	170	Not Available in Method	Not Available in Method	83	170	50-150	50-150	30	NA	50
Tetrachloro-m-xylene	877-09-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	70-125	NA
Decachlorobiphenyl	2051-24-3	NA	NA	NA	NA	NA	NA	NA	NA	NA	55-130	NA

NA = Not Applicable.

[1]Project action limits are based upon USEPA Region IX Preliminary Remediation Goals (PRG) – Industrial Scenario (October, 2004) on a dry weight basis. Compound gamma-BHC (Lindane) PRG value was used as surrogate PRG for delta-BHC. Compound endosulfan PRG value was used as surrogate PRG for endosulfan I, endosulfan II, and endosulfan sulfate. Compound endrin PRG value was used as surrogate PRG for endrin aldehyde and endrin keytone. The project quantitation limit goals are based upon a wet weight basis. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.* The listed MDLs and QLs are based upon a wet weight basis.

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.6 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil/Sediment
Analytical Group: Organic – Total PCBs by SW-846 3550B/8082
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Aroclor 1016	12674-11-2	740	17	Not Available in Method	Not Available in Method	8.3	17	40-140	40-140	30	NA	50
Aroclor 1221	11104-28-2	740	17	Not Available in Method	Not Available in Method	13	17	NA	NA	NA	NA	50
Aroclor 1232	11141-16-5	740	17	Not Available in Method	Not Available in Method	13	17	NA	NA	NA	NA	50
Aroclor 1242	53469-21-9	740	17	Not Available in Method	Not Available in Method	8.3	17	NA	NA	NA	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/kg)**	**MDLs (µg/kg)**	**Method QLs (µg/kg)**	**MDLs (µg/kg)**	**QLs (µg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Aroclor 1248	12672-29-6	740	17	Not Available in Method	Not Available in Method	8.3	17	NA	NA	NA	NA	50
Aroclor 1254	11097-69-1	740	17	Not Available in Method	Not Available in Method	8.3	17	NA	NA	NA	NA	50
Aroclor 1260	11096-82-5	740	17	Not Available in Method	Not Available in Method	8.3	17	60-130	60-130	30	NA	50
Tetrachloro-m-xylene	877-09-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	52-136	NA
Decachlorobiphenyl	2051-24-3	NA	NA	NA	NA	NA	NA	NA	NA	NA	60-125	NA

NA = Not Applicable

[1]Project action limits are based upon USEPA Region IX Preliminary Remediation Goals (PRG) – Industrial Scenario (October, 2004) on a dry weight basis. The project quantitation limit goals are based upon a wet weight basis. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.* The listed MDLs and QLs are based upon a wet weight basis.

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM. NA = Not Applicable.

QAPP Worksheet #15.7 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil/Sediment
Analytical Group: Organic – TPH DRO SW-846 3550B/8015M
Concentration Level: Low

				Analytical Method[2]		**Achievable Laboratory Limits[3]**		**Precision and Accuracy Method Performance Criteria[4]**				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/kg)**	**MDLs (mg/kg)**	**Method QLs (mg/kg)**	**MDLs (mg/kg)**	**QLs (mg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
TPH-DRO (C10-C28)	NA	100	8.3	Not Available in Method	Not Available in Method	5.0	8.3	62-114	31-136	36	NA	50
o-Terphenyl	84-15-1	NA	NA	NA	NA	NA	NA	NA	NA	NA	57-115	NA

NA = Not Applicable.

[1]Project action limits for soil/sediment TPH DRO are provided only to evaluate the laboratory limits and are taken from the *Vieques Puerto Rico Environmental Restoration Program Master QAPP* (CHM2Hill, May 2007). The project action limits are based upon a dry weight basis and the project quantitation limit goals are based upon a wet weight basis. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.* The listed MDLs and QLs are based upon a wet weight basis.

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.8 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil/Sediment
Analytical Group: Inorganic TAL Metals - SW-846 3050B/6010B/7471A
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/kg)**	**MDLs (mg/kg)**	**Method QLs (mg/kg)**	**MDLs (mg/kg)**	**QLs (mg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Aluminum	7429-90-5	100000	20	Not Available in Method	Not Available in Method	2.2	20	80-120	80-120	20	NA	50
Antimony	7440-36-0	410	6	Not Available in Method	Not Available in Method	0.53	6	80-120	80-120	20	NA	50
Arsenic	7440-38-2	1.6	0.5	Not Available in Method	Not Available in Method	0.39	0.5	80-120	80-120	20	NA	50
Barium	7440-39-3	67000	20	Not Available in Method	Not Available in Method	0.08	20	80-120	80-120	20	NA	50
Beryllium	7440-41-7	1900	5	Not Available in Method	Not Available in Method	0.04	5	80-120	80-120	20	NA	50
Cadmium	7440-43-9	450	0.4	Not Available in Method	Not Available in Method	0.02	0.4	80-120	80-120	20	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/kg)**	**MDLs (mg/kg)**	**Method QLs (mg/kg)**	**MDLs (mg/kg)**	**QLs (mg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Calcium[1]	7440-70-2	NA	500	Not Available in Method	Not Available in Method	5.7	500	80-120	80-120	20	NA	50
Chromium	7440-47-3	450	1	Not Available in Method	Not Available in Method	0.09	1	80-120	80-120	20	NA	50
Cobalt	7440-48-4	1900	5	Not Available in Method	Not Available in Method	0.06	5	80-120	80-120	20	NA	50
Copper	7440-50-8	41000	2.5	Not Available in Method	Not Available in Method	0.09	2.5	80-120	80-120	20	NA	50
Iron	7439-89-6	100000	10	Not Available in Method	Not Available in Method	1.2	10	80-120	80-120	20	NA	50
Lead	7439-92-1	800	10	Not Available in Method	Not Available in Method	0.2	10	80-120	80-120	20	NA	50
Magnesium[1]	7439-95-4	NA	500	Not Available in Method	Not Available in Method	0.74	500	80-120	80-120	20	NA	50
Manganese	7439-96-5	19000	1.5	Not Available in Method	Not Available in Method	0.06	1.5	80-120	80-120	20	NA	50
Mercury	7439-97-6	310	0.083	Not Available in Method	Not Available in Method	0.0067	0.083	80-120	80-120	20	NA	50
Nickel	7440-02-0	20000	4	Not Available in Method	Not Available in Method	0.1	4	80-120	80-120	20	NA	50
Potassium[1]	7440-09-7	NA	500	Not Available in Method	Not Available in Method	10	500	80-120	80-120	20	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/kg)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/kg)**	**MDLs (mg/kg)**	**Method QLs (mg/kg)**	**MDLs (mg/kg)**	**QLs (mg/kg)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Selenium	7782-49-2	5100	10	Not Available in Method	Not Available in Method	0.2	10	80-120	80-120	20	NA	50
Silver	7440-22-4	5100	1	Not Available in Method	Not Available in Method	0.09	1	75-120	75-120	20	NA	50
Sodium[1]	7440-23-5	NA	500	Not Available in Method	Not Available in Method	83	500	80-120	80-120	20	NA	50
Thallium	7440-28-0	67	1	Not Available in Method	Not Available in Method	0.23	1	80-120	80-120	20	NA	50
Vanadium	7440-62-2	1000	5	Not Available in Method	Not Available in Method	0.06	5	80-120	80-120	20	NA	50
Zinc	7440-66-6	100000	2	Not Available in Method	Not Available in Method	0.13	2	80-120	80-120	20	NA	50

NA = Not Applicable.

[1]Project action limits are based upon USEPA Region IX Preliminary Remediation Goals (PRG) – Industrial Scenario (October, 2004) on a dry weight basis. The metals calcium, magnesium, potassium, and sodium do have PRGs; therefore, noted as not applicable. The project quantitation limit goals are based upon a wet weight basis.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.* The listed MDLs and QLs are based upon a wet weight basis.

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.9 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Aqueous
Analytical Group: Organic – TPH GRO SW-846 8015M
Concentration Level: Low

				Analytical Method[2]		**Achievable Laboratory Limits[3]**		**Precision and Accuracy Method Performance Criteria[4]**				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
TPH-GRO (C6-C10)	NA	NA	0.1	Not Available in Method	Not Available in Method	0.05	0.1	63-126	56-171	31	NA	50
4-Bromofluorobenzene	460-00-4	NA	NA	NA	NA	NA	NA	NA	NA	NA	65-127	NA
aaa-Trifluorotoluene	98-08-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	65-124	NA

NA = Not Applicable.

[1]The surface water samples are to be collected to characterize the water at the surface and are not to be compared to any specific criteria at this time; therefore, the action limits are not applicable. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.10 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Aqueous
Analytical Group: TCL Volatiles Organic - SW-846 5030B/8260B
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Acetone	67-64-1	NA	25	Not Available in Method	Not Available in Method	5.0	25	40-140	40-140	30	NA	50
Benzene	71-43-2	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	80-120	80-120	30	NA	50
Bromodichloromethane	75-27-4	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	75-120	75-120	30	NA	50
Bromoform	75-25-2	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	70-130	70-130	30	NA	50
Chlorobenzene	108-90-7	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	80-120	80-120	30	NA	50
Chloroethane	75-00-3	NA	2.0	Not Available in Method	Not Available in Method	1.0	2.0	60-135	60-135	30	NA	50
Chloroform	67-66-3	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	65-135	65-135	30	NA	50
Carbon disulfide	75-15-0	NA	2.0	Not Available in Method	Not Available in Method	1.0	2.0	35-160	35-160	30	NA	50
Carbon tetrachloride	56-23-5	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	65-140	65-140	30	NA	50
1,1-Dichloroethane	75-34-3	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	70-135	70-135	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
1,1-Dichloroethylene	75-35-4	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	70-130	70-130	30	NA	50
1,2-Dichloroethane	107-06-2	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	80-120	80-120	30	NA	50
1,2-Dichloropropane	78-87-5	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	75-125	75-125	30	NA	50
Dibromochloromethane	124-48-1	NA	1.0	Not Available in Method	Not Available in Method	0.4	1.0	60-135	60-135	30	NA	50
cis-1,2-Dichloroethylene	156-59-2	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	70-125	70-125	30	NA	50
cis-1,3-Dichloropropene	10061-01-5	NA	1.0	Not Available in Method	Not Available in Method	0.3	1.0	70-130	70-130	30	NA	50
trans-1,2-Dichloroethylene	156-60-5	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	60-140	60-140	30	NA	50
trans-1,3-Dichloropropene	10061-02-6	NA	1.0	Not Available in Method	Not Available in Method	0.3	1.0	55-140	55-140	30	NA	50
Ethylbenzene	100-41-4	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	75-125	75-125	30	NA	50
2-Hexanone	591-78-6	NA	5.0	Not Available in Method	Not Available in Method	2.5	5.0	55-130	55-130	30	NA	50
4-Methyl-2-pentanone	108-10-1	NA	5.0	Not Available in Method	Not Available in Method	2.5	5.0	60-135	60-135	30	NA	50
Methyl bromide	74-83-9	NA	2.0	Not Available in Method	Not Available in Method	1.0	2.0	30-145	30-145	30	NA	50
Methyl chloride	74-87-3	NA	5.0	Not Available in Method	Not Available in Method	1.0	5.0	40-125	40-125	30	NA	50
Methylene chloride	75-09-2	NA	5.0	Not Available in Method	Not Available in Method	2.5	5.0	55-140	55-140	30	NA	50
Methyl ethyl ketone	78-93-3	NA	5.0	Not Available in Method	Not Available in Method	2.5	5.0	30-150	30-150	30	NA	50
Styrene	100-42-5	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	65-135	65-135	30	NA	50
1,1,1-Trichloroethane	71-55-6	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	65-130	65-130	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
1,1,2,2-Tetrachloroethane	79-34-5	NA	1.0	Not Available in Method	Not Available in Method	0.4	1.0	65-130	65-130	30	NA	50
1,1,2-Trichloroethane	79-00-5	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	75-125	75-125	30	NA	50
Tetrachloroethylene	127-18-4	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	45-150	45-150	30	NA	50
Toluene	108-88-3	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	75-120	75-120	30	NA	50
Trichloroethylene	79-01-6	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	70-125	70-125	30	NA	50
Vinyl chloride	75-01-4	NA	1.0	Not Available in Method	Not Available in Method	0.5	1.0	50-145	50-145	30	NA	50
Xylene (total)	1330-20-7	NA	3.0	Not Available in Method	Not Available in Method	1.5	3.0	80-120	80-120	30	NA	50
Dibromofluoromethane	1868-53-7	NA	NA	NA	NA	NA	NA	NA	NA	NA	85-115	NA
Toluene-D8	2037-26-5	NA	NA	NA	NA	NA	NA	NA	NA	NA	85-120	NA
4-Bromofluorobenzene	460-00-4	NA	NA	NA	NA	NA	NA	NA	NA	NA	75-120	NA
1,2-Dichloroethane-D4	17060-07-0	NA	NA	NA	NA	NA	NA	NA	NA	NA	70-120	NA

NA = Not Applicable.

[1]The surface water samples are to be collected to characterize the water at the surface and are not to be compared to any specific criteria at this time; therefore, the action limits are not applicable. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.11 UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Aqueous
Analytical Group: TCL Semivolatiles Organic - SW-846 3510C/8270C
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Benzoic acid	65-85-0	NA	20	Not Available in Method	Not Available in Method	10	20	10-125	10-125	30	NA	50
2-Chlorophenol	95-57-8	NA	5	Not Available in Method	Not Available in Method	2	5	35-105	35-105	30	NA	50
4-Chloro-3-methyl phenol	59-50-7	NA	5	Not Available in Method	Not Available in Method	2	5	45-110	45-110	30	NA	50
2,4-Dichlorophenol	120-83-2	NA	5	Not Available in Method	Not Available in Method	2	5	50-105	50-105	30	NA	50
2,4-Dimethylphenol	105-67-9	NA	5	Not Available in Method	Not Available in Method	2	5	30-110	30-110	30	NA	50
2,4-Dinitrophenol	51-28-5	NA	25	Not Available in Method	Not Available in Method	10	25	15-140	15-140	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
4,6-Dinitro-o-cresol	534-52-1	NA	10	Not Available in Method	Not Available in Method	5	10	40-130	40-130	30	NA	50
2-Methylphenol	95-48-7	NA	5	Not Available in Method	Not Available in Method	2	5	40-110	40-110	30	NA	50
3&4-Methylphenol	NA	NA	5	Not Available in Method	Not Available in Method	1	5	30-110	30-110	30	NA	50
2-Nitrophenol	88-75-5	NA	5	Not Available in Method	Not Available in Method	1	5	40-115	40-115	30	NA	50
4-Nitrophenol	100-02-7	NA	25	Not Available in Method	Not Available in Method	10	25	10-125	10-125	30	NA	50
Pentachlorophenol	87-86-5	NA	25	Not Available in Method	Not Available in Method	10	25	40-115	40-115	30	NA	50
Phenol	108-95-2	NA	5	Not Available in Method	Not Available in Method	2	5	10-115	10-115	30	NA	50
2,4,5-Trichlorophenol	95-95-4	NA	5	Not Available in Method	Not Available in Method	2	5	50-110	50-110	30	NA	50
2,4,6-Trichlorophenol	88-06-2	NA	5	Not Available in Method	Not Available in Method	2	5	50-115	50-115	30	NA	50
Acenaphthene	83-32-9	NA	5	Not Available in Method	Not Available in Method	1	5	45-110	45-110	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Acenaphthylene	208-96-8	NA	5	Not Available in Method	Not Available in Method	1	5	50-105	50-105	30	NA	50
Anthracene	120-12-7	NA	5	Not Available in Method	Not Available in Method	1	5	55-110	55-110	30	NA	50
Benzo(a)anthracene	56-55-3	NA	5	Not Available in Method	Not Available in Method	1	5	55-110	55-110	30	NA	50
Benzo(a)pyrene	50-32-8	NA	5	Not Available in Method	Not Available in Method	1	5	55-110	55-110	30	NA	50
Benzo(b)fluoranthene	205-99-2	NA	5	Not Available in Method	Not Available in Method	1	5	45-120	45-120	30	NA	50
Benzo(g,h,i)perylene	191-24-2	NA	5	Not Available in Method	Not Available in Method	2	5	40-125	40-125	30	NA	50
Benzo(k)fluoranthene	207-08-9	NA	5	Not Available in Method	Not Available in Method	1	5	45-125	45-125	30	NA	50
4-Bromophenyl phenyl ether	101-55-3	NA	5	Not Available in Method	Not Available in Method	1	5	50-115	50-115	30	NA	50
Butyl benzyl phthalate	85-68-7	NA	5	Not Available in Method	Not Available in Method	2	5	45-115	45-115	30	NA	50
Benzyl Alcohol	100-51-6	NA	5	Not Available in Method	Not Available in Method	1	5	30-110	30-110	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
2-Chloronaphthalene	91-58-7	NA	5	Not Available in Method	Not Available in Method	1	5	50-105	50-105	30	NA	50
4-Chloroaniline	106-47-8	NA	10	Not Available in Method	Not Available in Method	4	10	15-110	15-110	30	NA	50
Carbazole	86-74-8	NA	5	Not Available in Method	Not Available in Method	1	5	50-115	50-115	30	NA	50
Chrysene	218-01-9	NA	5	Not Available in Method	Not Available in Method	1	5	55-110	55-110	30	NA	50
bis(2-Chloroethoxy)methane	111-91-1	NA	5	Not Available in Method	Not Available in Method	1	5	45-105	45-105	30	NA	50
bis(2-Chloroethyl)ether	111-44-4	NA	5	Not Available in Method	Not Available in Method	2	5	35-110	35-110	30	NA	50
bis(2-Chloroisopropyl)ether	108-60-1	NA	5	Not Available in Method	Not Available in Method	1	5	25-130	25-130	30	NA	50
4-Chlorophenyl phenyl ether	7005-72-3	NA	5	Not Available in Method	Not Available in Method	1	5	50-110	50-110	30	NA	50
1,2-Dichlorobenzene	95-50-1	NA	5	Not Available in Method	Not Available in Method	1	5	35-100	35-100	30	NA	50
1,3-Dichlorobenzene	541-73-1	NA	5	Not Available in Method	Not Available in Method	1	5	30-100	30-100	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
1,4-Dichlorobenzene	106-46-7	NA	5	Not Available in Method	Not Available in Method	1	5	30-100	30-100	30	NA	50
2,4-Dinitrotoluene	121-14-2	NA	5	Not Available in Method	Not Available in Method	2	5	50-120	50-120	30	NA	50
2,6-Dinitrotoluene	606-20-2	NA	5	Not Available in Method	Not Available in Method	2	5	50-115	50-115	30	NA	50
3,3'-Dichlorobenzidine	91-94-1	NA	10	Not Available in Method	Not Available in Method	5	10	20-110	20-110	30	NA	50
Dibenzo(a,h)anthracene	53-70-3	NA	5	Not Available in Method	Not Available in Method	2	5	40-125	40-125	30	NA	50
Dibenzofuran	132-64-9	NA	5	Not Available in Method	Not Available in Method	1	5	55-105	55-105	30	NA	50
Di-n-butyl phthalate	84-74-2	NA	5	Not Available in Method	Not Available in Method	2	5	55-115	55-115	30	NA	50
Di-n-octyl phthalate	117-84-0	NA	5	Not Available in Method	Not Available in Method	2.5	5	35-135	35-135	30	NA	50
Diethyl phthalate	84-66-2	NA	5	Not Available in Method	Not Available in Method	2	5	40-120	40-120	30	NA	50
Dimethyl phthalate	131-11-3	NA	5	Not Available in Method	Not Available in Method	2	5	25-125	25-125	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
bis(2-Ethylhexyl)phthalate	117-81-7	NA	5	Not Available in Method	Not Available in Method	2.5	5	40-125	40-125	30	NA	50
Fluoranthene	206-44-0	NA	5	Not Available in Method	Not Available in Method	1	5	55-110	55-110	30	NA	50
Fluorene	86-73-7	NA	5	Not Available in Method	Not Available in Method	1	5	50-110	50-110	30	NA	50
Hexachlorobenzene	118-74-1	NA	5	Not Available in Method	Not Available in Method	1	5	50-110	50-110	30	NA	50
Hexachlorobutadiene	87-68-3	NA	5	Not Available in Method	Not Available in Method	2	5	25-105	25-105	30	NA	50
Hexachlorocyclopentadiene	77-47-4	NA	5	Not Available in Method	Not Available in Method	2	5	25-105	25-105	30	NA	50
Hexachloroethane	67-72-1	NA	5	Not Available in Method	Not Available in Method	2	5	30-95	30-95	30	NA	50
Indeno(1,2,3-cd)pyrene	193-39-5	NA	5	Not Available in Method	Not Available in Method	2	5	45-125	45-125	30	NA	50
Isophorone	78-59-1	NA	5	Not Available in Method	Not Available in Method	1	5	50-110	50-110	30	NA	50
2-Methylnaphthalene	91-57-6	NA	5	Not Available in Method	Not Available in Method	1	5	45-105	45-105	30	NA	50
2-Nitroaniline	88-74-4	NA	10	Not Available in Method	Not Available in Method	4	10	50-115	50-115	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
3-Nitroaniline	99-09-2	NA	10	Not Available in Method	Not Available in Method	4	10	20-125	20-125	30	NA	50
4-Nitroaniline	100-01-6	NA	10	Not Available in Method	Not Available in Method	4	10	35-120	35-120	30	NA	50
Naphthalene	91-20-3	NA	5	Not Available in Method	Not Available in Method	1	5	40-100	40-100	30	NA	50
Nitrobenzene	98-95-3	NA	5	Not Available in Method	Not Available in Method	1	5	45-110	45-110	30	NA	50
N-Nitroso-di-n-propylamine	621-64-7	NA	5	Not Available in Method	Not Available in Method	2	5	35-130	35-130	30	NA	50
N-Nitrosodiphenylamine	86-30-6	NA	5	Not Available in Method	Not Available in Method	2	5	50-110	50-110	30	NA	50
Phenanthrene	85-01-8	NA	5	Not Available in Method	Not Available in Method	1	5	50-115	50-115	30	NA	50
Pyrene	129-00-0	NA	5	Not Available in Method	Not Available in Method	1	5	50-130	50-130	30	NA	50
1,2,4-Trichlorobenzene	120-82-1	NA	5	Not Available in Method	Not Available in Method	1	5	35-105	35-105	30	NA	50
2-Fluorophenol	367-12-4	NA	NA	NA	NA	NA	NA	NA	NA	NA	50-110	NA
Phenol-d5	4165-62-2	NA	NA	NA	NA	NA	NA	NA	NA	NA	10-115	NA

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
2,4,6-Tribromophenol	118-79-6	NA	NA	NA	NA	NA	NA	NA	NA	NA	40-125	NA
Nitrobenzene-d5	4165-60-0	NA	NA	NA	NA	NA	NA	NA	NA	NA	40-110	NA
2-Fluorobiphenyl	321-60-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	50-110	NA
Terphenyl-d14	1718-51-0	NA	NA	NA	NA	NA	NA	NA	NA	NA	50-135	NA

NA = Not Applicable.

[1]The surface water samples are to be collected to characterize the water at the surface and are not to be compared to any specific criteria at this time; therefore, the action limits are not applicable. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.12 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Aqueous
Analytical Group: Explosives Organic - SW-846 3535A/8330
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
HMX	2691-41-0	NA	0.2	Not Available in Method	Not Available in Method	0.06	0.2	80-115	80-115	30	NA	50
RDX	121-82-4	NA	0.2	Not Available in Method	Not Available in Method	0.075	0.2	50-160	50-160	30	NA	50
1,3-Dinitrobenzene	99-65-0	NA	0.2	Not Available in Method	Not Available in Method	0.07	0.2	45-160	45-160	30	NA	50
2,6-Dinitrotoluene	606-20-2	NA	0.2	Not Available in Method	Not Available in Method	0.075	0.2	60-135	60-135	30	NA	50
2,4-Dinitrotoluene	121-14-2	NA	0.2	Not Available in Method	Not Available in Method	0.065	0.2	60-135	60-135	30	NA	50
2-amino-4,6-Dinitrotoluene	35572-78-2	NA	0.2	Not Available in Method	Not Available in Method	0.08	0.2	50-155	50-155	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
4-amino-2,6-Dinitrotoluene	19406-51-0	NA	0.2	Not Available in Method	Not Available in Method	0.07	0.2	55-155	55-155	30	NA	50
Nitrobenzene	98-95-3	NA	0.2	Not Available in Method	Not Available in Method	0.06	0.2	50-140	50-140	30	NA	50
o-Nitrotoluene	88-72-2	NA	0.2	Not Available in Method	Not Available in Method	0.06	0.2	45-135	45-135	30	NA	50
m-Nitrotoluene	99-08-1	NA	0.2	Not Available in Method	Not Available in Method	0.075	0.2	50-130	50-130	30	NA	50
p-Nitrotoluene	99-99-0	NA	0.2	Not Available in Method	Not Available in Method	0.075	0.2	50-130	50-130	30	NA	50
Tetryl	479-45-8	NA	0.2	Not Available in Method	Not Available in Method	0.075	0.2	20-175	20-175	30	NA	50
1,3,5-Trinitrobenzene	99-35-4	NA	0.2	Not Available in Method	Not Available in Method	0.095	0.2	65-140	65-140	30	NA	50
2,4,6-Trinitrotoluene	118-96-7	NA	0.2	Not Available in Method	Not Available in Method	0.08	0.2	50-145	50-145	30	NA	50
3,4-Dinitrotoluene	610-39-9	NA	NA	NA	NA	NA	NA	NA	NA	NA	61-124	NA

NA = Not Applicable.

[1]The surface water samples are to be collected to characterize the water at the surface and are not to be compared to any specific criteria at this time; therefore, the action limits are not applicable. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.13 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Aqueous
Analytical Group: TCL Pesticides Organic - SW-846 3510C/8081A
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Aldrin	309-00-2	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	25-140	25-140	30	NA	50
alpha-BHC	319-84-6	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	60-130	60-130	30	NA	50
beta-BHC	319-85-7	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	65-125	65-125	30	NA	50
delta-BHC	319-86-8	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	45-135	45-135	30	NA	50
gamma-BHC (Lindane)	58-89-9	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	25-135	25-135	30	NA	50
alpha-Chlordane	5103-71-9	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	65-125	65-125	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
gamma-Chlordane	5103-74-2	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	60-125	60-125	30	NA	50
Dieldrin	60-57-1	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	60-130	60-130	30	NA	50
4,4'-DDD	72-54-8	NA	0.1	Not Available in Method	Not Available in Method	0.02	0.1	25-150	25-150	30	NA	50
4,4'-DDE	72-55-9	NA	0.1	Not Available in Method	Not Available in Method	0.02	0.1	35-140	35-140	30	NA	50
4,4'-DDT	50-29-3	NA	0.1	Not Available in Method	Not Available in Method	0.02	0.1	45-140	45-140	30	NA	50
Endrin	72-20-8	NA	0.1	Not Available in Method	Not Available in Method	0.04	0.1	55-135	55-135	30	NA	50
Endosulfan sulfate	1031-07-8	NA	0.1	Not Available in Method	Not Available in Method	0.02	0.1	55-135	55-135	30	NA	50
Endrin aldehyde	7421-93-4	NA	0.1	Not Available in Method	Not Available in Method	0.02	0.1	55-135	55-135	30	NA	50
Endrin ketone	53494-70-5	NA	0.1	Not Available in Method	Not Available in Method	0.02	0.1	75-125	75-125	30	NA	50
Endosulfan-I	959-98-8	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	50-110	50-110	30	NA	50
Endosulfan-II	33213-65-9	NA	0.1	Not Available in Method	Not Available in Method	0.02	0.1	30-130	30-130	30	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Heptachlor	76-44-8	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	40-130	40-130	30	NA	50
Heptachlor epoxide	1024-57-3	NA	0.05	Not Available in Method	Not Available in Method	0.02	0.05	60-130	60-130	30	NA	50
Methoxychlor	72-43-5	NA	0.1	Not Available in Method	Not Available in Method	0.04	0.1	55-150	55-150	30	NA	50
Toxaphene	8001-35-2	NA	2.5	Not Available in Method	Not Available in Method	1.3	2.5	50-150	50-150	30	NA	50
Tetrachloro-m-xylene	877-09-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	25-140	NA
Decachlorobiphenyl	2051-24-3	NA	NA	NA	NA	NA	NA	NA	NA	NA	30-135	NA

NA = Not Applicable.

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[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.14 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Aqueous
Analytical Group: Organic – PCBs by SW-846 3510C/8082
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Aroclor 1016	12674-11-2	NA	0.50	Not Available in Method	Not Available in Method	0.25	0.50	25-145	25-145	30	NA	50
Aroclor 1221	11104-28-2	NA	0.50	Not Available in Method	Not Available in Method	0.40	0.50	NA	NA	NA	NA	50
Aroclor 1232	11141-16-5	NA	0.50	Not Available in Method	Not Available in Method	0.40	0.50	NA	NA	NA	NA	50
Aroclor 1242	53469-21-9	NA	0.50	Not Available in Method	Not Available in Method	0.25	0.50	NA	NA	NA	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Aroclor 1248	12672-29-6	NA	0.50	Not Available in Method	Not Available in Method	0.25	0.50	NA	NA	NA	NA	50
Aroclor 1254	11097-69-1	NA	0.50	Not Available in Method	Not Available in Method	0.25	0.50	NA	NA	NA	NA	50
Aroclor 1260	11096-82-5	NA	0.50	Not Available in Method	Not Available in Method	0.25	0.50	30-145	30-145	30	NA	50
Tetrachloro-m-xylene	877-09-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	49-124	NA
Decachlorobiphenyl	2051-24-3	NA	NA	NA	NA	NA	NA	NA	NA	NA	40-135	NA

NA = Not Applicable.

[1]The surface water samples are to be collected to characterize the water at the surface and are not to be compared to any specific criteria at this time; therefore, the action limits are not applicable. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.15 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Aqueous
Analytical Group: Organic – TPH DRO SW-846 3510C/8015M
Concentration Level: Low

				Analytical Method[2]		**Achievable Laboratory Limits[3]**		**Precision and Accuracy Method Performance Criteria[4]**				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
TPH-DRO (C10-C28)	NA	NA	8.3	Not Available in Method	Not Available in Method	5.0	8.3	57-116	41-146	29	NA	50
o-Terphenyl	84-15-1	NA	NA	NA	NA	NA	NA	NA	NA	NA	44-123	NA

NA = Not Applicable.

[1]The surface water samples are to be collected to characterize the water at the surface and are not to be compared to any specific criteria at this time; therefore, the action limits are not applicable. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.16 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Aqueous
Analytical Group: Inorganic TAL Metals - SW-846 3010A/6010B/7470A
Concentration Level: Low

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Aluminum	7429-90-5	NA	200	Not Available in Method	Not Available in Method	18	200	80-120	80-120	20	NA	50
Antimony	7440-36-0	NA	6.0	Not Available in Method	Not Available in Method	3.4	6.0	80-120	80-120	20	NA	50
Arsenic	7440-38-2	NA	10	Not Available in Method	Not Available in Method	2.8	10	80-120	80-120	20	NA	50
Barium	7440-39-3	NA	200	Not Available in Method	Not Available in Method	0.4	200	80-120	80-120	20	NA	50
Beryllium	7440-41-7	NA	4.0	Not Available in Method	Not Available in Method	0.4	4.0	80-120	80-120	20	NA	50
Cadmium	7440-43-9	NA	5.0	Not Available in Method	Not Available in Method	0.3	5.0	80-120	80-120	20	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Calcium	7440-70-2	NA	1000	Not Available in Method	Not Available in Method	42	1000	80-120	80-120	20	NA	50
Chromium	7440-47-3	NA	10	Not Available in Method	Not Available in Method	0.6	10	80-120	80-120	20	NA	50
Cobalt	7440-48-4	NA	50	Not Available in Method	Not Available in Method	0.6	50	80-120	80-120	20	NA	50
Copper	7440-50-8	NA	25	Not Available in Method	Not Available in Method	1.0	25	80-120	80-120	20	NA	50
Iron	7439-89-6	NA	300	Not Available in Method	Not Available in Method	15	300	80-120	80-120	20	NA	50
Lead	7439-92-1	NA	5.0	Not Available in Method	Not Available in Method	1.7	5.0	80-120	80-120	20	NA	50
Magnesium	7439-95-4	NA	5000	Not Available in Method	Not Available in Method	4.3	5000	80-120	80-120	20	NA	50
Manganese	7439-96-5	NA	15	Not Available in Method	Not Available in Method	1.5	15	80-120	80-120	20	NA	50
Mercury	7439-97-6	NA	1.0	Not Available in Method	Not Available in Method	0.1	1.0	80-120	80-120	20	NA	50
Nickel	7440-02-0	NA	40	Not Available in Method	Not Available in Method	0.8	40	80-120	80-120	20	NA	50
Potassium	7440-09-7	NA	10000	Not Available in Method	Not Available in Method	100	10000	80-120	80-120	20	NA	50

				Analytical Method[2]		Achievable Laboratory Limits[3]		Precision and Accuracy Method Performance Criteria[4]				
Analyte	**CAS Number**	**Project Action Limit[1] (µg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (µg/L)**	**MDLs (µg/L)**	**Method QLs (µg/L)**	**MDLs (µg/L)**	**QLs (µg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Selenium	7782-49-2	NA	10	Not Available in Method	Not Available in Method	2.8	10	80-120	80-120	20	NA	50
Silver	7440-22-4	NA	10	Not Available in Method	Not Available in Method	0.9	10	80-120	80-120	20	NA	50
Sodium	7440-23-5	NA	10000	Not Available in Method	Not Available in Method	500	10000	80-120	80-120	20	NA	50
Thallium	7440-28-0	NA	10	Not Available in Method	Not Available in Method	2.9	10	80-120	80-120	20	NA	50
Vanadium	7440-62-2	NA	50	Not Available in Method	Not Available in Method	1.1	50	80-120	80-120	20	NA	50
Zinc	7440-66-6	NA	20	Not Available in Method	Not Available in Method	1.6	20	80-120	80-120	20	NA	50

NA = Not Applicable.

[1]The surface water samples are to be collected to characterize the water at the surface and are not to be compared to any specific criteria at this time; therefore, the action limits are not applicable. Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Analytical MDLs and QLs are not available in this SW-846 for this method.

[3]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[4]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM.

QAPP Worksheet #15.17 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil
Analytical Group: Organics - TCLP Volatiles SW-846 1311/5030B/8260B
Concentration Level: Low

				Analytical Method		Achievable Laboratory Limits[2]		Precision and Accuracy Method Performance Criteria[3]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Benzene	71-43-2	0.5	0.01	Not Available in Method	Not Available in Method	0.005	0.01	80-120	NA	NA	NA	NA
Chlorobenzene	108-90-7	100	0.02	Not Available in Method	Not Available in Method	0.005	0.02	80-120	NA	NA	NA	NA
Chloroform	67-66-3	6.0	0.02	Not Available in Method	Not Available in Method	0.005	0.02	65-135	NA	NA	NA	NA
Carbon tetrachloride	56-23-5	0.5	0.02	Not Available in Method	Not Available in Method	0.005	0.02	65-140	NA	NA	NA	NA
1,1-Dichloroethylene	75-35-4	0.7	0.02	Not Available in Method	Not Available in Method	0.005	0.02	70-130	NA	NA	NA	NA
1,2-Dichloroethane	107-06-2	0.5	0.02	Not Available in Method	Not Available in Method	0.005	0.02	80-120	NA	NA	NA	NA
1,4-Dichlorobenzene	106-46-7	7.5	0.02	Not Available in Method	Not Available in Method	0.005	0.02	75-125	NA	NA	NA	NA
Methyl ethyl ketone	78-93-3	200	0.10	Not Available in Method	Not Available in Method	0.025	0.10	30-150	NA	NA	NA	NA
Tetrachloroethylene	127-18-4	0.7	0.02	Not Available in Method	Not Available in Method	0.005	0.02	45-150	NA	NA	NA	NA

				Analytical Method		Achievable Laboratory Limits[2]		Precision and Accuracy Method Performance Criteria[3]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Trichloroethylene	79-01-6	0.5	0.02	Not Available in Method	Not Available in Method	0.005	0.02	70-125	NA	NA	NA	NA
Vinyl chloride	75-01-4	0.2	0.01	Not Available in Method	Not Available in Method	0.005	0.01	50-145	NA	NA	NA	NA
Dibromofluoromethane	1868-53-7	NA	NA	NA	NA	NA	NA	NA	NA	NA	85-115	NA
Toluene-D8	2037-26-5	NA	NA	NA	NA	NA	NA	NA	NA	NA	85-120	NA
4-Bromofluorobenzene	460-00-4	NA	NA	NA	NA	NA	NA	NA	NA	NA	75-120	NA
1,2-Dichloroethane-D4	17060-07-0	NA	NA	NA	NA	NA	NA	NA	NA	NA	70-120	NA

NA = Not Applicable.

[1]Project action limits are based upon USEPA TCLP Maximum Concentration of Contaminants 40CFR 261 (June, 1996). Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[3]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM. No field duplicate or MS/MSD are required for TCLP analysis.

QAPP Worksheet #15.18 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil
Analytical Group: Organics - TCLP Semivolatiles SW-846 1311/3510C/8270C
Concentration Level: Low

				Analytical Method		Achievable Laboratory Limits[2]		Precision and Accuracy Method Performance Criteria[3]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
2-Methylphenol	95-48-7	200	0.05	Not Available in Method	Not Available in Method	0.02	0.05	40-110	NA	NA	NA	NA
3&4-Methylphenol	NA	200	0.05	Not Available in Method	Not Available in Method	0.02	0.05	30-110	NA	NA	NA	NA
Pentachlorophenol	87-86-5	100	0.25	Not Available in Method	Not Available in Method	0.1	0.25	40-115	NA	NA	NA	NA
2,4,5-Trichlorophenol	95-95-4	400	0.05	Not Available in Method	Not Available in Method	0.02	0.05	50-110	NA	NA	NA	NA
2,4,6-Trichlorophenol	88-06-2	2.0	0.05	Not Available in Method	Not Available in Method	0.02	0.05	50-115	NA	NA	NA	NA
1,4-Dichlorobenzene	106-46-7	7.5	0.05	Not Available in Method	Not Available in Method	0.01	0.05	30-100	NA	NA	NA	NA

				Analytical Method		Achievable Laboratory Limits[2]		Precision and Accuracy Method Performance Criteria[3]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
2,4-Dinitrotoluene	121-14-2	0.13	0.05	Not Available in Method	Not Available in Method	0.02	0.05	50-120	NA	NA	NA	NA
Hexachlorobenzene	118-74-1	0.13	0.05	Not Available in Method	Not Available in Method	0.01	0.05	50-110	NA	NA	NA	NA
Hexachlorobutadiene	87-68-3	0.5	0.05	Not Available in Method	Not Available in Method	0.02	0.05	25-105	NA	NA	NA	NA
Hexachloroethane	67-72-1	3.0	0.05	Not Available in Method	Not Available in Method	0.02	0.05	30-95	NA	NA	NA	NA
Nitrobenzene	98-95-3	2.0	0.05	Not Available in Method	Not Available in Method	0.01	0.05	45-110	NA	NA	NA	NA
Pyridine	110-86-1	5.0	0.10	Not Available in Method	Not Available in Method	0.03	0.10	50-150	NA	NA	NA	NA
2-Fluorophenol	367-12-4	NA	NA	NA	NA	NA	NA	NA	NA	NA	50-110	NA
Phenol-d5	4165-62-2	NA	NA	NA	NA	NA	NA	NA	NA	NA	10-115	NA
2,4,6-Tribromophenol	118-79-6	NA	NA	NA	NA	NA	NA	NA	NA	NA	40-125	NA
Nitrobenzene-d5	4165-60-0	NA	NA	NA	NA	NA	NA	NA	NA	NA	40-110	NA
2-Fluorobiphenyl	321-60-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	50-110	NA
Terphenyl-d14	1718-51-0	NA	NA	NA	NA	NA	NA	NA	NA	NA	50-135	NA

NA = Not Applicable.

[1]Project action limits are based upon USEPA TCLP Maximum Concentration of Contaminants 40CFR 261 (June, 1996). Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[3]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM. No field duplicate or MS/MSD are required for TCLP analysis.

QAPP Worksheet #15.19 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil
Analytical Group: Organics - TCLP Pesticide SW-846 1311/3510C/8081A
Concentration Level: Low

				Analytical Method		Achievable Laboratory Limits[2]		Precision and Accuracy Method Performance Criteria[3]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** *Equal to or Less Than*	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
gamma-BHC (Lindane)	58-89-9	0.4	0.0005	Not Available in Method	Not Available in Method	0.0002	0.0005	25-135	NA	NA	NA	NA
Chlordane	12789-03-6	0.03	0.005	Not Available in Method	Not Available in Method	0.0025	0.005	60-125	NA	NA	NA	NA
Endrin	72-20-8	0.02	0.001	Not Available in Method	Not Available in Method	0.0004	0.001	55-135	NA	NA	NA	NA
Heptachlor	76-44-8	0.008	0.0005	Not Available in Method	Not Available in Method	0.0002	0.0005	40-130	NA	NA	NA	NA
Heptachlor epoxide	1024-57-3	0.008	0.0005	Not Available in Method	Not Available in Method	0.0002	0.0005	60-130	NA	NA	NA	NA
Methoxychlor	72-43-5	10	0.0010	Not Available in Method	Not Available in Method	0.0004	0.0010	55-150	NA	NA	NA	NA
Toxaphene	8001-35-2	0.5	0.025	Not Available in Method	Not Available in Method	0.015	0.025	50-150	NA	NA	NA	NA

				Analytical Method		Achievable Laboratory Limits[2]		Precision and Accuracy Method Performance Criteria[3]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Tetrachloro-m-xylene	877-09-8	NA	NA	NA	NA	NA	NA	NA	NA	NA	25-140	NA
Decachlorobiphenyl	2051-24-3	NA	NA	NA	NA	NA	NA	NA	NA	NA	30-135	NA

NA = Not Applicable.

[1]Project action limits are based upon USEPA TCLP Maximum Concentration of Contaminants 40CFR 261 (June, 1996). Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[3]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM. No field duplicate or MS/MSD are required for TCLP analysis.

QAPP Worksheet #15.20 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil
Analytical Group: Organics - TCLP Herbicides SW-846 1311/8151A
Concentration Level: Low

				Analytical Method		**Achievable Laboratory Limits[2]**		**Precision and Accuracy Method Performance Criteria[3]**				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
2,4-D	94-75-7	10	0.015	Not Available in Method	Not Available in Method	0.011	0.015	35-115	NA	NA	NA	NA
2,4,5-TP (Silvex)	93-72-1	1.0	0.0020	Not Available in Method	Not Available in Method	0.0006	0.0020	50-115	NA	NA	NA	NA
2,4-Dichlorophenylacetic acid	19719-28-9	NA	NA	NA	NA	NA	NA	NA	NA	NA	50-150	NA

NA = Not Applicable.

[1]Project action limits are based upon USEPA TCLP Maximum Concentration of Contaminants 40CFR 261 (June, 1996). Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[3]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM. No field duplicate or MS/MSD are required for TCLP analysis.

QAPP Worksheet #15.21 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Next, list the published and achievable detection and quantitation limits for each analyte. Finally, enter the precision and accuracy method performance criteria for each analyte and surrogates (if applicable) to be used.

Matrix: Soil
Analytical Group: Inorganics - TCLP Metals SW-846 1311/3010A/6010B/7470A
Concentration Level: Low

				Analytical Method		Achievable Laboratory Limits[2]		Precision and Accuracy Method Performance Criteria[3]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Arsenic	7440-38-2	5.0	0.02	Not Available in Method	Not Available in Method	0.0029	0.02	80-120	NA	NA	NA	NA
Barium	7440-39-3	100	1.0	Not Available in Method	Not Available in Method	0.2	1.0	80-120	NA	NA	NA	NA
Cadmium	7440-43-9	1.0	0.005	Not Available in Method	Not Available in Method	0.0003	0.005	80-120	NA	NA	NA	NA
Chromium	7440-47-3	5.0	0.010	Not Available in Method	Not Available in Method	0.0006	0.010	80-120	NA	NA	NA	NA
Lead	7439-92-1	5.0	0.050	Not Available in Method	Not Available in Method	0.0017	0.050	80-120	NA	NA	NA	NA
Mercury	7439-97-6	0.2	0.010	Not Available in Method	Not Available in Method	0.0008	0.010	80-120	NA	NA	NA	NA

				Analytical Method		Achievable Laboratory Limits[2]		Precision and Accuracy Method Performance Criteria[3]				
Analyte	**CAS Number**	**Project Action Limit[1] (mg/L)** ***Equal to or Less Than***	**Project Quantitation Limit Goal[1] (mg/L)**	**MDLs (mg/L)**	**Method QLs (mg/L)**	**MDLs (mg/L)**	**QLs (mg/L)**	**LCS Control Limit (%R)**	**MS/MSD Control Limit (%R)**	**MS/MSD Precision Limit (RPD)**	**Surrogate Control Limit (%R)**	**Project Field Precision Limit (RPD)**
Selenium	7782-49-2	1.0	0.050	Not Available in Method	Not Available in Method	0.02	0.050	80-120	NA	NA	NA	NA
Silver	7440-22-4	5.0	0.010	Not Available in Method	Not Available in Method	0.0009	0.010	80-120	NA	NA	NA	NA

NA = Not Applicable.

[1]Project action limits are based upon USEPA TCLP Maximum Concentration of Contaminants 40CFR 261 (June, 1996). Project Action Limits presented in bold represent values below project quantitation limits and those presented in bold italic represent values below achievable method detection limits.

[2]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. *Laboratory Generated Limits are subject to change, the laboratory will use the most current limits at the time of analysis.*

[3]The laboratory precision and accuracy method performance criteria are based upon the *DoD Quality Systems Manual for Environmental Laboratories* (DoD QSM), Version 3, January 2006. If a compound/analyte is not listed, then the established laboratory in-house limits are listed per DoD QSM. No field duplicate or MS/MSD are required for TCLP analysis.

QAPP Worksheet #16
Project Schedule / Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Work Plan Preparation	FSSI/Shaw	07/25/2005	08/22/2007	Work Plan Document	08/22/2007
QAPP Preparation	FSSI/Shaw.	07/1/2006	08/22/2007	QAPP Document	08/22/2007
Waste Characterization Sampling & Laboratory Analysis	FSSI/Shaw	10/29/2007	11/21/2007	Analytical Report	12/05/2007
Field Sampling Audit	FSSI/Shaw	10/29/2007	11/21/2007	Daily QC Report	12/05/2007
Data Review & Sample Validation	FSSI/Shaw	11/21/2007	01/18/2008	Validation Report	01/18/2008
Site Mobilization: Personnel and Equipment	FSSI/Shaw	05/19/2008	05/20/2008	NA	NA
SWMU-7 Removal Action	FSSI/Shaw	05/20/2008	06/13/2008	NA	NA
SWMU-6 Removal Action	FSSI/Shaw	06/16/2008	07/09/2007	NA	NA
AOC-J Removal Action	FSSI/Shaw	07/10/2008	08/01/2008	NA	NA
AOC-R Removal Action	FSSI/Shaw	08/04/2008	09/03/2008	NA	NA
Demobilization	FSSI/Shaw	09/04/2008	09/05/2008	NA	NA
Closure Report Preparation	FSSI/Shaw	11/01/2008	01/31/2009	Project Closure Report	01/31/2009

QAPP Worksheet #17
Sampling Design and Rationale

WASTE CHARACTERIZATION SOIL SAMPLING

Waste characterization sampling is the sampling that will be done to guide the removal activity and waste disposal. That is, the data will be used to help estimate the extent of contaminated soil removal that will be conducted in conjunction with the debris removal and to determine the disposal options for the excavated materials.

Waste Characterization Sample Location Identification, Collection, and Analysis

Based on historical figures showing the extent of debris, vegetation clearance will be performed to expose the debris areas at all sites (AOCJ, AOC R, SWMU 6, and SWMU 7). The sites will then be visually inspected to confirm the extent of the debris, and additional vegetation clearance will be conducted if necessary to ensure the debris areas have been adequately cleared of vegetation.

Following vegetation clearance, pin flags will be placed at the locations of soil samples collected during previous investigations at the sites. Next, pin flags (of a different color from those representing historical soil sample locations) will be placed in a grid pattern across the debris area at each site at a spacing of one pin flag per 2,000 square feet (sf), including the pin flags representing samples from previous investigations. Data from previous sample locations will be included in the characterization dataset, rather than be re-sampled. Attached are maps for each of the sites showing the default locations of the proposed samples to be collected at this frequency. During placement of the pin flags, their locations will be adjusted to bias them toward observed debris, under the assumption that the debris is likely to represent areas with the highest concentrations of contaminants. At the direction of the Navy, additional samples may be added if there is evidence of a release, or other contamination. The addition of additional samples shall be agreed upon by the Navy and regulatory agencies.

Once all of the proposed waste characterization sample locations have been pin flagged, a site visit will be conducted by representatives from the Environmental Technical Subcommittee to concur upon the sample locations. The site visit will include the use of metal detectors to assist in confirming and/or adjusting sample locations.

Once the waste characterization sample locations have been concurred upon, the waste characterization samples will be collected with the aid of a backhoe. At each sample location, the backhoe will excavate debris/soil through the entire vertical extent of debris. A 3-point composite soil sample (representing the near-surface , middle, and near bottom vertical extent of the debris) will be collected for analysis of Target Compound List (TCL) semi-volatile organic compounds (SVOCs), pesticides, and polychlorinated biphenyls (PCBs), explosives, and Target Analyte List (TAL) metals. A photo-ionization detector (PID), or similar, will be used to screen the vertical debris interval and a discrete soil sample will be collected from the location of the highest PID reading (or visual

staining in the absence of an elevated PID reading) for TCL volatile organic compound (VOC), TCLP VOC's, and GRO analysis. In the absence of a PID reading above background, and in the absence of any visual soil staining, the soil sample for VOC analysis will be collected from the middle of the debris interval. The 3-point composite samples will also be analyzed for full toxicity characteristic leaching procedure (TCLP); reactivity, corrosivity, ignitability (RCI); and total petroleum hydrocarbons (TPH).

Evaluation of Waste Characterization Data to Determine Extent of Removal and Disposal Options

The removal action at each site will be a combination of debris and soil removal. The extent of debris removal will be based on visual observations. The extent of contaminated soil removal will be based on visual observations (e.g., staining, elevated PID readings) and removal goals (i.e., soil constituent concentrations) based on quantitative human health and ecological risk assessments performed using the waste characterization data. Because residual human health and ecological risk assessments will be performed using the post-removal confirmatory data, Target Contaminant Concentrations shall be developed through the results of the pre-removal risk assessment and will be used internally (Navy and removal contractor) to establish intermediate guidelines for the removal action. Therefore, these Target Contaminant Concentrations are not subject to regulatory comment. However, the information will be provided to the regulatory agencies for informational purposes.

The disposal option(s) for the debris and contaminated soil will be based on the results of the TCLP/RCI/TPH/VOC analysis and a disposal-specific human health risk assessment (see "Debris and Soil Disposal" below).

Debris and Soil Disposal

Debris and soil will be excavated from the site and be temporarily stockpiled on 2 layers of 8-mil plastic sheeting. The debris and soil shall be segregated by excavator, backhoe, and by manual labor, into two distinct plies: 1) Soil and Trash and 2) Metal and large debris. The metal shall be placed into a roll-off container for transport to Puerto Rico for recycling within an approved facility. The debris shall be placed within an additional roll-off for transport to an approved landfill facility on the island of Puerto Rico. The soil and trash pile shall be loaded onto haul trucks by an excavator for transport to the Vieques Municipal Landfill. All efforts shall be made to segregate as much of the trash, so that the soil may be used for daily cover. Trash and debris 6 inches in size or greater shall be segregated wherever possible. The remaining trash and soil shall be placed within the Vieques Municipal Landfill.

The TCL, TAL, and explosives analytical results from the waste characterization samples will be used to develop a risk assessment to determine the proper disposition for the soil. If the data show that the soil is hazardous and cannot be accepted by the municipal landfill on Vieques, arrangements will be needed to coordinate off-island hazardous waste disposal. If the TCLP/RCI data show that the soil is non-hazardous the disposition of the soil shall be guided by the following:

If there is no unacceptable risk for the intended land use, the soil can remain onsite. If there are unacceptable risks determined by the risk assessment, the soil cannot remain onsite without modifying the intended land use. The disposal disposition shall be as follows:

To determine whether the soil can be used as daily cover, the waste characterization data (including applicable historical data) will be used to perform a human health risk assessment. The risk assessment will be performed using industrial PRGs as screening criteria and a maintenance worker exposure scenario. West Vieques background soil metals data will be used for comparison to the concentrations of any metal constituents of concern (COCs) identified via the risk assessment. If no unacceptable hazards and risks are identified (over that of background), the soil will be proposed as daily cover. The Municipality of Vieques has provided Shaw and the Navy with a letter confirming that the soil proposed to be used as daily cover, will be segregated from the soil available to the public. Otherwise, disposal of the soil in the Vieques landfill will be proposed. Following completion of the disposal-specific risk assessment, the risk assessment results (including all the waste characterization data), the proposed disposal option(s), and the proposed confirmatory sample locations will be provided to the regulatory agencies for consideration and a meeting/call will be conducted to reach consensus on the debris and soil disposal option(s). During this meeting/call, the tentative locations of the confirmatory samples will also be concurred upon. The disposal option(s) and confirmatory sample protocol will be formalized in an attachment to the removal action work plan.

POST-EXCAVATION CONFIRMATORY SAMPLING, ANALYSIS, AND EVALUATION

Once the removal action has been preliminarily determined to be complete (i.e., based on visual observations and the results of any unofficial confirmatory data collected and compared to removal goals), post-excavation confirmatory sampling will be performed in accordance with the confirmatory sampling protocol provided in the work plan attachment (discussed above). The general post-excavation confirmatory sample collection process will be as follows:

If, after debris removal, only soil is encountered below the debris, only soil confirmatory samples will be collected from the excavated areas. All soil samples will be analyzed for TCL VOCs, SVOCs, pesticides, and PCBs; TAL metals; and explosives. These confirmatory data will be used to perform human and ecological risk assessments in accordance with the HHRA and ERA protocols in the Master QAPP (CH2M HILL, May 2007).

If, after debris removal, groundwater is encountered below isolated areas of debris, the confirmatory soil sample will be collected from the sidewall of the excavation just above the groundwater level. The remainder of the sampling, analysis, and evaluation will be as described above.

If, after debris removal, the area of an existing surface water body is expanded (i.e., at SWMU 6), surface water and sediment samples will be collected in lieu of confirmatory soil samples in the expanded surface water body area. All confirmatory surface water and sediment (and soil if applicable) samples collected from the excavation area will be analyzed as above. Surface Water and Sediment samples shall be used to characterize the surface water only, and shall not be used to perform human health and ecological risk assessments in accordance with the HHRA and ERA protocols in the Master QAPP (CH2M HILL, February 2007).

If, debris removal takes place within an existing body of water, the sediment directly below the debris and the surface water shall be sampled as described above.

POTENTIAL ADDITIONAL EXCAVATION AND SAMPLE COLLECTION

The risk assessments performed using the post-excavation confirmatory samples will determine whether the site can be proposed for unrestricted land use. If the results of the risk assessments suggest unrestricted land use is not appropriate, but that land use in accordance with the intended future use of the land is appropriate, the Navy may opt to deem the removal action complete and summarize the removal action and associated risk assessment findings, conclusions, and recommendations in the construction completion report. If, alternatively, the residual risk assessments indicate land use in accordance with the intended future use is not appropriate or if the Navy decides an unrestricted land use determination is possible if some isolated areas of contamination are removed, additional excavation may be performed. In this circumstance, additional confirmatory samples will be collected in the areas of additional excavation in accordance with the protocol discussed above. The data from these additional confirmatory samples will replace previous confirmatory data from those areas that were additionally excavated, and the HHRA and ERA will be revised accordingly.

QAPP Worksheet #18
Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (ft)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]	Rationale for Sampling Location
Excavation Areas/ PR-116701-01-DS-00X	Soil	--	Full TCLP (VOCs, SVOCs, Herbicides, Pesticides, Metals), TPH DRO/GRO, total cyanide and sulfide, corrosivity,ignitability, Explosives, TAL Metals, TCL Volatiles, TCL Semivolatiles, TCL Pest/PCBs	Low	1 per 2000 ft^2/as deemed necessary by visual observation		Profiling the soil to verify that it is not hazardous and/or if it can be used as daily cover or left onsite.
Floor of excavation/ PR-116701-01-FLR-00X	Soil/Sediment	0.5	Explosives, TAL Metals, TCL Volatiles, TCL Semivolatiles, TCL Pest/PCBs, TPH DRO/GRO	Low	TBD[2]		Confirm that the removal goals have been met.
Surface Water Floor of excavation/ PR-116701-01-SW-FLR-00X	Surface water	--	Explosives, TAL Metals, TCL Volatiles, TCL Semivolatiles, TCL Pest/PCBs, TPH DRO/GRO	Low	If encountered.		Confirm that the removal goals have been met.

[1]Specify the appropriate letter or number from the Project Sampling SOP References table (Worksheet #21).

[2]Confirmatory sampling protocol shall be determined after evaluation of initial sampling data compared to the site specific project action limits.

QAPP Worksheet #19 (UFP-QAPP Manual Section 3.1.1) -- Analytical SOP Requirements Table

For each matrix, analytical group, and concentration level, list the analytical and preparation method/SOP and associated sample volume, container specifications, preservation requirements, and maximum holding time.

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method / SOP Reference[2]	Sample Size	Containers (number, size, and type)[1]	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Soil	TPH GRO	Low	SW-846 5035A 8015M/ SOP # OP020/GC010	15 grams	(3) 5g EnCore Samplers	Cool 4°	48 hours to preservation/14 Days
Soil	TCL VOC	Low	SW-846 5035A 8260B/ SOP #MS005/OP020	15 grams		Cool 4°	48 hours to preservation/14 Days
Soil	TCL SVOC	Low	SW-846 3550B 8270C/ SOP # OP007/MS006	30 grams	(2)-8oz jar	Cool 4°	14 days / 40 days
Soil	Explosives	Low	SW-846 8330/SOP# GC016/OP019	2 grams		Cool 4°	14 days / 40 days
Soil	Pesticides	Low	SW-846 3550B 8081A/ SOP #OP009/GC015	30 grams		Cool 4°	14 days / 40 days
Soil	Total PCB's	Low	SW-846 3550B 8082/ SOP # OP009/GC014	30 grams		Cool 4°	14 days / 40 days
Soil	TPH DRO	Low	SW-846 3550B 8015M/ SOP #OP011/GC011	30 grams		Cool 4°	14 days / 40 days
Soil	TAL Metals – ICP	Low	SW-846 3050B 6010B/ SOP #MET104/MET100	2 grams		Cool 4°	6 months
Soil	Metals – Mercury	Low	SW-846 3050B 7471A/ SOP #MET 105	2 grams		Cool 4°	28 days
Sediment	TPH GRO	Low	SW-846 5035A 8015M/ SOP # OP020/GC010	15 grams	(3) 5g EnCore Samplers	Cool 4°	48 hours to preservation/14 Days
Sediment	TCL VOC	Low	SW-846 5035A 8260B/ SOP #MS005/OP020	15 grams		Cool 4°	48 hours to preservation/14 Days
Sediment	TCL SVOC	Low	SW-846 3550B 8270C/ SOP # OP007/MS006	30 grams	(2)-8oz jar	Cool 4°	14 days / 40 days
Sediment	Explosives	Low	SW-846 8330/SOP# GC016/OP019	2 grams		Cool 4°	14 days / 40 days
Sediment	Pesticides	Low	SW-846 3550B 8081A/ SOP # OP009/GC015	30 grams		Cool 4°	14 days / 40 days
Sediment	Total PCB's	Low	SW-846 3550B 8082/ SOP # OP009/GC014	30 grams		Cool 4°	14 days / 40 days
Sediment	TPH DRO	Low	SW-846 3550B 8015M/ SOP #OP011/GC011	30 grams		Cool 4°	14 days / 40 days
Sediment	TAL Metals – ICP	Low	SW-846 3050B 6010B/ SOP # MET104/MET100	2 grams		Cool 4°	6 months
Sediment	Metals – Mercury	Low	SW-846 3050B 7471A/ SOP #MET 105	2 grams		Cool 4°	28 days

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method / SOP Reference[2]	Sample Size	Containers (number, size, and type)[1]	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Blanks / Aqueous	TPH GRO	Low	SW-846 5030B 8015M/ SOP # OP021/GC010	120 mls	(3) 40 ml VOC vials	Cool 4°, pH <2 HCL	14 days
Blanks / Aqueous	TCL VOC	Low	SW-846 5030B 8260B/ SOP #MS005/OP021	120 mls		Cool 4°, pH <2 HCL	14 days
Blanks / Aqueous	TCL SVOC	Low	SW-846 3510C 8270C/ SOP # OP006/MS006	2000 mls	(2)-1 liter amber jar	Cool 4°	7 days / 40 days
Blanks / Aqueous	Explosives	Low	SW-846 8330/SOP# GC016/OP018	2000 mls	(2)-1 liter amber jar	Cool 4°	7 days / 40 days
Blanks / Aqueous	Pesticides	Low	SW-846 3510C 8081A/ SOP # OP008/GC015	2000 mls	(2)-1 liter amber jar	Cool 4°	7 days / 40 days
Blanks / Aqueous	Total PCB's	Low	SW-846 3510C 8082/ SOP # OP008/GC014	2000 mls	(2)-1 liter amber jar	Cool 4°	7 days / 40 days
Blanks / Aqueous	TPH DRO	Low	SW-846 3510C 8015M/ SOP #OP010/GC011	2000 mls	(2)-1 liter amber jar	Cool 4°, pH <2	7 days / 40 days
Blanks / Aqueous	TAL Metals – ICP	Low	SW-846 3010A 6010B/ SOP #MET 103/MET100	1000 mls	(1)-1 liter nalgene bottle	1:1, pH <2 HNO_3,	6 months
Blanks / Aqueous	Metals – Mercury	Low	SW-846 7470A/ SOP #MET106				28 days
Disposal Soil and Sediments	TCLP Volatiles	Low	SW-846 1311 5030B 8260B/ SOP #OP041/OP021/MS005	30 grams	(1)-4 oz glass jar (No Headspace)	Cool 4°	14 days to leaching/14 Days
Disposal Soil and Sediments	TCLP Semivolatiles	Low	SW-846 1311 3510C 8270C/ SOP #OP040/OP006/MS006	250 grams	(3)-8oz jar	Cool 4°	14 days to leaching 7 days / 40 days
Disposal Soil and Sediments	TCLP Pesticide	Low	SW-846 1311 3510C 8081A/ SOP # OP040/OP008/GC015			Cool 4°	14 days to leaching 7 days / 40 days
Disposal Soil and Sediments	TCLP Herbicides	Low	SW-846 1311 8151A/ SOP # OP040,[4]			Cool 4°	14 days to leaching 7 days / 40 days
Disposal Soil and Sediments	TCLP Metals ICP	Low	SW-846 1311 3010A 6010B/ SOP# OP040/MET 103/MET100			Cool 4°	180 Days
Disposal Soil and Sediments	TCLP Metals Hg	Low	SW-846 1311 7470A/ SOP # OP040/MET106			Cool 4°	28 days
Disposal Soil and Sediments	Cyanide, Total	Low	SW-846 9014/ SOP #GN113/GN115	15 grams		Cool 4°	14 Days
Disposal Soil and Sediments	Sulfide, Total	Low	EPA 376.1M/ SOP #GN140	15 grams		Cool 4°	14 Days
Disposal Soil and Sediments	Ignitability (Flashpoint)	Low	SW-846 1010/ SOP #GN121	5 grams		Cool 4°	14 Days
Disposal Soil and Sediments	Corrosivity as pH	Low	SW-846 CHAP7/9045/ SOP #GN179	5 grams		Cool 4°	Immediate
Disposal Soil and Sediments	Total PCB's	Low	SW-846 3550B 8082/ SOP # OP009/GC014	30 grams		Cool 4°	14 days / 40 days
Disposal Soil and Sediments	TPH DRO	Low	SW-846 3550B 8015M/ SOP # OP011/GC011	30 grams		Cool 4°	14 days / 40 days
Disposal Soil and Sediments	TPH GRO[3]	Low	SW-846 5035A 8015M/ SOP #OP020/GC010	15 grams	(3) 5g EnCore Samplers	Cool 4°	48 hours to preservation/14 Days

[1]Sample size is a minimum; the containers listed will be filled to compensate for any required re-analysis or re-extractions. For samples requiring Matrix Spike(MS)/Matrix Spike Duplicate(MSD) containers listed should be tripled.

[2]Laboratory Standard Operating Procedures are subject to revision and updates during duration of the project, lab will use the most current revision of the SOP at the time of analysis.

[3] VOC samples must be accompanied by 4-oz jar to determine %Solids.

[4]Analysis performed by Accutest Laboratories Gulf Coast, Inc., Houston, TX

QAPP Worksheet #20 (UFP-QAPP Manual Section 3.1.1) -- Field Quality Control Sample Summary Table

Summarize by matrix, analytical group, and concentration level the number of field QC samples that will be collected and sent to the laboratory.

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference	No. of Sampling Locations[7]	No. of Field Duplicate Pairs[6]	No. of MS[4]	No. of Field Blanks[5]	No. of Equip. Blanks[5]	No. of PT Samples	Total No. of Samples to Lab
Soil	TPH GRO	Low	SW-846 5035A 8015M/ SOP # OP020/GC010	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Soil	TCL VOC	Low	SW-846 5035A 8260B/ SOP #MS005/OP020	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Soil	TCL SVOC	Low	SW-846 3550B 8270C/ SOP # OP007/MS006	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Soil	Explosives	Low	SW-846 8330/SOP# GC016/OP019	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Soil	Pesticides	Low	SW-846 3550B 8081A/ SOP #OP009/GC015	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Soil	Total PCB's	Low	SW-846 3550B 8082/ SOP # OP009/GC014	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Soil	TPH DRO	Low	SW-846 3550B 8015M/ SOP #OP011/GC011	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Soil	TAL Metals – ICP	Low	SW-846 3050B 6010B/ SOP #MET104/MET100	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Soil	Metals – Mercury	Low	SW-846 3050B 7471A/ SOP #MET 105	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Sediment	TPH GRO	Low	SW-846 5035A 8015M/ SOP # OP020/GC010	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Sediment	TCL VOC	Low	SW-846 5035A 8260B/ SOP #MS005/OP020	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Sediment	TCL SVOC	Low	SW-846 3550B 8270C/ SOP # OP007/MS006	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Sediment	Explosives	Low	SW-846 8330/SOP# GC016/OP019	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Sediment	Pesticides	Low	SW-846 3550B 8081A/ SOP # OP009/GC015	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Sediment	Total PCB's	Low	SW-846 3550B 8082/ SOP # OP009/GC014	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Sediment	TPH DRO	Low	SW-846 3550B 8015M/ SOP #OP011/GC011	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Sediment	TAL Metals – ICP	Low	SW-846 3050B 6010B/ SOP # MET104/MET100	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Sediment	Metals – Mercury	Low	SW-846 3050B 7471A/ SOP #MET 105	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Laboratory Prepared Trip Blank [8]	TCL VOC	Low	SW-846 5030B 8260B/ SOP #MS005/OP021	NA	NA	NA	NA	NA	NA	NA
Laboratory Prepared Trip Blank [8]	TPH GRO	Low	SW-846 5030B 8015M/ SOP # OP021/GC010	NA	NA	NA	NA	NA	NA	NA
Water	TPH GRO	Low	SW-846 5030B 8015M/ SOP # OP021/GC010	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Water	TCL VOC	Low	SW-846 5030B 8260B/ SOP #MS005/OP021	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference	No. of Sampling Locations[7]	No. of Field Duplicate Pairs[6]	No. of MS[4]	No. of Field Blanks[5]	No. of Equip. Blanks[5]	No. of PT Samples	Total No. of Samples to Lab
Water	TCL SVOC	Low	SW-846 3510C 8270C/ SOP # OP006/MS006	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Water	Explosives	Low	SW-846 8330/SOP# GC016/OP018	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Water	Pesticides	Low	SW-846 3510C 8081A/ SOP # OP008/GC015	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Water	Total PCB's	Low	SW-846 3510C 8082/ SOP # OP008/GC014	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Water	TPH DRO	Low	SW-846 3510C 8015M/ SOP #OP010/GC011	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Water	TAL Metals – ICP	Low	SW-846 3010A 6010B/ SOP #MET 103/MET100	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Water	Metals – Mercury	Low	SW-846 7470A/ SOP #MET106	TBD	1 per 10	1 per 20	1 per 20	1 per 20	0 [1]	TBD
Disposal Soil and Sediments	TCLP Volatiles	Low	SW-846 1311 5030B 8260B/ SOP #OP041/OP021/MS005	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	TCLP Semivolatiles	Low	SW-846 1311 3510C 8270C/ SOP #OP040/OP006/MS006	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	TCLP Pesticide	Low	SW-846 1311 3510C 8081A/ SOP # OP040/OP008/GC015	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	TCLP Herbicides	Low	SW-846 1311 8151A/ SOP #TGN048/TGC011[4]	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	TCLP Metals ICP	Low	SW-846 1311 3010A 6010B/ SOP# OP040/MET 103/MET100	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	TCLP Metals Hg	Low	SW-846 1311 7470A/ SOP # OP040/MET106	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	Cyanide, Total	Low	SW-846 9014/ SOP #GN113/GN115	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	Sulfide, Total	Low	EPA 376.1M/ SOP #GN140	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	Ignitability (Flashpoint)	Low	SW-846 1010/ SOP #GN121	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	Corrosivity as pH	Low	SW-846 CHAP7/9045/ SOP #GN179	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	Total PCB's	Low	SW-846 3550B 8082/ SOP # OP009/GC014	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	TPH DRO	Low	SW-846 3550B 8015M/ SOP # OP011/GC011	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD
Disposal Soil and Sediments	TPH GRO[3]	Low	SW-846 5035A 8015M/ SOP #OP020/GC010	TBD	0 [3]	0 [4]	0 [2]	0 [2]	0 [1]	TBD

[1]Accutest at a minimum participates in two single blind studies per year for every parameter in aqueous and solid matrices for each field of testing for which the laboratory maintains accreditation. Data from these studies are reported to accrediting agencies. These studies are available upon request and meet the requirements for this project; no additional PT samples are required.

[2] Field blanks and Equipment blanks are not required for disposal characterization or fill placement samples.

[3] Duplicate samples are not required for disposal characterization or fill samples since data will be used to characterize waste for disposal or fill for placement.

[4] Matrix Spike/Matrix Spike Duplicate samples are not required for disposal characterization or fill samples since data will be used to characterize waste for disposal or fill for placement. However, the analytical methods and laboratory SOPS require that a Matrix Spike (MS)/Matrix Spike Duplicates (MSD) be analyzed one per twenty samples or one for each analytical batch. Additional MS/MSD samples may be analyzed per laboratory analytical batches. For some wet chemistry methods (Ignitability and Corrosivity) MS/MSD samples are not required the method and laboratory SOPs have additional QC requirements (performing samples in duplicate).

[5]Field and Equipment blank samples are collected at a frequency of five percent (1 per 20) of the total number of samples collected or per sampling event (i.e. one per day of sampling), whichever is greater per sampling technique. Field blanks and Equipment blanks are not required for disposal characterization or fill placement samples.

[6]1 per 10 analytical samples.

[7] Additional confirmatory sampling may be required after evaluation of initial sampling data is received and compared to the site specific project action limits.

[8]Trip Blanks will be included in each cooler used for shipping volatile samples. If more than one cooler will be sent on a given day, each cooler should contain a set of Trip Blank samples. To minimize the number of Trip Blanks being analyzed, care should be taken to place all volatile samples in one cooler.

QAPP Worksheet #21
Project Sampling SOP References Table

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
1	Chain of Custody Documentation – Paper, 09/08/2006, SOP EI-FS003[1]	Shaw Environmental and Infrastructure, Inc.	N/A	N	Provides requirements for the completion of Chain of Custody documentation
2	Custody Seals, 09/08/2006, SOP EI-FS005[1]	Shaw Environmental and Infrastructure, Inc..	N/A	N	Includes procedure for completion and attachment of custody seals on environmental samples and shipping containers
3	Sample Labeling, 09/08/2006, SOP EI-FS006[1]	Shaw Environmental and Infrastructure, Inc.	N/A	N	Provides requirements for completion and attachment of sample labels on environmental sample containers
4	Sample Homogenization, 09/08/2006, SOP EI-FS010[1]	Shaw Environmental and Infrastructure, Inc.	N/A	N	Establishes method for homogenizing soil, sediment, and other solid/semi-solid matrices so that a uniform matrix is available for sampling
5	Shipping and Packaging of Non Hazardous Samples, 09/08/2006, SOP EI-FS012[1]	Shaw Environmental and Infrastructure, Inc.	Shipping Container	N	Includes sample packaging, shipping, and requirements for Non Hazardous Samples

[1] The Field Sampling SOPs can be found in Appendix E of the Field Sampling and Analysis Plan (SAP)

6	Packaging and Shipping of DOT –Hazardous Samples, 09/01/2006, SOP EI-FS013[1]	Shaw Environmental and Infrastructure, Inc.	Shipping Container	N	Includes sample packaging, shipping, and requirements for Hazardous Samples
7	Decontamination of Contact Sampling Equipment, 09/08/2006, SOP EI-FS014[1]	Shaw Environmental and Infrastructure, Inc.	N/A	N	Standard to be implemented for decontamination of contact sampling equipment
8	Trowel/Spoon Surface Soil Sampling, 09/11/2006, SOP EI-FS101[1]	Shaw Environmental and Infrastructure, Inc.	Trowel/Spoon	N	Methods/procedures for sampling of surface soils using trowels/spoons
9	Jar Headspace Screening, 9/18/03, SOP T FS-203[1]	Shaw Environmental and Infrastructure, Inc.	N/A	N	Basic methods/guidance for Volatile Organic Compound screening of environmental samples using jar headspace technique
10	Surface Water-Pond, Lagoon, Stream, 09/21/2006, SOP EI-FS113[1]	Shaw Environmental and Infrastructure, Inc.	Pond Sampler	N	Methods/procedures for sampling of surface waters with a pond sampler.
11	Sampling for VOCs in soil – Sealed-Cap (EnCore®) Sampler, 06/05/2003, SOP T-FS-104[1]	Shaw Environmental and Infrastructure, Inc.	Encore Sampler	N	Provide information about the procedure for using EnCore® samplers for collecting soil samples for volatiles.
12	Sediment Sampling using a Core Sampler, 09/11/06, SOP EI-FS103[1]	Shaw Environmental and Infrastructure, Inc.	Sediment/Gravity corer with extension handles; Brass or Plastic Sleeves	N	Methods/procedures for collecting sediment samples using a sediment/gravity corer.

[1] The Field Sampling SOPs can be found in Appendix E of the Field Sampling and Analysis Plan (SAP)

QAPP Worksheet #22 (UFP-QAPP Manual Section 3.1.2.4) – Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures

Describes the methods, means, and procedures for calibration, maintenance, testing an inspection for equipment being utilized in the field.

Photoionization Detector (PID)

Calibration Methods

The PID is designed for trace gas analysis in ambient air and is calibrated at HNu with certified standards of benzene, vinyl chloride, and isobutylene. Other optional calibrations are available (e.g., ammonia ethylene oxide, H2S, ect.)

FSSI/Shaw will use a PID with a 10.6 eV lamp. This lamp has been determined to be the most responsive to the contaminants onsite. Optional probes containing lamps of 9.5 and 11.7 eV is interchangeable in use within individual read-out assemblies for different applications.

The approximate span settings for the probe that would give different readings of the amounts of trace gas of a particular species in a sample are based upon the relative photoionization sensitivities of various gases twice daily (beginning and end of shift).

Preventative Maintenance

Maintenance consists of changing pre-filters, cleaning the lamp and ion chamber, and replacement of the lamp or other component parts or subassemblies.

QAPP Worksheet #23 (UFP-QAPP Manual Section 3.2.1) -- Analytical SOP References Table

List all SOPs that will be used to perform on-site or off-site analysis. Indicate whether the procedure produces screening or definitive data. Sequentially number analytical SOP references in the Reference Number column. Include copies of the SOPs as attachments or reference in the QAPP. The reference number can be used throughout the QAPP to refer to a specific SOP.

Reference Number	Title, Revision Date, and / or Number [1]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
OP020	SOP for Sample Introduction via SW846-5035, March 2006	Definitive	Prep Method Volatiles SW-846 5035A	OI 4560/4552 Archon	Accutest Laboratories Southeast, Inc., Orlando, FL	No
GC010	Analysis Of Gasoline Range Organics By Gas Chromatography Using Flame Ionization Detector, March 2006	Definitive	TPH GRO SW-846 8015M	HP6890, HP5890, PID/FID	Accutest Laboratories Southeast, Inc., Orlando, FL	No
MS005	Analysis of Volatile Organics by EPA Method 8260B, March 2006	Definitive	Volatiles SW-846 8260B	HP5890/5970, HP5890/5973, HP6890/5975	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP007	Standard Operating Procedure For The Extraction Of Base-Neutral And Acid (BNAS) Extractables From Soil Samples, March 2006	Definitive	Prep method for Semivolatiles SW-846 3550B	Sonic Dismembrator 550	Accutest Laboratories Southeast, Inc., Orlando, FL	No
MS006	Analysis of Semivolatile Organics by method 8270, March 2006	Definitive	Semivolatiles SW-846 8270C	HP6890/5973, HP6890/5975	Accutest Laboratories Southeast, Inc., Orlando, FL	No
GC016	Analysis Of Nitroaromatics And Nitramines By HPLC, March 2006	Definitive	Explosives, SW-846 8330	HP1100	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP019	Standard Operating Procedure For The Extraction Of Nitroaromatics And Nitramines (Explosives) From Solid Samples For HPLC Analysis	Definitive	Explosives, SW-846 8330	Sonic Dismembrator 550	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP018	Standard Operating Procedure For The Extraction Of Nitroaromatics And Nitramines (Explosives) From Water Samples For HPLC Analysis	Definitive	Explosives, SW-846 8330	Solid Phase Manifold	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP009	SOP for the Extraction of Pesticides/PCBs from Solid Samples, March 2006	Definitive	Prep Method PCBs, Pesticides, PCBs SW-846 3550B	Sonic Dismembrator 550	Accutest Laboratories Southeast, Inc., Orlando, FL	No
GC015	Analysis Of Organochlorine Pesticides By Gas Chromatography, Electron Capture Detector	Definitive	Pesticides SW-846 8081A	HP5890, HP6890, Dual ECD	Accutest Laboratories Southeast, Inc., Orlando, FL	No

Reference Number	Title, Revision Date, and / or Number [1]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	Sw-846 8081, March 2006					
GC014	Analysis Of Polychlorinated Biphenyls By Gas Chromatography, Electron Capture Detector Sw-846 8082, March 2006	Definitive	Total PCB's SW-846 8082	HP5890, HP6890, Dual ECD	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP011	SOP for the Extraction of Diesel Range Organics (DRO) from Solid Samples, March 2006	Definitive	Prep Method DRO SW-846 3550B	Sonic Dismembrator 550	Accutest Laboratories Southeast, Inc., Orlando, FL	No
GC011	Analysis Of Diesel Range Organics By Gas Chromatography Using Flame Ionization Detector, March 2006	Definitive	TPH DRO SW-846 8015	HP6890/7683, HP5890/7673, Dual FID	Accutest Laboratories Southeast, Inc., Orlando, FL	No
MET104	Digestion of Soils for ICP Analysis, April 2006	Definitive	Prep Method Metals – ICP SW-846 3050B	SCP Science	Accutest Laboratories Southeast, Inc., Orlando, FL	No
MET100	Metals by ICP, April 2006	Definitive	Metals – ICP 6010B	TJA Trace	Accutest Laboratories Southeast, Inc., Orlando, FL	No
MET105	CVAA Analysis of Hg in Soil, April 2006	Definitive	Metals – Mercury 7471A	Leeman HydraAA/ Environmental Express digestion block	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP021	SOP for Sample Introduction via SW846-5035, March 2006	Definitive	Prep Method Volatiles SW-846 5030B	OI 4560/4552 Archon	Accutest Laboratories Southeast, Inc., Orlando, FL	No
GC010	Analysis Of Gasoline Range Organics By Gas Chromatography Using Flame Ionization Detector, March 2006	Definitive	SW-846 8015	HP6890/7683, HP5890/7673, Dual FID	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP006	Standard Operating Procedure For The Extraction Of Base-Neutral And Acid (BNAS) Extractables From Water Samples, March 2006	Definitive	Prep method for Semivolatiles SW-846 3510C	Glas-Col 3-D SepFunnel Shaker	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP008	SOP for the Extraction of Pesticides/PCBs from Aqueous Samples, March 2006	Definitive	Prep Method PCBs, Pesticides, PCBs SW-846 3510C	Glas-Col 3-D SepFunnel Shaker	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP010	Standard Operating Procedure For The Extraction Of Diesel Range Organics (DRO) From Water Samples	Definitive	SW-846 3510C	Glas-Col 3-D SepFunnel Shaker	Accutest Laboratories Southeast, Inc., Orlando, FL	No
MET103	Digestion of Waters for ICP Analysis, April 2006	Definitive	Prep Method Metals – ICP SW-846 3010A	SCP Science	Accutest Laboratories Southeast, Inc., Orlando, FL	No

Reference Number	Title, Revision Date, and / or Number [1]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
MET106	CVAA Analysis of Hg in Water, April 2006	Definitive	Metals – Mercury 7470A	Leeman HydraAA/ Environmental Express digestion block	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP041	Standard Operating Procedure For The Toxicity Characteristic Leaching Of Volatile Organics (TCLP), February 2006	Definitive	TCLP Volatiles ZHE SW-846 1311	Millipore Tumbler	Accutest Laboratories Southeast, Inc., Orlando, FL	No
OP040	Standard Operating Procedure For The Toxicity Characteristic Leaching Of Semivolatile Organics And Metals (TCLP), February 2006	Definitive	TCLP Procedure Herbicides, Pesticides, Semivolatiles, Metals SW-846 1311	Tumbler	Accutest Laboratories Southeast, Inc., Orlando, FL	No
TGC011	Herbicide analysis by Method 8151 Dual ECD; May 2004	Definitive	Herbicides SW-846 8151A	HP6890/7673	Accutest Laboratories Gulf Coast, Houston, TX	No
GN113 - GN115	Cyanide Distillation/Aqueous And Solid Samples; Cyanide, Total, June 2006	Definitive	Cyanide, Total SW-846 9014	Lachat QuickChem 8500	Accutest Laboratories Southeast, Inc., Orlando, FL	No
GN140	Sulfide, March 2004	Definitive	Sulfide, Total EPA 376.1M	burette	Accutest Laboratories Southeast, Inc., Orlando, FL	No
GN121	Ignitability, March 2005	Definitive	Ignitability (Flashpoint) SW-846 1010	Pensky-Marten	Accutest Laboratories Southeast, Inc., Orlando, FL	No
GN179	Corrosivity in soil as pH, July 2004	Definitive	Corrosivity as pH SW-846 9045C	pH meter(Fisher)	Accutest Laboratories Southeast, Inc., Orlando, FL	No

[1] Laboratory Standard Operating Procedures are subject to revision and updates during duration of the project, lab will use the most current revision of the SOP at the time of analysis.

QAPP Worksheet #24 (UFP-QAPP Manual Section 3.2.2) -- Analytical Instrument Calibration Table

Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
HP5890, HP6890, Dual ECD	SW-846 8081A	As needed See Worksheets 28.5 and 28.12 for Details	%RSD <20%, or Correlation coefficient R≥0.995	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	GC015
HP5890, HP6890, Dual ECD	SW-846 8082	As needed See Worksheet 28.6 for Details	%RSD <20%, or Correlation coefficient R≥0.995	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	GC014
HP1100	SW-846 8330	As needed See Worksheet 28.4 for Details	%RSD <20%, or Correlation coefficient R≥0.995	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	GC016
HP6890/5973, HP6890/5975	SW-846 8270C	As needed See Worksheets 28.3 and 28.11 for Details	%RSD <15%, Correlation coefficient R≥0.995	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	MS006, 008
HP5890/5970, HP5890/5973, HP6890/5975	SW-846 8260B	As needed See Worksheets 28.2 and 28.10 for Details	%RSD <15%, Correlation coefficient R≥0.995	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	MS005
HP5890/7673	SW-846 8151A	As needed See Worksheet 28.13 for Details	%RSD <20%, or Correlation coefficient R≥0.995	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	TGC011
HP6890/7683, HP5890/7673, Dual FID	DRO (SW-846 8015M)	As needed See Worksheet 28.7 for Details	%RSD <20%, or Correlation coefficient R≥0.995	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	GC011
HP6890, HP5890, PID/FID	GRO (SW-846 8015M)	As needed See Worksheet 28.1 for Details	%RSD <20%, or Correlation coefficient R≥0.995	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	GC010
Lachat QuickChem 8500	SW-846 9014	As needed See Worksheet 28.16 for Details	Correlation coefficient R≥0.995; intercept <RL	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	GN115
Leeman HydraAA/	SW-846 7470A/7471A	As needed See Worksheet 28.9 and 28.15 for Details	Correlation coefficient R≥0.995	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	MET105, 106
TJA Trace	SW-846 6010B	As needed See Worksheet 28.8 and 28.14 for Details	%RSD <5%, or Correlation coefficient R≥0.995	Instrument maintenance, standard inspection, recalibration	Laboratory Analyst	MET100

[1]Laboratory Standard Operating Procedures are subject to revision and updates during duration of the project, lab will use the most current revision of the SOP at the time of analysis.

QAPP Worksheet #25 (UFP-QAPP Manual Section 3.2.3) -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Identify all analytical instrumentation that requires maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
HP5890, HP6890, Dual ECD	Injector port, column maintenance	SW-846 8081A	Leak test, column and injector port inspection	Need for maintenance determined by passing calibration and DDT/Endrin breakdown – see GC015	Passing DDT and Endrin breakdown; Passing CCV	Column clipping, seals and liners replacement, recalibrate and reanalyze affected samples	Laboratory Analyst	GC015
HP5890, HP6890, Dual ECD	Injector port, column maintenance	SW-846 8082	Leak test, column and injector port inspection	Need for maintenance determined by passing calibration– see GC014	Passing CCV	Column clipping, seals and liners replacement, recalibrate and reanalyze affected samples	Laboratory Analyst	GC014
HP6890/5973, HP6890/5975	Injector port, column maintenance, source cleaning	SW-846 8270C, 8270CSIM	Leak test, column and injector port inspection, source insulator integrity	Need for maintenance determined by passing calibration and DFTPP/DDT/PCP/ Benzidine breakdown – see MS006, 008	Passing DFTPP etc. and CCV, passing Internal Standard response	Column clipping and/or reconditioning, seal and liners replacement, filaments and insulators as needed	Laboratory Analyst	MS006, 008
HP5890/5970, HP5890/5973, HP6890/5975	Injector port, column maintenance, source cleaning	SW-846 8260B	Leak test, column and injector port inspection, source insulator integrity	Need for maintenance determined by passing calibration and BFB – see MS005	Passing BFB and CCV, passing Internal Standard response	Column clipping and/or reconditioning, seal and liners replacement, filaments and insulators as needed	Laboratory Analyst	MS005

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
HP5890/7673	Injector port, column maintenance	SW-846 8151A	Leak test, column and injector port inspection	Frequency determined by instrument remaining in calibration and free of interference – See TGC011	Passing calibration.	Column clipping, seals and liners replacement, recalibrate and reanalyze affected samples	Laboratory Analyst	TGC011
HP6890/7683, HP5890/7673, Dual FID	Injector port, column maintenance	DRO (SW-846 8015M)	Leak test, column and injector port inspection	Frequency determined by instrument remaining in calibration and free of interference – GC011	Passing calibration	Column clipping, seals and liners replacement, recalibrate and reanalyze affected samples	Laboratory Analyst	GC011
HP1100	Lamp and guard column inspection. Pump maintenance.	SW-846 8330	Leak and pressure test, guard column and lamp performance	Frequency determined by instrument remaining in calibration and free of interference – GC016	Passing calibration	Replace lamp, replace guard column, tighten fittings, recalibrate, reanalyze	Laboratory Analyst	GC016
HP6890, HP5890, PID/FID	Injector port, column maintenance	GRO (SW-846 8015M)	Leak test, column and injector port inspection	Frequency determined by instrument remaining in calibration and free of interference – GC010	Passing calibration	Column clipping, seals and liners replacement, recalibrate and reanalyze affected samples	Laboratory Analyst	GC010
Lachat QuickChem 8500	Pump maintenance, photocell cleaning	SW-846 9014	Clean or replace tubing, check connections	Frequency determined by instrument remaining in calibration and free of interference – GN115	Passing calibration	Reconnect tubes, check pump rate. Rerun calibration and samples	Laboratory Analyst	GN115

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Leeman HydraAA/	Pump tubing, absorption cell, and lens cleaning.	SW-846 7470A/7471 A	Check connections, flush sample lines	Frequency determined by instrument remaining in calibration and free of interference – Met 105,106	Passing calibration	Reconnect sample pathways, recalibrate, reanalyze affected samples	Laboratory Analyst	MET105, 106
TJA Trace	Torch, nebulizer, spray chamber, autosampler, pump tubing maintenance,	SW-846 6010B	Check connections, flush lines, clean nebulizer	Frequency determined by instrument remaining in calibration and free of interference – Met 100	Passing calibration	Reconnect sample pathways, recalibrate, reanalyze affected samples	Laboratory Analyst	MET100

[1] Laboratory Standard Operating Procedures are subject to revision and updates during duration of the project, lab will use the most current revision of the SOP at the time of analysis.

QAPP Worksheet #26
Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): TBD/Shaw Environmental, Inc.
Sample Packaging (Personnel/Organization): TBD/Shaw Environmental, Inc.
Coordination of Shipment (Personnel/Organization): TBD/Shaw Environmental, Inc.
Type of Shipment/Carrier: Federal Express – Priority Overnight
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Aaron Ben David, Sample Management Supervisor, Accutest Laboratories Southeast, Inc.
Sample Custody and Storage (Personnel/Organization): Sample Management Technicians with oversight from Aaron Ben David, Sample Management Supervisor, Accutest Laboratories Southeast, Inc.
Sample Preparation (Personnel/Organization): Sample Prep Technicians with oversight from Rick Watkins, Organics Supervisor, Accutest Laboratories Southeast, Inc.
Sample Determinative Analysis (Personnel/Organization): Natasha Sullivan, Shaw Lantdiv Chemist and Heather Wandrey, Project Manager, Accutest Laboratories Southeast, Inc.
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): minimum 30 days after final report sent to the client.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): minimum 30 days after final report sent to the client
Biological Sample Storage (No. of days from sample collection): Not Required, No biological samples.
SAMPLE DISPOSAL
Personnel/Organization: Sample Management Technicians, with oversight from Aaron Ben David, Sample Management Supervisor, Accutest Laboratories Southeast, Inc.
Number of Days from Analysis: minimum 30 days after final report sent to the client.

QAPP Worksheet #27 (UFP-QAPP Manual Section 3.3.3) – Sample Custody Requirements Table

Describe the procedures that will be used to maintain sample custody and integrity. Include examples of chain-of-custody forms, traffic reports, sample identification, custody seals, laboratory sample receipt forms, and laboratory sample transfer forms. Attach or reference applicable SOPs.

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):

Sample custody can be defined as physical possession of samples, having samples within visual range, or having samples located in a restricted access area. Sample possession during all sampling efforts must be traceable from the time of collection until the results are verified and reported. The sample custody procedures provide a mechanism for documentation of all information related to sample collection and handling. The primary piece of documentation to ensure sample custody is the Chain of Custody Form (COC). Shaw personnel are responsible for providing evidence of sample custody from the time of collection until the laboratory receives the samples. The laboratory will be able to provide documentation of sample custody from that point to sample disposal.

As part of appropriate documentation, all sample bottles will be adequately labeled. The label will present sample identification and collection information. It will be pre-printed from the sample tracking system or completed with indelible ink. At a minimum, all sample labels will include the following sample information:

- Field sample location and unique sample identifier
- Project name and number
- Analysis requested for each bottle
- Method of preservation for each bottle
- Date and time of collection
- Initials of sample technician.

Transfer of custody and shipping procedures will include:

- The Site Supervisor instructing sampling team personnel in the proper COC procedures before sampling begins,
- A COC entry made in the field for each sample. This document will accompany the samples in shipment, and a copy will be maintained at the site for placement in the project files at the conclusion of field activities. The custody of individual sample containers will be documented by recording each sample identification and the number of bottles on the appropriate COC form.
- COC records initiated in the field will be placed in a plastic bag and taped to the underside of the top of the shipping cooler used for sample transport,
- Each time responsibility for custody of the sample changes, the new custodian will sign and date the record,
- All coolers must be secured at the site with two custody seals prior to transport. Custody seals should be signed and dated by the person relinquishing custody of the samples being shipped. They should be placed over the opening of each cooler so that the cooler cannot be opened without breaking the seal.

Sample packaging and shipment
Samples that are collected for off-site laboratory analysis that require overnight shipment will be generally prepared by:

- Sealing the container in an outer ziplock storage bag,
- Securely wrapping and taping each collected bottle in bubble wrap (or other similar shock-adsorbing material).
- A temperature blank will be included in each cooler. The temperature will be recorded upon receipt at the laboratory to verify sample temperatures during transport.
- When collecting volatile samples, including GRO samples, the field personnel will include trip blanks in each of the coolers containing volatile samples.
- At least three sides of the container must be wrapped or surrounded with material when placing the samples into the shipping cooler. Adequate ice will be placed in doubled ziplock bags and added to the cooler around and over the top of the sample containers to form a cooling layer to help ensure proper preservation during shipment.
- Samples should be pre-cooled to the desired temperature prior to packing for shipment.
- Trip blanks are volatile bottles that have been prepped by the laboratory using laboratory grade water and are shipped with the field sample containers, and returned unopened to the laboratory for volatile analysis. Trip blanks are used to indicate if any contamination during shipment of containers to the field, storage of containers at the site, or shipment from the site to the laboratory has occurred. Trip Blanks will be included in each cooler used for shipping volatile samples. If more than one cooler will be sent on a given day; each cooler should contain a set of Trip Blank samples. To minimize the number of Trip Blanks being analyzed, care should be taken to place all volatile samples in one cooler.
- Temperature blanks are nalgene bottles containing water that will be included in each sample cooler.
- Completed and signed COCs will be placed into the cooler in a protective ziplock bag and taped to the underside of the cooler lid. A minimum of 2 custody seals will be applied across the opening of the cooler and the lid secured by wrapping the cooler with clear plastic packing tape.

- The cooler will then be ready for shipment according to the methods required by the overnight delivery service. At a minimum, the laboratory address, telephone number, and contact name should be included on the original air bill and, if multiple packages are sent, on each sample cooler.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):

All samples to be analyzed by the fixed-base laboratory will be shipped via overnight courier service. Upon receipt, per the attached Laboratory Standard Operating Procedure (SOP), a representative of the laboratory shall check the integrity of the custody seals, then locate, sign, and date the COC. The temperature will be recorded to verify the sample temperatures during transport. The laboratory is responsible for verifying that the COC and containers are in agreement. The COC, a Cooler Receipt Form, and information regarding any discrepancies between the COC and bottle labels will be faxed to the Project Chemist prior to preparation for analysis. The Laboratory Information Management System will provide evidence of sample custody from receipt by the laboratory until appropriate disposal see **Worksheet 26** and laboratory SOPs.

Sample Identification Procedures:

A sample numbering system will be utilized in the field to uniquely identify each sample collected at Vieques, Puerto Rico, sites SWMU-6, SWMU-7, AOC J, and AOC-R. The sample number will be traceable to the site, SWMU/AOC, and location. The sample identification and description will be recorded by the Site Supervisor or representative in the sample collection logs.
The sites shall be designated by the X character as follows: SWMU 6 – 1, SWMU 7 – 2, AOC J – 3, AOC R – 4.

Sampling Nomenclature as follows:

Confirmation Soil/Sediment Samples:	PR-116701-0X-CON-00Y
Confirmation Surface Water Samples:	PR-116701-0X-CSW-00Y
Equipment Blank Samples:	PR-116701-0X-EBLANK-00Y
Field Blank Samples:	PR-116701-0X-FBLANK-00Y
Trip Blank Samples:	PR-116701-0X-TBLANK-00Y
Waste Characterization Soil Samples:	PR-116701-0X-WC-00Y

Chain-of-Custody Procedures:

- Project Name will be identified on the COC Form.
- Project Number will be identified on the COC Form.
- Shaw contact information will be listed on the COC Form, this information should include Shaw Project Manager, Shaw Lantdiv Chemist, Shaw address, telephone numbers and facsimile number.
- For analysis and required analytical methods that should be listed on the COC form refer to **Worksheet 19**.
- Required turnaround time and report format will be listed on the COC form.
- Each sample should be listed on the COC form using the sampling nomenclature listed above, sample description, date of sampling, time of sampling, number of containers being submitted to the laboratory.
- Sampler will sign and relinquish COC form, dates and times of relinquishment will be included.

QAPP Worksheet #28.1(UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid, Aqueous
Analytical Group	GRO Petroleum
Concentration Level	Low
Sampling SOP	SOP T FS-104 SOP EI-FS-113
Analytical Method / SOP Reference	SW-846 8015/ LAB SOP#GC010
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous
Trip Blank	1 per cooler	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration. Flagging criteria are not appropriate.	Analyst	Laboratory Precision	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Retention time window position establishment and verification for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift for position establishment. Each calibration verification standard for retention time verification.	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift. Analyte shall be within established window for each calibration verification. For method 8015, check state methods for use of modified retention time markers with gasoline range organics (GRO).	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window. Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window. No samples shall be run without a verified retention time window at the initial verification.	Analyst	Laboratory Representativeness	Each analyte shall be within established window. For method 8015, check state methods for use of modified retention time markers with gasoline range organics (GRO).
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration	Value of second source for all analytes within ±20%D of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Precision	Value of second source for all analytes within ±20%D of expected value (initial source)
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	All analytes within ±20%D of expected value from the ICAL	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification. As long as the CCV is acceptable, a new initial instrument calibration is not necessary.	Analyst	Laboratory Precision	All analytes within ±20%D of expected value from the ICAL
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate.	Analyst	Laboratory Representativeness	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	One per analytical batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous	Correct problem, if required, reprep then reanalyze method blank and all samples processed with the contaminated blank. Any sample associated with a blank that fails these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be flagged. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous
Laboratory Control Sample (LCS)	One LCS per analytical batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike (MS)	One MS per analytical batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%
Matrix Spike Duplicates (MSD)	One per analytical batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100% Alternative surrogates are recommended when there are obvious chromatographic interferences.	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.1 for TPHGRO Solids Worksheet 15.9 for TPHGRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%
Results reported between LOD and LOQ	All positive results must be confirmed	Not Applicable	Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	Not Applicable

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.2 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid, Aqueous
Analytical Group	Volatiles
Concentration Level	Low
Sampling SOP	SOP T FS-104 SOP EI-FS-113
Analytical Method / SOP Reference	SW-846 8260B/ LAB SOP#MS005
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. For the common laboratory contaminants methylene chloride, acetone, and 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. For the common laboratory contaminants methylene chloride, acetone, and 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. For the common laboratory contaminants methylene chloride, acetone, and 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. For the common laboratory contaminants methylene chloride, acetone, and 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous
Trip Blank	1 per cooler	All Target Compounds <1/2 RL. For the common laboratory contaminants methylene chloride, acetone, and 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. For the common laboratory contaminants methylene chloride, acetone, and 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous
MS Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples. Flagging criteria are not appropriate and problem must be corrected. No samples may be accepted without a valid tune.	Analyst	Laboratory Accuracy	Refer to method for specific ion criteria.
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	<u>Average response factor (RF) for SPCCs</u>: VOCs - RF≥0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥0.1 for chloromethane, bromoform,and 1,1-dichloroethane. <u>RSD for RFs for CCCs</u>: VOCs - RSD≤30%and one option below; Option 1: RSD for each analyte ≤15% Option 2: linear least squares regression R ≥0.995 Option 3: non-linear regression - coefficient of determination (COD) R^2 ≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)	If the acceptance criteria were not met, correct problem and re-calibration is performed before any samples may be analyzed.	Analyst	Laboratory Precision	<u>%RSDs and correlations:</u> %RSD≤15% (≤30% for CCCs) or Correlation coefficient R≥0.995 or COD R^2≥0.99 <u>RF for SPCCs</u>: VOCs – RF≥0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥0.1 for chloromethane, bromoform,and 1,1-dichloroethane.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration and before field samples	Value of second source for all analytes within ±25% of expected value (initial source).	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Analyst	Laboratory Accuracy	Value of second source for all analytes within ± 25% of expected value (initial source).
Continuing Calibration Verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time	Average RF for SPCCs: VOCs RF≥0.30 for Chlorobenzene and 1,1,2,2- tetrachlorolethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. %Difference/Drift for CCCs: VOCs and SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration.)	Correct problem, then rerun CV. If that fails, repeat initial calibration. Corrective action may include re-analysis of samples. Apply Q-flag if no sample material remains and analyte exceeds criteria.	Analyst	Laboratory Precision	%Ds or %Drift: %D≤20%; %Drift≤20% RF for SPCCs: VOCs - RF≥0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥ 0.1 for chloromethane, bromoform,and 1,1-dichloroethane.
Method Blank (MB)	One per analytical batch per matrix	All Target Compounds <1/2 RL. For the common laboratory contaminants methylene chloride, acetone, and 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code “B”.	Analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. For the common laboratory contaminants methylene chloride, acetone, and 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per analytical batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Matrix Spike (MS)	One MS per analytical batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicates (MSD) or Sample Duplicates	One per analytical batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.2 for VOCs Solids Worksheet 15.10 for VOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal standards (IS) verification	In all field samples and standards	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory. If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Analyst	Accuracy (Instrument sensitivity control)	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard
Quantitation Verification	All field samples and standards	RRT of each target analyte in each calibration standard within ±0.06 RRT units.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate. Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	RRT of each target analyte in each calibration standard within ±0.06 RRT units.

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.3 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid, Aqueous
Analytical Group	Semivolatiles
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101 SOP EI-FS113
Analytical Method / SOP Reference	SW-846 8270C/ LAB SOP#MS006
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. For the common laboratory contaminants phthalate esters, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. For the common laboratory contaminants phthalate esters, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. For the common laboratory contaminants phthalate esters, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. For the common laboratory contaminants phthalate esters, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous
MS Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples. Flagging criteria are not appropriate and problem must be corrected. No samples may be accepted without a valid tune.	Analyst	Laboratory Accuracy	Refer to method for specific ion criteria.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	Average response factor (RF) for SPCCs: SVOCs - RF≥0.050. RSD for RFs for CCCs: SVOCs - RSD≤30%and one option below; Option 1: RSD for each analyte ≤15% Option 2: linear least squares regression R ≥0.995 Option 3: non-linear regression - coefficient of determination (COD) R^2 ≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)	If the acceptance criteria were not met, correct problem and re-calibration is performed before any samples may be analyzed.	Analyst	Laboratory Precision	%RSDs and correlations: %RSD≤15% (≤30% for CCCs) or Correlation coefficient R≥0.995 or COD R^2≥0.99 RF for SPCCs: SVOCs - RF≥0.050.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration and before field samples	Value of second source for all analytes within ±25%D of expected value (initial source).	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Analyst	Laboratory Precision	Value of second source for all analytes within ±25%D of expected value (initial source).
Continuing Calibration Verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time	Average RF for SPCCs: SVOCs RF≥0.050. %Difference/Drift for CCCs: SVOCs ≤20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration.)	Correct problem, then rerun CV. If that fails, repeat initial calibration. Corrective action may include re-analysis of samples. Apply Q-flag if no sample material remains and analyte exceeds criteria.	Analyst	Laboratory Precision	%Ds or %Drift: %D≤20%; %Drift≤20% RF for SPCCs: SVOCs - RF≥0.050.
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. For the common laboratory contaminants phthalate esters, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code "B".	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. For the common laboratory contaminants phthalate esters, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Matrix Spike (MS)	One MS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Prep analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicates (MSD) or Sample Duplicates	One per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.3 for SVOCs Solids Worksheet 15.11 for SVOCs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal standards (IS) verification	In all field samples and standards	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory. If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Analyst	Accuracy (Instrument sensitivity control)	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard
Quantitation Verification	All field samples and standards	RRT of each target analyte in each calibration standard within ±0.06 RRT units.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate. Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	RRT of each target analyte in each calibration standard within ±0.06 RRT units.

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.4 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid, Aqueous
Analytical Group	Explosives
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101 SOP EI-FS113
Analytical Method / SOP Reference	SW-846 8330/ LAB SOP#GC016
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration. Flagging criteria are not appropriate.	Analyst	Laboratory Precision	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)
Retention time window position establishment and verification for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift for position establishment. Each calibration verification standard for retention time verification.	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift. Analyte shall be within established window for each calibration verification.	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window. Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window. No samples shall be run without a verified retention time window at the initial verification.	Analyst	Laboratory Representativeness	Each analyte shall be within established window.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration	Value of second source for all analytes within ±20%D of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Precision	Value of second source for all analytes within ±20%D of expected value (initial source)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	All analytes within ±20%D of expected value from the ICAL	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification. As long as the CCV is acceptable, a new initial instrument calibration is not necessary.	Analyst	Laboratory Precision	All analytes within ±20%D of expected value from the ICAL
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate.	Analyst	Laboratory Representativeness	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code “B”.	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Matrix Spike (MS)	One MS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Prep analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicates (MSD) or Sample Duplicates	One per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.4 for explosives Solids Worksheet 15.12 for explosives Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Quantitation Verification and Confirmation	All positive results must be confirmed.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column or detector RPD ≤40%.	Apply J-flag if RPD >40% or Q-flag if sample is not confirmed. Discuss in the case narrative. Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative. Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness and Precision	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column or detector RPD ≤40%.

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.5 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid, Aqueous
Analytical Group	Pesticides
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101 SOP EI-FS113
Analytical Method / SOP Reference	SW-846 8081/ LAB SOP#GC015
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration. Flagging criteria are not appropriate.	Analyst	Laboratory Precision	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)
Retention time window position establishment and verification for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift for position establishment. Each calibration verification standard for retention time verification.	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift. Analyte shall be within established window for each calibration verification.	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window. Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window. No samples shall be run without a verified retention time window at the initial verification.	Analyst	Laboratory Representativeness	Each analyte shall be within established window.
Breakdown check (Endrin/DDT)	Daily prior to analysis of samples	Degradation ≤ 15%D for both Endrin and DDT.	Correct problems then repeat breakdown check. Flagging criteria are not appropriate. No samples shall be run until degradation ≤15%.	Analyst	Laboratory Precision	Degradation ≤ 15%D for both Endrin and DDT.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration	Value of second source for all analytes within ±20%D of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Precision	Value of second source for all analytes within ±20%D of expected value (initial source)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	All analytes within ±20%D of expected value from the ICAL	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification. As long as the CCV is acceptable, a new initial instrument calibration is not necessary.	Analyst	Laboratory Precision	All analytes within ±20%D of expected value from the ICAL
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate.	Analyst	Laboratory Representativeness	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code “B”.	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Matrix Spike (MS)	One MS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Prep analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicates (MSD) or Sample Duplicates	One per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.5 for pesticides Solids Worksheet 15.13 for pesticides Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Quantitation Verification and Confirmation	All positive results must be confirmed (exclude toxaphene and technical chlordane)	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column or detector RPD ≤40%.	Apply J-flag if RPD >40% or Q-flag if sample is not confirmed. Discuss in the case narrative. Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative. Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness and Precision	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column or detector RPD ≤40%.

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.6 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid, Aqueous
Analytical Group	PCBs
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101 SOP EI-FS113
Analytical Method / SOP Reference	SW-846 8082/ LAB SOP#GC014
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order) For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of another Aroclor (e.g., 1232). In addition, a mid-level or lower standard for each of the remaining Aroclors is analyzed for pattern recognition and response factor.	Correct problem then repeat initial calibration. Flagging criteria are not appropriate.	Analyst	Laboratory Precision	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order) For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of another Aroclor (e.g., 1232). In addition, a mid-level or lower standard for each of the remaining Aroclors is analyzed for pattern recognition and response factor.
Retention time window position establishment and verification for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift for position establishment. Each calibration verification standard for retention time verification.	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift. Analyte shall be within established window for each calibration verification.	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window. Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window. No samples shall be run without a verified retention time window at the initial verification.	Analyst	Laboratory Representativeness	Each analyte shall be within established window.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration	Value of second source for all analytes within ±20%D of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Precision	Value of second source for all analytes within ±20%D of expected value (initial source)
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	All analytes within ±20%D of expected value from the ICAL	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification. As long as the CCV is acceptable, a new initial instrument calibration is not necessary.	Analyst	Laboratory Precision	All analytes within ±20%D of expected value from the ICAL
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate.	Analyst	Laboratory Representativeness	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code “B”.	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Matrix Spike (MS)	One MS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Prep analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicates (MSD) or Sample Duplicates	One per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.6 for PCBs Solids Worksheet 15.14 for PCBs Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Quantitation Verification and Confirmation	All positive results must be confirmed	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column or detector RPD ≤40%.	Apply J-flag if RPD >40% or Q-flag if sample is not confirmed. Discuss in the case narrative. Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative. Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness and Precision	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column or detector RPD ≤40%.

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.7 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid, Aqueous
Analytical Group	DRO Petroleum
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101 SOP EI-FS113
Analytical Method / SOP Reference	SW-846 8015 DRO/ LAB SOP#GC011
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration. Flagging criteria are not appropriate.	Analyst	Laboratory Precision	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)
Retention time window position establishment and verification for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift for position establishment. Each calibration verification standard for retention time verification.	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift. Analyte shall be within established window for each calibration verification. For method 8015, check state methods for use of modified retention time markers with gasoline range organics (GRO) or diesel range organics (DRO).	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window. Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window. No samples shall be run without a verified retention time window at the initial verification.	Analyst	Laboratory Representativeness	Each analyte shall be within established window. For method 8015, check state methods for use of modified retention time markers with gasoline range organics (GRO) or diesel range organics (DRO).
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration	Value of second source for all analytes within ±20%D of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Precision	Value of second source for all analytes within ±20%Dof expected value (initial source)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	All analytes within ±20%D of expected value from the ICAL	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification. As long as the CCV is acceptable, a new initial instrument calibration is not necessary.	Analyst	Laboratory Precision	All analytes within ±20%D of expected value from the ICAL
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate.	Analyst	Laboratory Representativeness	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code “B”.	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Matrix Spike (MS)	One MS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Prep analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicates (MSD) or Sample Duplicates	One per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.7for DRO Solids Worksheet 15.15 for DRO Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%
Results reported between LOD and LOQ	All positive results must be confirmed	Not Applicable	Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	Not Applicable

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.8 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid, Aqueous
Analytical Group	ICP Metals
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101 SOP EI-FS113
Analytical Method / SOP Reference	SW-846 6010B/ LAB SOP#Met 104, Met 100, Met 103
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis.	Initial calibration for all analytes (ICAL): Minimum one high standard and a calibration blank; No acceptance criteria unless more than one standard is used, in which case r ≥0.995.	Correct problem then repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until ICAL has passed.	Analyst	Laboratory Precision	Initial calibration for all analytes (ICAL): Minimum one high standard and a calibration blank; No acceptance criteria unless more than one standard is used, in which case r ≥0.995.
Linear dynamic range or High-level calibration check standard	Every 6 months	Within ±10%R of expected value.	Not Applicable	Analyst	Laboratory Accuracy	Within ±10%R of expected value.
Low-level calibration check standard	Daily, after one-point initial calibration	Within ±20%R of expected value. Low-level calibration check standard should be less than or equal to the reporting limit.	Correct problem then repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until ICAL has passed.	Analyst	Laboratory Accuracy	Within ±20%R of expected value. Low-level calibration check standard should be less than or equal to the reporting limit.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration, prior to sample analysis.	Value of second source for all analyte(s) within ±10%R of expected value (initial source).	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Accuracy	Value of second source for all analyte(s) within ±10%R of expected value (initial source).

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	ICV and CCV within ±10% of expected value.	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful CCV. If that fails, then repeat initial calibration..	Analyst	Laboratory Accuracy	ICV and CCV within ±10% of expected value.
Calibration blanks (initial [ICB] and continuing [CCB])	ICB: Daily, before sample run. CCB: After every 10 field samples and at the end of the analysis sequence	ICB and CCB ≤2x MDL.	ICB and CCB: Correct problem, then repeat ICB and CCB and reanalyze all samples since last successful CCB. If that fails, then Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	Analyst	Laboratory Representativeness (Absence of interference/ contamination)	ICB and CCB ≤2x MDL.
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code "B".	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Matrix Spike (MS)	One MS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Prep analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicates (MSD) or Sample Duplicates	One per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)
Interference check solutions (ICS)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all nonspiked analytes <2x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS. Flagging criteria are not appropriate. No samples may be analyzed without a valid ICS.	Analyst	Accuracy	ICS-A: Absolute value of concentration for all nonspiked analytes <2x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value
Serial Dilution Test	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >50x MDL for ICP.	Perform post-digestion spike (PDS) addition. Flagging criteria are not appropriate.	Analyst	Precision (field samples)	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >50x MDL for ICP.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Post-digestion spike (PDS) addition	When dilution test fails or analyte concentration in all samples <50x MDL	Recovery within 75-125% of expected result. The spike addition should produce a level between 10x to 100x MDL.	Run samples by method of standard additions (MSA) or Apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition.	Analyst	Accuracy	Recovery within 75-125% of expected result. The spike addition should produce a level between 10x to 100x MDL.
Method of standard additions (MSA) or Internal Standard calibration	When matrix interference is suspected	Document use of MSA in the case narrative.	Not Applicable	Analyst	Accuracy	Document use of MSA in the case narrative.
Results reported between LOD and LOQ	All positive results must be confirmed	Not Applicable	Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	Not Applicable

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.9 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid, Aqueous
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101 SOP EI-FS113
Analytical Method / SOP Reference	SW-846 7470, 7471/ LAB SOP#MET105, 106
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis.	Initial calibration for all analytes (ICAL) Minimum 5 standards and a calibration blank with linear least squares regression: R≥0.995.	Correct problem then repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until ICAL has passed.	Analyst	Laboratory Precision	Initial calibration for all analytes (ICAL) Minimum 5 standards and a calibration blank with linear least squares regression: R≥0.995.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration, prior to sample analysis.	Value of second source for all analyte(s) within ±10%R of expected value (initial source).	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Accuracy	Value of second source for all analyte(s) within ±10%R of expected value (initial source).
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	ICV within ±10% of expected value and CCV within ±20% of expected value.	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful CCV. If that fails, then repeat initial calibration.	Analyst	Laboratory Accuracy	ICV within ±10% of expected value and CCV within ±20% of expected value.
Calibration blanks (initial [ICB] and continuing [CCB])	ICB: Daily, before sample run. CCB: After every 10 field samples and at the end of the analysis sequence	ICB and CCB ≤2x MDL.	ICB and CCB: Correct problem, then repeat ICB and CCB and reanalyze all samples since last successful CCB. If that fails, then Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	Analyst	Laboratory Representativeness (Absence of interference/ contamination)	ICB and CCB ≤2x MDL.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code "B".	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous.
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike (MS)	One MS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Prep analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%
Matrix Spike Duplicates (MSD) or Sample Duplicates	One per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.8 for metals Solids Worksheet 15.16 for metals Aqueous Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial Dilution Test	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >25x MDL for CVAA.	Perform matrix spike. Flagging criteria are not appropriate.	Analyst	Precision (field samples)	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >25x MDL for CVAA.
Method of standard additions (MSA) or Internal Standard calibration	When matrix interference is suspected	Document use of MSA in the case narrative.	Not Applicable	Analyst	Accuracy	Document use of MSA in the case narrative.
Results reported between LOD and LOQ	All positive results must be confirmed	Not Applicable	Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	Not Applicable

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.10 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid
Analytical Group	TCLP Volatiles
Concentration Level	Low
Sampling SOP	SOP EI-FS101
Analytical Method / SOP Reference	SW-846 8260B/ LAB SOP#MS005
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples. Flagging criteria are not appropriate and problem must be corrected. No samples may be accepted without a valid tune.	Analyst	Laboratory Accuracy	Refer to method for specific ion criteria.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	Average response factor (RF) for SPCCs: VOCs - RF≥0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥0.1 for chloromethane, bromoform,and 1,1-dichloroethane. RSD for RFs for CCCs: VOCs - RSD≤30%and one option below; Option 1: RSD for each analyte ≤15% Option 2: linear least squares regression R ≥0.995 Option 3: non-linear regression - coefficient of determination (COD) R^2 ≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)	If the acceptance criteria were not met, correct problem and re-calibration is performed before any samples may be analyzed.	Analyst	Laboratory Precision	%RSDs and correlations: %RSD≤15% (≤30% for CCCs) or Correlation coefficient R≥0.995 or COD R^2≥0.99 RF for SPCCs: VOCs – RF≥0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥0.1 for chloromethane, bromoform,and 1,1-dichloroethane.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration and before field samples	Value of second source for all analytes within ±25% of expected value (initial source).	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Analyst	Laboratory Accuracy	Value of second source for all analytes within ± 25% of expected value (initial source).
Continuing Calibration Verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time	Average RF for SPCCs: VOCs RF≥0.30 for Chlorobenzene and 1,1,2,2- tetrachlorolethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. %Difference/Drift for CCCs: VOCs and SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration.)	Correct problem, then rerun CV. If that fails, repeat initial calibration. Corrective action may include re-analysis of samples. Apply Q-flag if no sample material remains and analyte exceeds criteria.	Analyst	Laboratory Precision	%Ds or %Drift: %D≤20%; %Drift≤20% RF for SPCCs: VOCs - RF≥0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥ 0.1 for chloromethane, bromoform,and 1,1-dichloroethane.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	One per analytical batch per matrix	All Target Compounds <1/2 RL. For the common laboratory contaminant 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.17 for TCLP VOCs Solids	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code "B".	Analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. For the common laboratory contaminant 2-butanone, all analytes <RL. Project QLs for all target compounds are specified in: Worksheet 15.17 for TCLP VOCs Solids
Laboratory Control Sample (LCS)	One LCS per analytical batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.17 for TCLP VOCs Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.17 for TCLP VOCs Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.17 for TCLP VOCs Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.17 for TCLP VOCs Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%
Internal standards (IS) verification	In all field samples and standards	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory. If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Analyst	Accuracy (Instrument sensitivity control)	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard
Quantitation Verification	All field samples and standards	RRT of each target analyte in each calibration standard within ±0.06 RRT units.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate. Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	RRT of each target analyte in each calibration standard within ±0.06 RRT units.

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.11 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid
Analytical Group	TCLP Semivolatiles
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101
Analytical Method / SOP Reference	SW-846 8270/ LAB SOP#MS006
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples. Flagging criteria are not appropriate and problem must be corrected. No samples may be accepted without a valid tune.	Analyst	Laboratory Accuracy	Refer to method for specific ion criteria.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	Average response factor (RF) for SPCCs: SVOCs - RF≥0.050. RSD for RFs for CCCs: SVOCs - RSD≤30%and one option below; Option 1: RSD for each analyte ≤15% Option 2: linear least squares regression R ≥0.995 Option 3: non-linear regression - coefficient of determination (COD) R^2 ≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)	If the acceptance criteria were not met, correct problem and re-calibration is performed before any samples may be analyzed.	Analyst	Laboratory Precision	%RSDs and correlations: %RSD≤15% (≤30% for CCCs) or Correlation coefficient R≥0.995 or COD R^2≥0.99 RF for SPCCs: SVOCs - RF≥0.050.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration and before field samples	Value of second source for all analytes within ±25%D of expected value (initial source).	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Analyst	Laboratory Precision	Value of second source for all analytes within ±25%D of expected value (initial source).
Continuing Calibration Verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time	Average RF for SPCCs: SVOCs RF≥0.050. %Difference/Drift for CCCs: SVOCs ≤20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration.)	Correct problem, then rerun CV. If that fails, repeat initial calibration. Corrective action may include re-analysis of samples. Apply Q-flag if no sample material remains and analyte exceeds criteria.	Analyst	Laboratory Precision	%Ds or %Drift: %D≤20%; %Drift≤20% RF for SPCCs: SVOCs - RF≥0.050.
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.18 for TCLP SVOCs Solids	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code “B”.	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.18 for TCLP SVOCs Solids

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.18 for TCLP SVOCs Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.18 for TCLP SVOCs Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.18 for TCLP SVOCs Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.18 for TCLP SVOCs Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%
Internal standards (IS) verification	In all field samples and standards	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory. If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Analyst	Accuracy (Instrument sensitivity control)	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Quantitation Verification	All field samples and standards	RRT of each target analyte in each calibration standard within ±0.06 RRT units.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate. Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	RRT of each target analyte in each calibration standard within ±0.06 RRT units.

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.12 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid
Analytical Group	TCLP Pesticides
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101
Analytical Method / SOP Reference	SW-846 8081/ LAB SOP#GC015
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration. Flagging criteria are not appropriate.	Analyst	Laboratory Precision	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Retention time window position establishment and verification for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift for position establishment. Each calibration verification standard for retention time verification.	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift. Analyte shall be within established window for each calibration verification.	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window. Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window. No samples shall be run without a verified retention time window at the initial verification.	Analyst	Laboratory Representativeness	Each analyte shall be within established window.
Breakdown check (Endrin/DDT)	Daily prior to analysis of samples	Degradation ≤ 15%D for both Endrin and DDT.	Correct problems then repeat breakdown check. Flagging criteria are not appropriate. No samples shall be run until degradation ≤15%.	Analyst	Laboratory Precision	Degradation ≤ 15%D for both Endrin and DDT.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration	Value of second source for all analytes within ±20%D of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Precision	Value of second source for all analytes within ±20%D of expected value (initial source)
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	All analytes within ±20%D of expected value from the ICAL	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification. As long as the CCV is acceptable, a new initial instrument calibration is not necessary.	Analyst	Laboratory Precision	All analytes within ±20%D of expected value from the ICAL

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate.	Analyst	Laboratory Representativeness	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.19 for TCLP pesticides Solids	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code “B”.	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.19 for TCLP pesticides Solids.
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.19 for TCLP pesticides Solids Ref.: DoD QSM, if available, otherwise laboratory’s own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.19 for TCLP pesticides Solids Ref.: DoD QSM, if available, otherwise laboratory’s own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.19 for TCLP pesticides Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.19 for TCLP pesticides Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%
Quantitation Verification and Confirmation	All positive results must be confirmed (exclude toxaphene and technical chlordane)	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column or detector RPD ≤40%.	Apply J-flag if RPD >40% or Q-flag if sample is not confirmed. Discuss in the case narrative. Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative. Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness and Precision	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column or detector RPD ≤40%.

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.13 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Soil
Analytical Group	TCLP Herbicides
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101
Analytical Method / SOP Reference	SW-846 8151/ LAB SOP# TGC 011
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Gulf Coast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis as needed (see CCV passing criteria below)	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration. Flagging criteria are not appropriate.	Analyst	Laboratory Precision	One of the options below: Option 1: RSD≤20% for each analyte. Option 2: linear least squares regression: R≥0.995 Option 3: non-linear regression: coefficient of determination (COD) R^2≥0.99 (6 points shall be used for second order, 7 points shall be used for third order)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Retention time window position establishment and verification for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift for position establishment. Each calibration verification standard for retention time verification.	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift. Analyte shall be within established window for each calibration verification.	Correct problem, and then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window. Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window. No samples shall be run without a verified retention time window at the initial verification.	Analyst	Representativeness	Each analyte shall be within established window.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration	Value of second source for all analytes within ±20%D of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Precision	Value of second source for all analytes within ±20%D of expected value (initial source)
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	All analytes within ±20%D of expected value from the ICAL	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification. As long as the CCV is acceptable, a new initial instrument calibration is not necessary.	Analyst	Laboratory Precision	All analytes within ±20%D of expected value from the ICAL
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.	Correct problem, then rerun ICAL. Flagging criteria are not appropriate.	Analyst	Laboratory Representativeness	RT width is ± 3 times standard deviation for each analyte RT from 72-hour study.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.20 for TCLP herbicides Solids	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code “B”.	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.20 for TCLP herbicides Solids.
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.20 for TCLP herbicides Solids Ref.: DoD QSM, if available, otherwise laboratory’s own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.20 for TCLP herbicides Solids Ref.: DoD QSM, if available, otherwise laboratory’s own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate Spikes	All field and QC samples	QC acceptance criteria for all target compounds as specified in: Worksheet 15.20 for TCLP herbicides Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Accuracy (Individual sample preparation efficiency control)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.20 for TCLP herbicides Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%
Quantitation Verification and Confirmation	All positive results must be confirmed	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column or detector RPD ≤40%.	Apply J-flag if RPD >40% or Q-flag if sample is not confirmed. Discuss in the case narrative. Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative. Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness and Precision	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column or detector RPD ≤40%.

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.14 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid
Analytical Group	TCLP ICP Metals
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101
Analytical Method / SOP Reference	SW-846 6010B/ LAB SOP# Met 100, Met 103
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis.	Initial calibration for all analytes (ICAL): Minimum one high standard and a calibration blank; No acceptance criteria unless more than one standard is used, in which case r ≥0.995.	Correct problem then repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until ICAL has passed.	Analyst	Laboratory Precision	Initial calibration for all analytes (ICAL): Minimum one high standard and a calibration blank; No acceptance criteria unless more than one standard is used, in which case r ≥0.995.
Linear dynamic range or High-level calibration check standard	Every 6 months	Within ±10%R of expected value.	Not Applicable	Analyst	Laboratory Accuracy	Within ±10%R of expected value.
Low-level calibration check standard	Daily, after one-point initial calibration	Within ±20%R of expected value. Low-level calibration check standard should be less than or equal to the reporting limit.	Correct problem then repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until ICAL has passed.	Analyst	Laboratory Accuracy	Within ±20%R of expected value. Low-level calibration check standard should be less than or equal to the reporting limit.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration, prior to sample analysis.	Value of second source for all analyte(s) within ±10%R of expected value (initial source).	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Accuracy	Value of second source for all analyte(s) within ±10%R of expected value (initial source).
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	ICV and CCV within ±10% of expected value.	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful CCV. If that fails, then repeat initial calibration..	Analyst	Laboratory Accuracy	ICV and CCV within ±10% of expected value.
Calibration blanks (initial [ICB] and continuing [CCB])	ICB: Daily, before sample run. CCB: After every 10 field samples and at the end of the analysis sequence	ICB and CCB ≤2x MDL.	ICB and CCB: Correct problem, then repeat ICB and CCB and reanalyze all samples since last successful CCB. If that fails, then Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	Analyst	Laboratory Representativeness (Absence of interference/ contamination)	ICB and CCB ≤2x MDL.
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.21 for TCLP metals Solids	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code "B".	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.21 for TCLP metals Solids

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.21 for TCLP metals Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.21 for TCLP metals Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Interference check solutions (ICS)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all nonspiked analytes <2x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS. Flagging criteria are not appropriate. No samples may be analyzed without a valid ICS.	Analyst	Accuracy	ICS-A: Absolute value of concentration for all nonspiked analytes <2x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value
Serial Dilution Test	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >50x MDL for ICP.	Perform post-digestion spike (PDS) addition. Flagging criteria are not appropriate.	Analyst	Precision (field samples)	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >50x MDL for ICP.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Post-digestion spike (PDS) addition	When dilution test fails or analyte concentration in all samples <50x MDL	Recovery within 75-125% of expected result. The spike addition should produce a level between 10x to 100x MDL.	Run samples by method of standard additions (MSA) or Apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition.	Analyst	Accuracy	Recovery within 75-125% of expected result. The spike addition should produce a level between 10x to 100x MDL.
Method of standard additions (MSA) or Internal Standard calibration	When matrix interference is suspected	Document use of MSA in the case narrative.	Not Applicable	Analyst	Accuracy	Document use of MSA in the case narrative.
Results reported between LOD and LOQ	All positive results must be confirmed	Not Applicable	Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	Not Applicable

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.15 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Solid
Analytical Group	TCLP Mercury
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP EI-FS101
Analytical Method / SOP Reference	SW-846 7470 LAB SOP#MET106
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis.	Initial calibration for all analytes (ICAL) Minimum 5 standards and a calibration blank with linear least squares regression: R≥0.995.	Correct problem then repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until ICAL has passed.	Analyst	Laboratory Precision	Initial calibration for all analytes (ICAL) Minimum 5 standards and a calibration blank with linear least squares regression: R≥0.995.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration, prior to sample analysis.	Value of second source for all analyte(s) within ±10%R of expected value (initial source).	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Accuracy	Value of second source for all analyte(s) within ±10%R of expected value (initial source).

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	ICV within ±10% of expected value and CCV within ±20% of expected value.	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful CCV. If that fails, then repeat initial calibration..	Analyst	Laboratory Accuracy	ICV within ±10% of expected value and CCV within ±20% of expected value.
Calibration blanks (initial [ICB] and continuing [CCB])	ICB: Daily, before sample run. CCB: After every 10 field samples and at the end of the analysis sequence	ICB and CCB ≤2x MDL.	ICB and CCB: Correct problem, then repeat ICB and CCB and reanalyze all samples since last successful CCB. If that fails, then Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	Analyst	Laboratory Representativeness (Absence of interference/ contamination)	ICB and CCB ≤2x MDL.
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.21 for TCLP Mercury Solids	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code "B".	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.21 for TCLP Mercury Solids

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.21 for TCLP Mercury Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.21 for TCLP Mercury Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Serial Dilution Test	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >25x MDL for CVAA.	Perform matrix spike. Flagging criteria are not appropriate.	Analyst	Precision (field samples)	Five-fold dilution must agree within ± 10% of the original determination. Only applicable for samples with concentrations >25x MDL for CVAA.
Method of standard additions (MSA) or Internal Standard calibration	When matrix interference is suspected	Document use of MSA in the case narrative.	Not Applicable	Analyst	Accuracy	Document use of MSA in the case narrative.
Results reported between LOD and LOQ	All positive results must be confirmed	Not Applicable	Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	Not Applicable

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.16 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Soil
Analytical Group	Total Cyanide
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP T-FS-011
Analytical Method / SOP Reference	SW-846 9014 LAB SOP#GN113, 115
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Cyanide Solids	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Cyanide Solids

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Cyanide Solids	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Cyanide Solids
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Cyanide Solids	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Cyanide Solids
Initial Calibration (ICAL)	Initial calibration prior to sample analysis.	Initial calibration for all analytes (ICAL) Minimum 6 standards and a calibration blank with linear least squares regression: R≥0.995.	Correct problem then repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until ICAL has passed.	Analyst	Laboratory Precision	Initial calibration for all analytes (ICAL) Minimum 6 standards and a calibration blank with linear least squares regression: R≥0.995.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Distilled standards (one high and one low)	Once per multipoint calibration	Within ±15%R of true value.	Correct problem, then repeat distilled standards. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until distilled standards have passed.	Analyst	Laboratory Accuracy	Within ±15%R of true value.
Initial calibration verification (ICV) (Second Source)	Once after each initial calibration, prior to sample analysis.	Value of second source for all analyte(s) within ±15%D of expected value (initial source).	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until calibration has been verified.	Analyst	Laboratory Precision	Value of second source for all analyte(s) within ±15%D of expected value (initial source).
Continuing Calibration Verification (CCV)	Opening CCV, then every 10 samples, with closing CCV	%D≤10%	Correct problem then repeat CCV and reanalyze all samples since last successful CCV. If that fails, then repeat initial calibration.	Analyst	Laboratory Precision	%D≤10%
Method Blank (MB)	One per preparatory batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Cyanide Solids	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code “B”.	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Cyanide Solids

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One LCS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Cyanide Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Cyanide Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%
Matrix Spike (MS)	One MS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Cyanide Solids Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Prep analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Cyanide Solids Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicates (MSD) or Sample Duplicates	One per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Cyanide Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Cyanide Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)
Results reported between LOD and LOQ	All positive results must be confirmed	Not Applicable	Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	Not Applicable

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.17 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Soil
Analytical Group	Total Sulfide
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP T-FS-011
Analytical Method / SOP Reference	USEPA 376.1M/ LAB SOP#GN140
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Blank	1 per 20 field samples or per day	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Sulfide Solids	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Sulfide Solids

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	1 per 20 field samples or per day per matrix per sampling technique	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Sulfide Solids	If the criterion is not met for the field blanks, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the blank contamination and usefulness of the data. Apply U-flag (Region II) to all results for the specific analyte(s) in all samples in the associated preparatory batch using the 5x/10x rule.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Sulfide Solids
Field Duplicate	1 per 10 field samples per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Sulfide Solids	If the criterion is not met for the field duplicates, a careful examination of the sampling techniques, sample media, and analytical procedure in conjunction with other analytical quality control criteria will be conducted to identify the cause of the high RPD and the usefulness of the data. If one of the duplicate pair is detected above the method reporting limit (RL) and the remaining pair is non-detect, then the data will be qualified as estimated or rejected depending upon the severity (i.e. >2RL). Apply J-flag (Region II) to sample and duplicate pair.	Field Personnel/Lantdiv Shaw Chemist/Data Validator	Field Precision	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Sulfide Solids

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (MB)	One per analytical batch per matrix	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Sulfide Solids	The source of the contamination is investigated and eliminated before proceeding with further analysis. Correct the problem. Any sample associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying code “B”.	Analyst/Prep analyst	Laboratory Representativeness (Absence of interference/ contamination)	All Target Compounds <1/2 RL. Project QLs for all target compounds are specified in: Worksheet 15.22 for Total Sulfide Solids
Laboratory Control Sample (LCS)	One LCS per analytical batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Sulfide Solids Ref.: DoD QSM, if available, otherwise laboratory’s own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Source of poor recovery is investigated and eliminated before proceeding with further analysis. If corrective action fails or insufficient volumes, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Analyst/Prep analyst	Laboratory Accuracy	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Sulfide Solids Ref.: DoD QSM, if available, otherwise laboratory’s own in-house criteria. %Recovery = (Calculated Value/True Value) *100%%Recovery = (Calculated Value/True Value) *100%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike (MS)	One MS per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Sulfide Solids Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%	Examine the project-specific DQOs. MS is for matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. The Shaw Chemist will decide to either report the data as is with a notation in the analytical narrative or if the samples should be re-extract and re-analyzed. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst/Prep analyst	Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Sulfide Solids Ref.: DoD QSM. QC MS acceptance criteria uses LCS criteria as specified by DoD QSM. %Recovery = (Calculated Value/True Value) *100%
Matrix Spike Duplicates (MSD) or Sample Duplicates	One per preparatory batch per matrix	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Sulfide Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)	See matrix spike corrective actions noted above. The data shall be evaluated to determine the source of difference.	Analyst	Precision and Accuracy (field samples)	QC acceptance criteria for all target compounds as specified in: Worksheet 15.22 for Total Sulfide Solids Ref.: DoD QSM, if available, otherwise laboratory's own in-house criteria. %Recovery = (Calculated Value – Sample Value/True Value) *100% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)
Results reported between LOD and LOQ	All positive results must be confirmed	Not Applicable	Apply J-flag to all results between LOD and LOQ.	Analyst	Representativeness	Not Applicable

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.18 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Soil
Analytical Group	Flash Point
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP T-FS-011
Analytical Method / SOP Reference	SW-846 1010/ LAB SOP#GN121
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Opening sequence, then every 10 samples and at the end	p-Xylene, first source flash point of p-Xylene is 77 ± 4.3°F	Second analyst witnessing flash. Calibrate thermometer	Analyst	Laboratory Accuracy	flash point of p-Xylene is 77 ± 4.3°F
Initial calibration verification (ICV) (Second Source)	1 per day	p-Xylene, second source flash point of p-Xylene is 77 ± 4.3°F	Second analyst witnessing flash. Calibrate thermometer	Analyst	Laboratory Accuracy	flash point of p-Xylene is 77 ± 4.3°F

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #28.19 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Soil
Analytical Group	Corrosivity as pH
Concentration Level	Low
Sampling SOP	SOP T FS-010 SOP T-FS-011
Analytical Method / SOP Reference	SW-846 9045/ LAB SOP#GN179
Sampler's Name	TBD
Field Sampling Organization	Shaw Environmental
Analytical Organization	Accutest Laboratories Southeast, Inc.
Number of Sample Locations	TBD

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration (ICAL)	Initial calibration prior to sample analysis.	Initial calibration for all analytes (ICAL): Minimum three standards. pH 4.00, 7.00, and 10.00. Percent recoveries for initial calibration must be within ±0.05 units of standard true value for pH.	Correct problem then repeat initial calibration. Flagging criteria are not appropriate. Problem must be corrected. No samples may be run until ICAL has passed.	Analyst	Laboratory Accuracy	Initial calibration for all analytes (ICAL): Minimum three standards. pH 4.00, 7.00, and 10.00. Percent recoveries for initial calibration must be within ±0.05 units of standard true value for pH.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Continuing Calibration Verification (CCV)	Every 10 samples	Percent recoveries for continuing calibration must be within ±0.05 units of standard true value for pH.	Rerun standard, and/or recalibrate instrument and reanalyzed all samples since last acceptable CCV.	Analyst	Laboratory Accuracy	Percent recoveries for continuing calibration must be within ±0.05 units of standard true value for pH.
Sample Duplicate	One per batch per matrix or every 20 samples	QC acceptance criteria for all target compounds as: %RPD≤10% RPD = (Difference between S and SD) * 100 / (Average of S and SD)	If sufficient sample volume is available, reanalyze affected samples. Qualify data as needed.	Analyst	Laboratory Precision	QC acceptance criteria for all target compounds as: %RPD≤10% RPD = (Difference between MS and MSD) * 100 / (Average of MS and MSD)

Ref: USEPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Update IIIB (USEPA, 2004) and DoD Quality Systems Manual for Environmental Laboratories, Final Version 3 (DoD, 2006).

QAPP Worksheet #29
Project Documents and Records Table

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field/ Communication Log Books	Not Applicable	Chains-of-Custody Records	Laboratory QSM Manual	
Daily Tailgate Safety Meeting Forms		Sample Receipt Confirmation Forms	Laboratory Accreditation Certificates or Letters	
Site Maps with Sampling Locations		Internal Sample Tracking Forms	Communication Logbooks	
Chain-of-Custody Records		Extraction and Prep Logs Books	Data Review-noted in Logbooks	
Custody Seals		LIMs Login	EDDs with site specific goals evaluated and or entered	
Airbill Records		Standard Logbooks	PDF of Final Laboratory Data Report	
		Non Conformance Records	Weekly Health and Safety Communications	
		Communication Logbooks	Safety Audit Checklists	
		Sample Chronology (time of receipt, extraction, and analysis)	Validation Reports on applicable samples	
		Identification of QC Samples (Blanks, Duplicates, MS/MSD, LCS)		
		Definitions of Laboratory Data Qualifiers		
		Documentation of Lab QC Issues		
		Instrument Calibration Log Books		
		Instrument Maintenance Log Books		
		Electronic Data Deliverables		
		Laboratory Name		

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
		Case Narrative		
		Laboratory Sample Accession Numbers		
		Reporting Forms		
		Reporting Checklists- for Completeness		
		Signature of laboratory sign-off		
		MDL Studies		
		PE Results		
		Laboratory Accreditation Certificates or Letters		
		Site QAPP		
		Sample Disposal Records		

QAPP Worksheet #30 (UFP-QAPP Manual Section 3.5.2.3) -- Analytical Services Table

Identify all laboratories or organizations that will provide analytical services for the project, including on-site screening, on-site definitive, and off-site laboratory analytical work. Group by matrix, analytical group, concentration, and sample location or ID number. If applicable, identify the subcontractor laboratories and backup laboratory or organization that will be used if the primary laboratory or organization cannot be used.

Matrix	Analytical Group	Concentration Level	Sample Locations/ID Number	Analytical SOP	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization [1] (name and address, contact person and telephone number)
Soil	TPH GRO	Low	TBD	SW-846 5035A 8015M/ SOP # OP020/GC010	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Soil	TCL VOC	Low	TBD	SW-846 8260B/ SOP #MS005/OP020	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Soil	TCL SVOC	Low	TBD	SW-846 3550B 8270C/ SOP # OP007/MS006	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Soil	Explosives	Low	TBD	SW-846 8330/SOP# GC016/OP019	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Soil	Pesticides	Low	TBD	SW-846 3550B 8081A/ SOP #OP009/GC015	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700

Matrix	Analytical Group	Concentration Level	Sample Locations/ID Number	Analytical SOP	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization [1] (name and address, contact person and telephone number)
Soil	Total PCB's	Low	TBD	SW-846 3550B 8082/ SOP # OP009/GC014	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Soil	TPH DRO	Low	TBD	SW-846 3550B 8015M/ SOP #OP011/GC011	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Soil	TAL Metals – ICP	Low	TBD	SW-846 3050B 6010B/ SOP #MET104/MET100	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Soil	Metals – Mercury	Low	TBD	SW-846 3050B 7471A/ SOP #MET 105	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Sediment	TPH GRO	Low	TBD	SW-846 5035A 8015M/ SOP # OP020/GC010	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Sediment	TCL VOC	Low	TBD	SW-846 8260B/ SOP #MS005/OP020	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700

Matrix	Analytical Group	Concentration Level	Sample Locations/ID Number	Analytical SOP	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization [1] (name and address, contact person and telephone number)
Sediment	TCL SVOC	Low	TBD	SW-846 3550B 8270C/ SOP # OP007/MS006	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Sediment	Explosives	Low	TBD	SW-846 8330/SOP# GC016/OP019	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Sediment	Pesticides	Low	TBD	SW-846 3550B 8081A/ SOP # OP009/GC015	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Sediment	Total PCB's	Low	TBD	SW-846 3550B 8082/ SOP # OP009/GC014	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Sediment	TPH DRO	Low	TBD	SW-846 3550B 8015M/ SOP #OP011/GC011	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Sediment	TAL Metals – ICP	Low	TBD	SW-846 3050B 6010B/ SOP # MET104/MET100	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700

Sediment	Metals – Mercury	Low	TBD	SW-846 3050B 7471A/ SOP #MET 105	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Blanks / Aqueous	TPH GRO	Low	TBD	SW-846 5030B 8015M/ SOP # OP021/GC010	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Blanks / Aqueous	TCL VOC	Low	TBD	SW-846 5030B 8260B/ SOP #MS005/OP021	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Blanks / Aqueous	TCL SVOC	Low	TBD	SW-846 3510C 8270C/ SOP # OP006/MS006	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Blanks / Aqueous	Explosives	Low	TBD	SW-846 8330/SOP# GC016/OP018	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Blanks / Aqueous	Pesticides	Low	TBD	SW-846 3510C 8081A/ SOP # OP008/GC015	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Blanks / Aqueous	Total PCB's	Low	TBD	SW-846 3510C 8082/ SOP # OP008/GC014	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700

Blanks / Aqueous	TPH DRO	Low	TBD	SW-846 3510C 8015M/ SOP #OP010/GC011	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Blanks / Aqueous	TAL Metals – ICP	Low	TBD	SW-846 3010A 6010B/ SOP #MET 103/MET100	CLP Like / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Blanks / Aqueous	Metals – Mercury	Low	TBD	SW-846 7470A/ SOP #MET106	CLP Like / 7 Calendar Days	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	TCLP Volatiles	Low	TBD	SW-846 1311 5030B 8260B/ SOP #OP041/OP021/MS005	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	TCLP Semivolatiles	Low	TBD	SW-846 1311 3510C 8270C/ SOP #OP040/OP006/MS006	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	TCLP Pesticide	Low	TBD	SW-846 1311 3510C 8081A/ SOP # OP040/OP008/GC015	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	TCLP Herbicides	Low	TBD	SW-846 1311 8151A/ SOP #TGN048/TGC011[4]	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700

Disposal Soil and Sediments	TCLP Metals ICP	Low	TBD	SW-846 1311 3010A 6010B/ SOP# OP040/MET 103/MET100	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	TCLP Metals Hg	Low	TBD	SW-846 1311 7470A/ SOP # OP040/MET106	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	Cyanide, Total	Low	TBD	SW-846 9014/ SOP #GN113/GN115	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	Sulfide, Total	Low	TBD	EPA 376.1M/ SOP #GN140	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	Ignitability (Flashpoint)	Low	TBD	SW-846 1010/ SOP #GN121	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	Corrosivity as pH	Low	TBD	SW-846 CHAP7/9045/ SOP #GN179	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	Total PCB's	Low	TBD	SW-846 3550B 8082/ SOP # OP009/GC014	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700

Disposal Soil and Sediments	TPH DRO	Low	TBD	SW-846 3550B 8015M/ SOP # OP011/GC011	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700
Disposal Soil and Sediments	TPH GRO	Low	TBD	SW-846 5035A 8015M/ SOP #OP020/GC010	Summary Report / 7 Calendar Days	Accutest Orlando 4405 Vineland Road Suite C-1 Orlando, FL 32811 Heather Wandrey (407)425-6700	Accutest Gulf Coast 10165 Harwin Drive Suite 150 Houston, TX 77036 Heather Wandrey Phone: (713) 271-4700

[1] If a backup laboratory is required due to laboratory loading or any other issues Accutest Houston will serve as a back up laboratory. Heather Wandrey will still serve as the prime laboratory project manager for this Shaw project. Alternatively, Accutest New Jersey may also serve as a back up laboratory. Accutest Orlando will notify the Shaw Program Chemist, Natasha Sullivan, prior to any sample transfers. All Accutest laboratories carry NEELAC certification and have proper instrumentation and qualifications to perform the analysis required by this project.

QAPP Worksheet #31
Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Review of QAPP with Field Staff	1/prior to sampling start up	Internal	Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc.	Cano Hernandez Project Superintendent Shaw Environmental, Inc.	Cano Hernandez Project Superintendent Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc.
Daily QC Report	Daily	Internal	Shaw Environmental, Inc.	Cano Hernandez Project Superintendent Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc.	Cano Hernandez Project Superintendent Shaw Environmental, Inc.
Laboratory Assessment for appropriate Certifications, Capacity and QAPP Review with Staff	1/prior to sampling start up	Internal	Shaw Environmental, Inc.	Natasha Sullivan, Program Chemist Shaw Environmental, Inc.	Harry Behazadi Laboratory Manager, Accutest Orlando Svetlana Izosimova, Laboratory QAO Accutest Orlando	Harry Behazadi Laboratory Manager, Accutest Orlando Svetlana Izosimova, Laboratory QAO Accutest Orlando	Natasha Sullivan, Program Chemist Shaw Environmental, Inc.
Daily Tailgate Safety Meeting	Daily	Internal	Shaw Environmental, Inc.	Cano Hernandez Project Superintendent Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc. Kym Edelman, Health and Safety, Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc. Cano Hernandez Project Superintendent Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc. Cano Hernandez Project Superintendent Shaw Environmental, Inc.
Field Sampling and COC Review Against QAPP Requirements	Daily	Internal	Shaw Environmental, Inc.	Natasha Sullivan, Program Chemist Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc. Cano Hernandez Project Superintendent Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc. Cano Hernandez Project Superintendent Shaw Environmental, Inc.	Natasha Sullivan, Program Chemist Shaw Environmental, Inc.
Laboratory Report Deliverables and Analytical Results Against QAPP Requirements	Per Sample Delivery Group	Internal	Shaw Environmental, Inc.	Natasha Sullivan, Program Chemist Shaw Environmental, Inc.	Svetlana Izosimova, Laboratory QAO Accutest Orlando	Svetlana Izosimova, Laboratory QAO Accutest Orlando	Natasha Sullivan, Program Chemist Shaw Environmental, Inc.

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Validation	Confirmation Samples below site specific project action limits.	Internal	Shaw Environmental, Inc.	Eric Malarek, Chemist Shaw Environmental, Inc.	Svetlana Izosimova, Laboratory QAO Accutest Orlando	Svetlana Izosimova, Laboratory QAO Accutest Orlando	Eric Malarek, Chemist Shaw Environmental, Inc.

QAPP Worksheet #32
Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Review of QAPP with Field Staff	Contained with written report Daily QC Report for that day.	William Hughes, Project Manager, Shaw Environmental, Inc.	Immediately not to exceed with 24 hours.	Daily QC Report would be amended with corrective action.	William Hughes, Project Manager, Shaw Environmental, Inc.	Immediate within 24 hours.
Laboratory Assessment for appropriate Certifications, Capacity and QAPP Review with Staff	Receipt of copies of certifications. Email traffic concerning lab capacity prior to sampling start-up. QAPP Sign-off sheet received from laboratory.	William Hughes, Project Manager, Shaw Environmental, Inc.	Immediate.	Response to email.	William Hughes, Project Manager, Shaw Environmental, Inc.	48 hours after notification.
Daily Tailgate Safety Meeting	Verbal debriefing and daily sign off log. If a safety violation occurs, a Supervisor Injury Employee Report is completed.	William Hughes, Project Manager, Shaw Environmental, Inc. Kym Edelman, Health and Safety, Shaw Environmental, Inc.	Immediately not to exceed 24 hours.	Included as part of the process of the Supervisor Injury Employee Report.	Cano Hernandez Project Superintendent Shaw Environmental, Inc	Immediate within 24 hours.
Daily QC Report	Contained with written report.	William Hughes, Project Manager, Shaw Environmental, Inc.	Immediately not to exceed 24 hours.	Daily QC Report would be amended with corrective action.	Cano Hernandez Project Superintendent Shaw Environmental, Inc.	Immediate within 24 hours.
Field Sampling and COC Review Against QAPP Requirements	Communication may be in the form of email traffic	William Hughes, Project Manager, Shaw Environmental, Inc. Cano Hernandez Project Superintendent Shaw Environmental, Inc.	24 hours after sampling.	Response to email.	William Hughes, Project Manager, Shaw Environmental, Inc.	48 hours after notification.

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Laboratory Report Deliverables and Analytical Results Against QAPP Requirements	Communication may be in the form of email traffic	William Hughes, Project Manager, Shaw Environmental, Inc. Heather Wandrey Project Manager Accutest Orlando	24 hours after completion of analytical	If required laboratory reports will be amended and corrections noted in the analytical narrative.	William Hughes, Project Manager, Shaw Environmental, Inc.	72 hours after notification.
Validation	Communication may be in the form of email traffic requesting additional laboratory form's, back up data that may be missing and or clarification of the analytical report.	Svetlana Izosimova, Laboratory QAO Accutest Orlando	24 hours after finding deficiency.	If required laboratory reports will be amended and corrections noted in the analytical narrative and contained with the validation report.	Eric Malarek, Chemist Shaw Environmental, Inc	Up to 7 days.

QAPP Worksheet #33
QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Progress Reports	Monthly	Monthly after project start up	William Hughes, Project Manager, Shaw Environmental, Inc.	Kevin Cloe, Remedial Project Manager, Navy Anselmo Martinez, ROICC, Navy Public Works Department
Construction QC Meetings	Biweekly	Conducted biweekly following mobilization	Archie Vega, QC Representative, Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc. Cano Hernandez, Project Superintendent, Shaw Environmental, Inc. Kevin Cloe, Remedial Project Manager, Navy Anselmo Martinez, ROICC, Navy Public Works Department
Validation Report	Once at End of Project	30 days after completion of analytical data	Eric Malarek, Chemist Shaw Environmental, Inc.	William Hughes, Project Manager, Shaw Environmental, Inc. Natasha Sullivan, Program Chemist Shaw Environmental, Inc
Final Project Closure Report	Once at End of Project	30 days after demobilization	William Hughes, Project Manager, Shaw Environmental, Inc. Natasha Sullivan, Program Chemist, Shaw Environmental, Inc.	Kevin Cloe, Remedial Project Manager, Navy Anselmo Martinez, ROICC, Navy Public Works Department

QAPP Worksheet #34
Verification (Step I) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Field Staff Training	Personnel assigned to the project, including field personnel and subcontractors, will be qualified to perform the tasks to which they are assigned. Said personnel will meet requirements set forth in OPNAVINST 5090.1B 25-5.8. This includes but is not limited to basic sampling techniques; field testing methodology, task-specific sampling methods, maintenance of environmental paperwork, and how to avoid cross contamination. In addition to education and experience, specific training may be required to qualify individuals to perform certain activities. Training will be documented appropriately and the forms placed in the project file as a record. Project personnel will receive an orientation to the full SAP and the HASP as appropriate to their responsibilities before participation in project activities. Training of field personnel will be provided by the Site Supervisor, the QA Officer, or by a qualified designee.	Internal	William Hughes / Shaw E&I Cano Hernandez / Shaw E&I Kym Edelman / Shaw E&I Natasha Sullivan / Shaw E&I Svetlana Izosimova / Accutest Orlando Harry Behazadi / Accutest Orlando
QAPP	A copy of the reviewed and approved version of the QAPP will be distributed to the laboratory and be available for review for all Shaw personnel involved in this project. It is the responsibility of the Shaw Program Chemist to ensure delivery of a copy of QAPP to the laboratory. The laboratory quality control manager is responsible for review of QAPP with laboratory staff. The Shaw project manager and site supervisor be responsible for ensuring that all staff have reviewed the final QAPP.	Internal/External	William Hughes / Shaw E&I Cano Hernandez / Shaw E&I Natasha Sullivan / Shaw E&I

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Laboratory Quality Systems Manual	Accutest has a detailed Quality Systems Manual, Volume VII, Revision I, dated March 2006 that is designed to meet the quality program requirements of NELAC and ISO Guide 25. This Quality Systems Manual is included in Appendix B. Accutest will provide Shaw Environmental a copy of the latest QSM prior to project start up.	Internal/External	Svetlana Izosimova / Accutest Orlando Harry Behazadi / Accutest Orlando Natasha Sullivan / Shaw E&I
Laboratory Staff Training	Laboratory senior management staff retains oversight responsibility for the data integrity program and retains the ultimate responsibility for execution of the data integrity program elements. Senior laboratory management staff is responsible for providing the resources required to conduct SOPs, ethics training, and operate data integrity evaluation procedures. Laboratory employees receive technical ethics training during new employee orientation. All employees are required to attend ethics refresher training and to sign an ethical conduct agreement annually, which verifies their understanding of the laboratories ethics policy and the analyst's ethical responsibilities. Training on data integrity procedures and SOPs are conducted by the individual departments' group leaders within the laboratory. All records of training are retained at the laboratory in the individual staff training folders and are maintained by the laboratory quality assurance officer. All information related to staff qualifications, experience, external training courses, and education are placed into the individuals training file. Verification documentation for laboratory orientation, health and safety, and quality assurance training is also maintained with the training file. Additional training documentation is added to the files as it occurs. This includes data for initial and continuing demonstrations of proficiency, performance evaluations, study data and notes, and attendance lists from individual and group training sessions.	Internal	Svetlana Izosimova / Accutest Orlando Harry Behazadi / Accutest Orlando

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Laboratory Certifications	Accutest has current National Environmental Laboratories Accreditation Conference (NELAC), Army Corp validation and a Navy approval letter. Accutest will provide to Shaw copies of all current certifications prior to project start up.	Internal/External	Svetlana Izosimova / Accutest Orlando Harry Behazadi / Accutest Orlando Natasha Sullivan / Shaw E&I
Field Logbooks	The sample number will be traceable to the site, location, and depth (where applicable). The sample identification and description will be recorded by the Site Supervisor or representative in the sample collection logs. Site Supervisor will perform daily reviews of field log books each day of sampling.	Internal	Cano Hernandez / Shaw E&I
Sample Location Verification	The site supervisor will verify that the sample technicians have collected the samples from the proper locations and depths as described in worksheet 18.	Internal	Cano Hernandez / Shaw E&I
Chain-of-Custody-*Field Level*	Shaw Program Chemist will generate electronic COCs forms prior to field sampling in accordance to the sample matrices and analytical tests required as described in worksheet 19. Upon, completion of the COCs forms by the field technicians and prior to placement in the cooler the Site supervisor will review the COCs against the field logbooks, worksheet 18 and worksheet 19 to insure that the samples, sample volumes, and sample nomenclature match the COC forms and the required analytical tests have been notated. A review of the COC form for completeness will also be conducted.	Internal	Cano Hernandez / Shaw E&I Natasha Sullivan / Shaw E&I
Chain-of-Custody-*Shaw Chemist*	Upon, completion of the COC the field technician will either fax or email the completed COC form to the Shaw Program Chemist. A review of the COC form against worksheet 18 and worksheet 19 will be conducted to ensure proper analytical test.	Internal	Natasha Sullivan / Shaw E&I

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody-*Analytical Laboratory*	All samples to be analyzed by the fixed-base laboratory will be shipped via overnight courier service. Upon receipt, a representative of the laboratory shall check the integrity of the custody seals, then locate, sign, and date the COC. The laboratory is responsible for verifying that the COC and containers are in agreement. The COC, a Cooler Receipt Form, and information regarding any discrepancies between the COC and bottle labels will be faxed to the Shaw Program Chemist prior to preparation for analysis. The Laboratory Information Management System will provide evidence of sample custody from receipt by the laboratory until appropriate disposal.	Internal	Aaron David / Accutest Orlando Sample Management Technicians / Accutest Orlando
LIMs Login – Analytical Laboratory	A review of the COC form against the laboratory LIMs login and the project analytical requirement as contained within worksheet 19 will be conducted to ensure proper analytical tests have been assigned and a review of the login for correctness will be conducted.	Internal	Heather Wandrey / Accutest Orlando
LIMs Login –Shaw Chemist	A secondary review of the COC form against the laboratory LIMs login and the project analytical requirement as contained within worksheet 19 will be conducted to ensure proper analytical tests have been assigned and a review of the login for correctness will be conducted.	External	Natasha Sullivan / Shaw E&I
Sample Receipt Form – Shaw Chemist	Accutest will provide within 48 hours of receipt of samples a copy of the sample receipt form any discrepancies between the COC and the sample containers will be noted and contained as part of the analytical record.	External	Natasha Sullivan / Shaw E&I

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Laboratory Corrective Action and Report Procedure	Routine corrective action is defined as procedures used to return out of control analytical systems back to control. This level of corrective action applies to all analytical quality control parameters and analytical system specification as defined in the laboratory SOPs. Bench analysts have full responsibility and authority for performing routine corrective action. Routine corrective actions are documented as part of the analytical record. Defective processes, holding time violations, systematic errors and quality defects that occur are to be reported by the bench chemist immediately to the section supervisor and a non-conformance record initiated. The section supervisor will notify the designated Shaw Laboratory Project Manager (Heather Wandrey) who will then notify the Shaw Program Chemist (Natasha Sullivan). All notifications must be made in a timely manner. The non-conformance record should become part of the analytical record.	Internal/External	Heather Wandrey / Accutest Orlando Natasha Sullivan / Shaw E&I
Analytical Data Package – Laboratory	All data produced by the laboratory will be required to undergo several levels of review, which will include two levels of management review at the laboratory. The laboratory will review the data packages internally for completeness and verify that all of the required forms and raw data are included for each data package type. Random data packages may be chosen by the Accutest QA Officer for additional audits.	Internal	Harry Behazadi / Accutest Orlando Heather Wandrey / Accutest Orlando Svetlana Izosimova / Accutest Orlando
Analytical Data Package / Laboratory Quality Control – Shaw Chemist	The Shaw Program Chemist will verify that data has been received for all samples that have been sent to the laboratory. An evaluation of this data will be performed to determine whether the laboratory met the QC requirements for the analytical as stated in the analytical methods and laboratory SOPs. Refer to worksheets 19 and 28.	External	Natasha Sullivan / Shaw E&I

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Laboratory Electronic Data Deliverables	The laboratory will provide an electronic data deliverables in excel format that has been generated by the laboratory LIMs system. The Shaw Program Chemist will review these files for correctness and completeness. Project specific action goals as defined in worksheet 15 will be added and evaluated. Any quality control issues that may impact the data use will be evaluated. The project manager and site supervisor will be notified immediately of any samples that exceed the project action goals.	External	Natasha Sullivan / Shaw E&I

QAPP Worksheet #35
Validation (Steps IIa and IIb) Process Table

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field SOPs	Ensure that all sampling SOPs were followed.	Archie Vega, FSSI/Shaw
IIa	Analytical SOPs	Ensure that all laboratory analytical SOPs were followed.	Svetlana Izosimova, Accutest Orlando
IIa	Documentation of Method QC Results	Establish that all method quality control were analyzed for and in control as listed in the analytical SOPS. If the method QA was not in control, the laboratory will have contacted Shaw of non-conformant situation prior to report generation for guidance.	Natasha Sullivan, FSSI/Shaw
IIa/IIb	Documentation of QAPP QC Samples Results	Establish that all QAPP required QC samples were collected. Establish that the collected QC samples met the required limits as established in the QAPP.	Archie Vega, FSSI/Shaw Natasha Sullivan, FSSI/Shaw
IIa/IIb	Documentation of Analytical Reports for Completeness	Ensure that from the Chain-of-Custody generated in the field to the delivery of the analytical data that the appropriate analytical samples have been collected, appropriate site identifications have been used, and the correct analytical methods have been applied. Review the analytical reports to establish that all required forms, case narratives, samples, Chains-of-Custody, logbooks, and raw data have been included.	Natasha Sullivan, FSSI/Shaw
IIb	Project Quantitation Limits	Review laboratory analytical results to ensure the project quantitation limits were met as specified in QAPP worksheet 15.	Natasha Sullivan, FSSI/Shaw
IIa/IIb	Project Action Limits	Review and add project action limits to the laboratory electronic data deliverable. Flag results and notify project manager of samples that exceed the project action limits as listed in QAPP worksheet 15.	Natasha Sullivan, FSSI/Shaw
IIa/IIb	Data Validation	Full data validation will be performed on all samples found except TCLP, RCI, and TPH, when they are being used for Waste Characterization. Those Samples will go through a data verification process instead. Data will be validated in accordance with criteria as specified in QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers will be consistent with the USEPA Region II Validation SOPs where available; else, the USEPA National Functional Guidelines for Organic Data Review (October, 1999) and the National Functional Guidelines for Inorganic Data Review (October, 2004) will be used.	Eric Malarek, FSSI/Shaw

QAPP Worksheet #36 (UFP-QAPP Manual Section 5.2.2) -- Validation (Steps IIa and IIb) Summary Table

Identify the matrices, analytical groups, and concentration levels that each entity performing validation will be responsible for, as well as criteria that will be used to validate those data.

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Soil	TPH GRO	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Soil	TCL VOC	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SOP for the Validation of Organic Data Acquired Using SW-846 Method 8260B (SOP HW-24, June 1999).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Soil	TCL SVOC	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8270C (SOP HW-22, June 2001).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Soil	Explosives	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8330 Nitroaromatics and Nitroamines by HPLC (SOP HW HW-16, September 1994).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Soil	Pesticides	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8080A/8000A (SOP HW-23, April 1995).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Soil	PCB's	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8082 (SOP HW-23B, May 2002).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Soil	TPH DRO	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Soil	TAL Metals – ICP	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II Evaluation of Metals Data for the Contract Laboratory Program (SOP HW-2, January 1992).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Soil	Metals – Mercury	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II Evaluation of Metals Data for the Contract Laboratory Program (SOP HW-2, January 1992).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Sediment	TPH GRO	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Sediment	TCL VOC	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SOP for the Validation of Organic Data Acquired Using SW-846 Method 8260B (SOP HW-24, June 1999).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Sediment	TCL SVOC	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8270C (SOP HW-22, June 2001).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Sediment	Explosives	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8330 Nitroaromatics and Nitroamines by HPLC (SOP HW HW-16, September 1994).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Sediment	Pesticides	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8080A/8000A (SOP HW-23, April 1995).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Sediment	PCB's	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8082 (SOP HW-23B, May 2002).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Sediment	TPH DRO	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II Modifications to the National Functional Guidelines for Organic Data Review (September, 1994)	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Sediment	TAL Metals – ICP	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II Evaluation of Metals Data for the Contract Laboratory Program (SOP HW-2, January 1992).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Sediment	Metals – Mercury	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II Evaluation of Metals Data for the Contract Laboratory Program (SOP HW-2, January 1992).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Blanks / Water	TPH GRO	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Blanks / Water	TCL VOC	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SOP for the Validation of Organic Data Acquired Using SW-846 Method 8260B (SOP HW-24, June 1999).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Blanks / Water	TCL SVOC	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8270C (SOP HW-22, June 2001).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Blanks / Water	Explosives	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8330 Nitroaromatics and Nitroamines by HPLC (SOP HW HW-16, September 1994).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Blanks / Water	Pesticides	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8080A/8000A (SOP HW-23, April 1995).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Blanks / Water	PCB's	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II SW-846 Method 8082 (SOP HW-23B, May 2002).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Blanks / Water	TPH DRO	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Blanks / Water	TAL Metals – ICP	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II Evaluation of Metals Data for the Contract Laboratory Program (SOP HW-2, January 1992).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Blanks / Water	Metals – Mercury	Low	Project Validation Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology. Validation Qualifiers applied as per USEPA Region II Evaluation of Metals Data for the Contract Laboratory Program (SOP HW-2, January 1992).	Natasha Sullivan, Shaw E&I, Inc. Eric Malarek, FSSI/Shaw
IIa/IIb	Disposal Soil and Sediments	TCLP Volatiles	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	TCLP Semivolatiles	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	TCLP Pesticide	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	TCLP Herbicides	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	TCLP Metals ICP	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	TCLP Metals Hg	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	Cyanide, Total	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	Sulfide, Total	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	Ignitability (Flashpoint)	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Disposal Soil and Sediments	Corrosivity as pH	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	Total PCB's	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	TPH DRO	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.
IIa/IIb	Disposal Soil and Sediments	TPH GRO	Low	Project Criteria as per QAPP worksheets 12, 15, 19, and 28 and cited USEPA SW-846 methodology.	Natasha Sullivan, Shaw E&I, Inc.

QAPP Worksheet #37 (UFP-QAPP Manual Section 5.2.3) -- Usability Assessment

Describe the procedures / methods / activities that will be used to determine whether data are of the right type, quality, and quantity to support environmental decision-making for the project. Describe how data quality issues will be addressed and how limitations on the use of the data will be handled.

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:
It is the joint responsibility of the project team listed in this QAPP to ensure that the data collected meets the requirements listed in this QAPP.

Describe the evaluative procedures used to assess overall measurement error associated with the project:
The data sets will be validated in accordance with the QAPP, the analytical methods, national EPA and Region II data validation guidance to assess conformance with the quality control. Usability is not limited to data validation and includes review and assessment of the procedures as defined in the QAPP these will be monitored by the project team throughout the project. The data validation reports, qualifiers applied to data in conjunction with this QAPP, field logs books, progress reports, and corrective action reports will be used to assess overall usability as it applies to data sets. Project completeness will be addressed in the project close out reports.

Identify the personnel responsible for performing the usability assessment:
Various members of the project team as listed in this QAPP are responsible for the ensuring the usability of the data sets as defined in this QAPP.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

During the project close out process the risk assessments performed using the post-excavation confirmatory samples will determine whether the site can be proposed for unrestricted land use. If the results of the risk assessments suggest unrestricted land use is not appropriate, but that land use in accordance with the intended future use of the land is appropriate, the Navy may opt to deem the removal action complete and summarize the removal action and associated risk assessment findings, conclusions, and recommendations in the construction completion report. If, alternatively, the residual risk assessments indicate land use in accordance with the intended future use is not appropriate or if the Navy decides an unrestricted land use determination is possible if some isolated areas of contamination are removed, additional excavation may be performed. In this circumstance, additional confirmatory samples will be collected in the areas of additional excavation in accordance with the protocol discussed above. The data from these additional confirmatory samples will replace previous confirmatory data from those areas that were additionally excavated, and the HHRA and ERA will be revised accordingly

ANALYSIS OF NITROAROMATICS AND NITRAMINES BY HPLC

Prepared by: Norm Farmer Date: 03/06/06

Reviewed by: Stephanie Weber Date: 03/06/06

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Document Control

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TEST NAME: ANALYSIS OF NITROAROMATICS AND NITRAMINES BY HPLC

METHOD REFERENCE: SW846 8330A

DEPT: GC

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of specific nitroaromatics and nitramines in water and solid matrices utilizing an HPLC equipped with a diode array detector.

1.1.2 The following compounds can be reported by this method:

Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
1,3,5-Trinitrobenzene
1,3-Dinitrobenzene
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
Nitrobenzene
2,4,6-Trinitrotoluene
4-Amino-2,6-dinitrotoluene
2-Amino-4,6-dintirotoluene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
2-Nitrotoluene
3-Nitrotoluene
4-Nitrotoluene
TNX
DNX
MNX

1.1.3 The reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. Reporting limits may vary depending on matrix complications and volumes. Reporting limits for this method are in the range of 0.2 to 0.5 ug/l for extracted aqueous samples, 50 ug/l for direct injection aqueous samples, and 200 to 500 ug/kg for solid samples.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

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1.1.5 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

1.2 Summary

1.2.1 This method is adapted from SW846 Method 8330A.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with Accutest SOP OP018 and OP019.

1.2.4 The extracts are analyzed on an HPLC equipped with a diode array detector.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples.

2.1.2 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be stored at 4°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts should be analyzed as soon as possible, but must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. All of these materials must be demonstrated to be free from interferences.

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3.3 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain tetryl should be diluted with acetonitrile prior to filtration and acidified to pH < 3. Samples and extracts should not be exposed to temperatures above room temperature.

3.4 The nitrotoluenes co-elute on the CN column. Positive results must be reported from the C-18 or RP column.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

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4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Water – HPLC grade or equivalent

5.2 Acetonitrile – HPLC grade or equivalent

5.3 Methanol – HPLC grade or equivalent

5.4 Calcium chloride solution – prepared by dissolving 5 grams of calcium chloride in 1 liter of HLPC grade water.

5.5 Explosives stock standards – Traceable to Certificate of Analysis

5.6 Surrogate standards – 3,4-Dinitrotoluene

6.0 APPARATUS

6.1 HPLC – Agilent Technologies 1100 or HP 1050

Suitable HPLC equipped with an autosampler, pump, and diode array detector.

Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Data System – Agilent Technologies LC Chemstation rev. 06.00 or rev. 08.00
Agilent Technologies MS Chemstation rev. BA 02.00

6.2.1 A computer system interfaced to the HPLC that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

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6.2.2 The software should allow for the viewing of the entire UV Spectra acquired over the analytical run. Comparisons can then be made between spectra from standards and samples.

6.2.3 Data is archived to magnetic tape for long term storage.

6.3 Primary Column (C-18) – Phenomenex Ultracarb ODS 5u – 4.6mm X 250mm or equivalent

6.4 Confirmation Column (amine bonded C-18) – Zorbax Bonus RP 5u - 4.6mm X 250mm or equivalent

6.5 Alternate Column (CN) – Phenomenex Luna CN 5u – 4.6mm X 250mm or equivalent

6.6 Suitable syringes, syringe filters, and glassware for dilutions of standards and extracts.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards should be stored at 4 °C, or as recommended by the manufacturer.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a “Certificate of Analysis” with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor’s expiration date. Once opened, the hold time is reduced to one year or the vendor’s expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with acetonitrile. The hold time for intermediate standards is six months or the vendor’s expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicate analyte degradation or concentration changes.

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7.1.3 Calibration Standards

Calibration standards for the explosives are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. Calibration standards are prepared in 75/25 (v/v) water/acetonitrile. The low standard is at a concentration at or below the RL and the remaining standards define the working range of the detector.

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 HPLC Conditions

7.2.1 HPLC Conditions - Primary Column – (C-18)

100ul autosampler injection

Mobile phase – Isocratic 47% water 53% methanol

Column temperature – 27.0 °C

Flow – 0.9 ml/min

Diode Array Detector – Set to acquire and process data at 254-nm wavelengths using a 10-nm bandwidth. Secondary wavelength may be set to 214-nm or 235-nm. The 235-nm wavelength switches to 270-nm just prior to the elution of the nitrotoluenes. All data from 200-nm to 450-nm wavelengths is stored for spectral evaluation.

HPLC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.2.2 HPLC Conditions - Confirmation Column – (amine bonded C-18)

100 ul autosampler injection

Mobile phase – Isocratic 47% water, 53% methanol

Column temperature – 24.0 °C

Flow – 0.9 ml/min

Diode Array Detector – Set to acquire and process data at 254-nm wavelengths using a 10-nm bandwidth. Secondary wavelength may be set to 214-nm or 235-nm. All data from 200-nm to 450-nm wavelengths is stored for spectral evaluation.

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HPLC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.2.3 HPLC Conditions - Alternate Column – (CN)

100 ul autosampler injection

Gradient program –

Water (%)	MeOH (%)	ACN (%)	Flow (ml/min)	Time (min)
78	3	19	1.1	0
78	3	19	1.1	28
0	0	100	1.65	30
0	0	100	1.25	35
78	3	19	1.1	36
78	3	19	1.1	43

Column temperature – 23.0 °C

Diode Array Detector – Set to acquire and process data at 254-nm wavelengths using a 10-nm bandwidth. Secondary wavelength may be set to 214-nm or 235-nm. All data from 200-nm to 450-nm wavelengths is stored for spectral evaluation.

HPLC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3. Sample Preparation

7.3.1 Water Samples (extracted)

A 1000ml aliquot of sample is extracted utilizing a solid phase extraction cartridge. The cartridge is eluted with 3ml of acetonitrile. The final volume is then adjusted to 10ml with reagent water.

7.3.2 Water Samples (direct inject)

A 5ml aliquot of sample is mixed with 5ml of acetonitrile. The extract is filtered through a .45um Teflon syringe filter to remove any particulate.

7.3.3 Solid Samples

A 2-gram aliquot of sample is extracted with 8ml of acetonitrile utilizing a chilled ultrasonic bath. A 2ml aliquot of extract is mixed with 3ml of calcium chloride solution. The extract is filtered through a .45um Teflon syringe filter to remove any particulate.

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7.4. HPLC Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, the HPLC system must be calibrated, and retention time windows must be determined.

7.4.1.1 External Standard Calibration

A minimum 5-point calibration curve is created for the explosives and surrogates.

Calibration factors (CF) for the explosives and surrogates are determined at each concentration by dividing the area of each compound by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the response factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively if the %RSD > 20% a calibration curve of response vs. amount can be plotted. If the correlation coefficient (r) is $\geq$0.995 ($r^2 \geq$0.990) then the curve can be used to quantitate target analytes in the samples.

7.4.1.2 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV should be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be $\leq$ 15%. If the ICV does not meet this criteria, a second standard should be prepared. If the ICV still does not meet criteria, analyze an ICV prepared from a third source. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, determine which two standards agree. Make fresh

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calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

NOTE: Second source standards may not be available for TNX, DNX, and MNX.

7.4.1.3 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations. HPLC retention times tend to shift more than GC retention times.

The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. The analyst should monitor the retention

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times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential "hits". The analyst will then review these "hits" and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

7.4.2 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the explosives are prepared at various concentrations; at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally a CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| should be ≤ 15% for each analyte. If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated.

If the |%D| is greater than 15%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported without qualification. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed or the data must be qualified.

7.4.3 Sample Extract Analysis

7.4.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Initial Calibration Standards (or Initial CCV)
QC Extracts
Sample Extracts
CCV Standards

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7.4.3.2 One hundred microliters (same amount as standards) of extract is injected into the HPLC by the autosampler. The data system then records the resultant peak responses and retention times.

7.4.3.3 Tentative identification of an analyte occurs when the peaks from the sample extract fall within the established retention time windows for a calibrated compound.

7.4.3.4 The diode array detector is capable of spectral evaluation; second column confirmation may not be necessary in some instances. Peak spectra can be compared to a spectral library that has been created for the target analytes. However, the UV spectra for some of the analytes are not very unique. **If there is any doubt to the presence of a compound, it must be confirmed on the confirmation column.**

7.4.3.5 If the peaks of interest fall within the retention time windows on the confirmation column, the identification is confirmed. Quantitation of the analyte on the primary and confirmation column should agree within 40%. If the difference is greater than 40% and no obvious reason can be found, the higher result should be reported and flagged as “estimated”; otherwise, the result from the primary column should be reported.

7.4.3.6 If the compound identification does not confirm, then the result should be reported as ND or “U”.

7.4.3.7 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.3.8 If peak identification is prevented by the presence of interferences, further cleanup may be required or the extract must be diluted so that the interference does not mask any analytes. Analysis on the confirmation column may also be beneficial.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

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Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Surrogates

9.1.1 3,4-Dinitrotoluene is used as the surrogate standard to monitor the efficiency of the extraction.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for the results to be acceptable.

9.1.2 If the surrogate recovery is not within the established control limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, integrations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly.

9.1.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, re-extract and reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary. If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

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9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis should be reported on the final report.

9.2 Method Blank

9.2.1 The method blank is either de-ionized water or cleaned sand (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high level samples. The method blank must be free of any analytes of interest or interferences at ½ the required reporting level to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination should be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination should be investigated and documented. The samples should be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is either de-ionized water or cleaned sand (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

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The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable. The large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of the established control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary.

Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A marginal exceedance in the Blank Spike is defined as a recovery being outside of 3 standard deviations but within 4 standard deviations of the mean.

The number of allowable marginal exceedances is based on the number of analytes in the Blank Spike. Marginal Exceedances must be random. If the same analyte exceeds the BS control limits repeatedly, it is an indication of a systematic problem and corrective action must be taken.

The number of allowable marginal exceedances is as follows:

1) 11-30 analytes in BS, 1 analyte allowed in ME range;

2) < 11 analytes in BS, no analytes allowed in ME range

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.3.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary and the sample results can be reported without qualification.

9.3.2.4 If the recovery of an analyte in the BS is high and the associated sample is non-detect, then the sample results can be reported without qualification.

9.3.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

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9.3.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4 Matrix Spike and Matrix Spike Duplicate

9.4.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.4.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.4.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.4.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are an indication of the sample matrix effects.

9.4.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If more than 33% of the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the

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control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

10.0 CALCULATIONS

The concentration of each explosive in the original sample is calculated as follows:

Water (ug/l) = $(CONC_{inst}) \times (V_F / V_I) \times DF$

Soil (ug/kg) = $[(CONC_{inst}) \times (V_F / W_I) \times DF]$

$CONC_{inst}$	=	Instrument concentration calculated from the initial calibration using mean CF, linear curve, or quadratic curve
DF	=	Dilution Factor
V_F	=	Volume of final extract (ml)
V_I	=	Volume of sample extracted (ml)
W_I	=	Weight of sample extracted (g)

Soils are air dried prior to extraction; therefore, %solids is not used in the calculation.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst should follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment should be used by all analysts.

11.2 Pollution Prevention

Wastewater, methanol, and acetonitrile from the instrument are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

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12.0 REFERENCES

SW846 Method 8000B Revision 2, December 1996

SW846 Method 8000C Revision 3, March 2003

SW846 Method 8330 Revision 0, September 1994

SW846 Method 8330A Revision 1, January 1998

SW846 Method 3535A Draft Revision 1A, November 1998

SW846 Method 8332 Revision 0, December 1996

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STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF NITROAROMATICS AND NITRAMINES (EXPLOSIVES) FROM WATER SAMPLES FOR HPLC ANALYSIS

Prepared by: Norm Farmer Date: 03/10/06

Reviewed by: Stephanie Weber Date: 03/10/06

Annual Review

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TEST NAME: **STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF NITROAROMATICS AND NITRAMINES (EXPLOSIVES) FROM WATER SAMPLES FOR HPLC ANALYSIS**

Method: SW846 3535A/8330A

Dept: OP

1.0 Summary, Scope and Application

1.1 Summary

Aqueous samples are extracted using solid-phase extraction cartridges, eluted with acetonitrile, and stored in amber glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to aqueous samples submitted for explosives analysis by HPLC method SW-846 8330A.

2.0 Discussion and Comments

This procedure is adapted from SW-846 methods 8330A, 3500B and 3535A. The method utilizes “solid-phase extraction”; it is not applicable to samples requiring the “salting out” procedure. Samples expected to contain high levels of explosives should be screened using the direct injection technique.

Sample particulates may clog the solid-phase media and result in extremely slow sample extractions. Use of the appropriate filter aid may shorten the extractions without loss of analytes. Even when a filter aid is employed, this method may not be appropriate for samples high levels of suspended solids. It may be necessary to use smaller sample volumes with these types of samples. This should be noted on the sample prep sheet.

The HPLC detector is extremely sensitive and will respond to many organic compounds. It is important to minimize extraneous contaminants and carryover by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass, stainless steel, or Teflon.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.0 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples.

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3.1.1 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be refrigerated at 4°C until analysis.

3.2 Holding Time

3.2.0 Aqueous samples must be extracted within 7 days of collection. A sample is considered extracted after the methylene chloride aliquots have been combined in the Erlenmeyer flask.

3.2.1 Extracts must be analyzed within 40 days of extraction.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

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4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Acetonitrile – HPLC grade or equivalent

5.2 Methanol – HPLC grade or equivalent

5.3 RDX SPE Cartridges – Restek Corporation or equivalent

5.4 Reagent water – HPLC grade or equivalent - free of interferences

5.5 Explosives Surrogate Solution – prepared in acetonitrile at a concentration specified by the HPLC analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.6 Explosives Spike Solution – prepared in acetonitrile at a concentration specified by the HPLC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

6.0 Glassware and Apparatus

6.1 Solid-phase cartridge extraction system – suitable for use with extraction cartridges

6.2 Vacuum pump

6.3 Connector with Teflon intake lines

6.4 10.0ml Receiver tubes

6.5 1000ml graduated cylinder

6.6 0.7 um glass fiber filters

6.7 0.25ml, 0.5ml or 1.0ml syringes

6.8 Disposable transfer pipette

6.9 2.0ml amber glass screw cap vials – caps must have Teflon lined septa

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7.0 Procedure

7.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: sample ID, bottle number, initial volume, final volume, pHs, solvent lot numbers, spike and surrogate lot numbers, batch numbers, and dates. The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the GC analyst with the extracts.

7.2 Assemble the solid-phase extraction system. Label the side of each cartridge with the sample ID.

7.3 Wash each cartridge with 10 to 15 ml of acetonitrile. Use gravity flow if possible or a 1 to 2 ml/min flow rate. This will have to be done in 5ml aliquots. Do not allow the cartridge to go dry.

7.4 When only a small layer of acetonitrile remains above the sorbent bed, add 20 to 30 ml of reagent water. This will have to be done in 5ml aliquots. Do not allow the cartridge to go dry. Stop the flow just before the cartridge goes dry.

7.5 Fill each cartridge with about 5ml of reagent water.

7.6 Attach a connector and Teflon intake line to each of the cartridges.

7.7 Mark the level of the sample on each of the bottles.

7.8 Use 1 liter amber bottles for the method blank (MB) and blank spike (BS). Fill each of these bottles with 1000ml of reagent water.

7.9 Use a separate bottle for the matrix spike (MS) and the matrix spike duplicate (MSD). If there is only one bottle for the MS/MSD, it can be split into two aliquots. This should be noted on the prep sheet. Record the sample ID, bottle number, and volume on the prep sheet.

7.10 Check the pH of each sample by dipping a clean glass-stirring rod into the sample and touching it to the pH paper. Record the pH on the prep sheet. Samples for Explosives analysis should be extracted at the pH at which they were received.

7.11 Using the dedicated surrogate syringe add **0.25ml** of surrogate solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

7.12 Using the dedicated spike syringe add **0.25ml** of spike solution to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

7.13 Place the intake lines in the appropriate sample bottles.

7.14 Turn on the vacuum and draw the sample through the cartridge at a rate of about 10 ml/min, until the entire sample has passed through the cartridge. As

particulate clogs the cartridge, increase the vacuum to maintain a reasonable flow rate.

7.15 Once the entire sample has been pulled through the cartridge shut off the vacuum and remove the connector. Add 5ml of reagent water to the cartridge and turn on the vacuum. Draw the reagent water through the cartridge. Shut off the vacuum once the water has passed through the cartridge.

7.16 Fill each sample bottle to the sample mark with tap water. Transfer the water to a 1000ml graduated cylinder and record the sample volume. Discard the tap water.

7.17 Place an appropriately labeled 10.0ml receiver tube under each cartridge.

7.18 Add 3 to 4 ml of acetonitrile to each cartridge and allow it to pass through the cartridge under gravity flow. A slight vacuum may need to be applied if the cartridge media is clogged.

7.19 Adjust the volume to 10.0ml with reagent water and mix thoroughly.

7.20 Transfer the extracts to appropriately labeled amber 2ml screw cap vial. Store the extracts in the “extract refrigerator” until they are needed for analysis.

8.0 Quality Assurance and Quality Control

8.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

8.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

9.0 Safety and Waste Disposal

9.1 Safety

9.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

9.1.2 Material Safety Data Sheets (MSDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS prior to using any new reagents or solvents.

9.1.3 Acetonitrile is an inhalation hazard and suspected carcinogen. Fume hoods must be used to minimize exposure to vapors.

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9.2 Waste Disposal

9.2.1 Waste acetonitrile is placed in the “non-chlorinated waste” container.

9.2.2 Spent solid-phase extraction cartridges may be disposed of in the trash.

9.2.3 Extracted water samples are rinsed down the drain with large amounts of water.

10.0 References

10.1 SW-846 Method 3535A, Draft Rev. 1A, 11/98

10.2 SW-846 Method 8330A, Rev. 1, 11/98

10.3 SW-846 Method 8332, Rev. 0, 12/96

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STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF NITROAROMATICS AND NITRAMINES (EXPLOSIVES) FROM SOLID SAMPLES FOR HPLC ANALYSIS

Prepared by: Norm Farmer Date: 03/12/06

Reviewed by: Stephanie Weber Date: 03/12/06

Annual Review

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TEST NAME: **STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF OF NITROAROMATICS AND NITRAMINES (EXPLOSIVES) FROM SOLID SAMPLES FOR HPLC ANALYSIS**

Method: SW846 8330A

Dept: OP

1.0 Summary, Scope and Application

1.1 Summary

Solid samples are extracted with acetonitrile in a chilled ultrasonic bath. The extracts are filtered and stored in amber glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to solid samples, including soils and sediments, submitted for Explosives analysis by HPLC method SW-846 8330A.

2.0 Discussion and Comments

This procedure is adapted from SW-846 method 8330A. The method outlined in this SOP is designed for low and high concentration samples. Samples expected to contain high levels of explosives should be screened using method 8510 or 8515. If the samples contain more than 2% explosives, they should not be ground by mortar and pestle.

The HPLC detector is extremely sensitive and will respond to many organic compounds. It is important to minimize extraneous contaminants and carryover by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass, stainless steel, wood, or Teflon.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.0 Samples shall be collected in amber glass jars with Teflon lined caps. 250ml jars are recommended for solid samples.

3.1.1 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be refrigerated at 4°C until analysis.

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3.2 Holding Time

3.2.0 Solid samples must be extracted within 14 days of collection. A sample is considered extracted after it has been sonicated for a minimum of 8 hours.

3.2.1 Extracts should be analyzed as soon as possible, but must be analyzed within 40 days of extraction.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples that are extracted at the same time.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

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4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Acetonitrile – HPLC grade or equivalent

5.2 Methanol – HPLC grade or equivalent

5.3 Calcium Chloride Solution – prepared by dissolving 5 grams of calcium chloride in one liter of HPLC grade water

5.4 Blank Sand – precleaned to remove contaminants

5.5 Explosives Surrogate Solution – prepared in acetonitrile at a concentration specified by the HPLC analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.6 Explosives Spike Solution – prepared in acetonitrile at a concentration specified by the HPLC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

6.0 Glassware and Apparatus

6.1 Aluminum weighing dishes or watch glasses

6.2 16ml amber screw top vials with Teflon lined caps

6.3 Spatula – stainless steel, wooden, or Teflon

6.4 0.5ml or 1.0ml syringes

6.5 Mortar and Pestle

6.6 10.0ml graduated pipette

6.7 Desiccator cabinet

6.8 2.0ml amber glass screw cap vials – caps must have Teflon lined septa

6.9 Ultrasonic water bath capable of running continuously

6.10 Chiller capable of maintaining the ultrasonic bath temperature at less than 20 ºC

6.11 Hi-Lo Thermometer

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6.12 Disposable 3.0ml syringes

6.13 0.45um Teflon syringe filters

6.14 Top loading balance – capable of weighing samples to +/- 0.01 grams

7.0 Procedure

7.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, and dates. The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the GC analyst with the extracts.

7.2 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. Discard the top layer of soil and thoroughly mix the sample with a wooden spatula. Samples with obvious layers may need to be transferred to a larger container for proper mixing. Refer to SOP QA035 for more information on sample homogenization.

7.3 Transfer 10 to 20 grams of each sample to the appropriately labeled weighing dish. Use a clean spatula for each sample. Record the sample ID and bottle number on the prep sheet.

7.4 Place the samples in the desiccator cabinet, and allow them to dry at room temperature. Samples must not be heated and should not be exposed to direct sunlight.

7.5 If needed, grind each sample in an acetonitrile rinsed mortar and pestle. Soil samples that are known to contain high concentrations or explosives should not be ground. They may DETONATE.

7.6 Transfer approximately 2.0 grams of each of the samples to the appropriately labeled amber 16ml vials. Be sure to label the vial cap, because the ultrasonic bath may remove any labels from the sides of the vials. Record the weight to the nearest 0.01 gram on the prep sheet.

7.7 Transfer approximately 2.0 grams of each of the QC samples to the appropriately labeled vials. This includes the method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD). Use 2.0 grams of clean sand for the MB and BS. Use additional 2.0 gram aliquots of a sample for the MS, MSD, and DUP. Record the sample ID, bottle number, and weight on the prep sheet.

7.8 Using the dedicated surrogate syringe add 0.5ml of surrogate solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

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7.9 Using the dedicated spike syringe add 0.5ml of spike solution to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

7.10 Using a graduated pipette, add **7.5ml** of acetonitrile or each of the sample vials, the method blank (MB) and sample duplicate (DUP). Add **7.0ml** of acetonitrile to the BS, MS, and MSD. This will result in **8.0ml** of acetonitrile in each of the vials.

7.11 Put the cap on each vial and shake briefly to mix.

7.12 Place the vials in the rack inside the chilled ultrasonic bath.

7.13 Place the Hi-Lo thermometer probe in the bath. Reset the temperature.

7.14 Sonicate the sample for a minimum of 8 hours, but not more than 18 hours.

7.15 After sonication, remove the vials from the bath and allow them to settle for 30 minutes.

7.16 Record the maximum temperature of the bath on the prep sheet. The temperature should not have exceeded 20 ^{o}C. Notify the analyst if this temperature was exceeded.

7.17 Using a disposable 3.0ml syringe, remove 2.0ml of supernatant and combine it with 3.0ml of calcium chloride solution in a 16ml vial. Shake and let stand for 15 minutes.

7.18 Transfer ~3ml of extract to disposable syringe. Attach a Teflon syringe filter to the disposable syringe.

7.19 Filter the extract into an appropriately labeled amber 2.0ml screw cap vial. Store the extracts in the “extract refrigerator” until they are needed for analysis. **WARNING:** Wear safety glasses, the extract may spray if the filter clogs.

7.20 Store the extracts in the “extract refrigerator” until they are needed for analysis.

8.0 Quality Assurance and Quality Control

8.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. Samples can not be added to the batch after the sonication procedure has started.

8.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

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9.0 Safety and Waste Disposal

9.1 Safety

9.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

9.1.2 Avoid grinding samples that may contain high levels of explosives. The grinding action may cause them to DETONATE.

9.1.3 **Hearing protection** must be worn while operating the ultrasonic bath. The high frequency could cause permanent hearing loss.

9.1.4 Avoid touching the ultrasonic bath while it is active. Contact may cause tissue damage.

9.1.5 Avoid touching the chiller probe. Contact may cause tissue damage.

9.1.6 Material Safety Data Sheets (MSDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS prior to using any new reagents or solvents.

9.1.7 Acetonitrile is an inhalation hazard and suspected carcinogen. Fume hoods must be used to minimize exposure to vapors.

9.2 Waste Disposal

9.2.1 Waste acetonitrile is placed in the “non-chlorinated waste” container.

9.2.2 Extracted soil samples and residual acetonitrile may be poured into the “non-chlorinated waste” container or the entire vial may be lab packed with the “extract waste”.

9.2.3 Waste soil from the homogenizing process should be place in the “soil waste” container. **Note:** Waste soil from foreign soils must follow “foreign soil” disposal requirements.

10.0 References

10.1 SW-846 Method 8330A, Rev. 1, 01/98

10.2 SW-846 Method 8332, Rev. 0, 12/96

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ANALYSIS OF SEMIVOLATILE ORGANICS BY GC/MS

Prepared by: Norm Farmer Date: 03/18/06

Reviewed by: Stephanie Weber Date: 03/18/06

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TEST NAME: ANALYSIS OF SEMIVOLATILE ORGANICS BY GC/MS

METHOD REFERENCE: SW846 8270C

DEPT: MS

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of various semivolatile organic compounds in water and solid matrices utilizing a gas chromatograph equipped with a mass spectrometer detector. Routine compounds can be found in Table 1.

1.1.2 Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. Reporting limits may vary depending on matrix complications and sample volumes. Reporting limits for this method are in the range of 5.0 ug/l for aqueous samples and 170 ug/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.3 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.1.4 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I” qualifier. Some program or project specifications may require that no values below the RL be reported.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8270C.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with Accutest SOP OP006 and OP007.

1.2.4 The extracts are analyzed on a gas chromatograph equipped with mass spectrometer detector.

1.2.5 The peaks detected are identified by comparison to characteristic ions and retention times specific to the known target list of compounds.

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1.2.6 Additional unknown peaks with a response > 12.5% of the closest internal standard may be processed through a library search with comparison to a database of approximately 98,000 spectra. An estimated concentration is quantitated by assuming a response factor of 1.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples.

2.1.2 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be refrigerated at –10°C to –20°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Interferences from phthalate esters can be eliminated by using plastic-free solvent containers and solvent rinsed glassware.

3.3 Other organic compounds, including chlorinated hydrocarbons, petroleum hydrocarbons, and phthalate esters may be coextracted by this method.

3.4 Aldol condensation byproducts may form when samples are extracted using solvent mixes containing acetone. Concrete samples in particular are prone to forming aldol condensation byproducts at levels that can interfere with the analysis and therefore should be extracted with 100% methylene chloride.

3.5 Benzidine and benzaldehyde are extremely reactive in the calibration mix and will quickly break down. If either analyte is detected in a sample it may be necessary to recalibrate the instrument using the individual analyte, and then reanalyze the sample.

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4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Internal Standards: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Internal standards for Mass Spec methods are often deuterated forms of target analytes. Internal standards are used to compensate for retention time and response shifts during an analytical run.

4.6 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

4.7 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

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4.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.12 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.13 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Methylene Chloride – pesticide grade or equivalent

5.2 Semivolatile stock standards – Various mixes, traceable to Certificate of Analysis

5.3 Decafluorotriphenylphosphine mix (DFTPP) – Also contains pentachlorophenol, benzidine and DDT.

5.4 Base/neutral surrogate standards –

Nitrobenzene-d5
2-Fluorobiphenyl
P-Terphenyl-d14

5.5 Acid surrogate standards –

Phenol-d6
2-Fluorophenol
2,4,6-Tribromophenol

5.6 Internal standards –

1,4-Dichlorobenzene-d4	Naphthalene-d8
Acenaphthene-d10	Phenanthrene-d10
Chrysene-d12	Perylene-d12

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6.0 APPARATUS

6.1 Gas Chromatograph – Agilent Technologies 6890 with 7683 Autosampler

6.1.1 Gas Chromatograph

The analytical system that is complete with a temperature programmable gas chromatograph and all required accessories, analytical columns, and gases.

6.1.2 The injection port is designed for split-splitless injection with capillary columns.

6.1.3 Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Mass Spectrometer – Agilent Technologies 5973 and 5975

The mass spectrometer must be capable of scanning from 35-500 amu every second or less utilizing a 70-volt (nominal) electron energy in the electron impact ionization mode. It must also be capable of producing a mass spectrum that meets all the criteria in section 7.4.1.1 when injecting 50 ng of Decafluorotriphenylphosphine (DFTPP).

6.3 Data System – Agilent Technologies MS Chemstation rev. BA 02.00 and DA 02.00

6.3.1 A computer system interfaced to the mass spectrometer that allows for the continuous acquisition and storage of all mass spectral data obtained throughout the duration of the chromatographic program.

6.3.2 The computer utilizes software that allows searching any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP).

6.3.3 The software should allow for integrating the abundances in any EICP between specific time or scan number limits.

6.3.4 The most recent version of the EPA/NIST mass spectral library should be available.

6.3.5 Data is archived to magnetic tape for long term storage.

6.4 DB-5MS Column or equivalent: 30m X 0.25mm X 0.25um

6.5 Suitable syringes and glassware for dilutions of standards and extracts.

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7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards should be stored at temperatures between –10 °C and –20 °C, or as recommended by the manufacturer.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to one year or the vendor's expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with methylene chloride. The hold time for intermediate standards is six months or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicate analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the semivolatile organics are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the RL and the remaining standards define the working range of the detector.

Benzidine and benzaldehyde are extremely reactive in the calibration mix and will quickly break down. Fresh standards may need to be made if these analytes are expected to be present in the samples.

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Gas Chromatograph Conditions

1ul or 2ul autosampler injection

Pulsed splitless or 4:1 split injection

Carrier gas – UHP Helium (1.0 to 1.7 ml/min ramped flow)

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Injection port temperature – 280 °C

Transfer line temperature – 280 °C

Source temperature – 230 °C

Quad temperature – 150 °C

Oven program – 40 °C for 2.0 minutes
35 °C/min to 130 °C for 0 minutes
12 °C/min to 320 °C for 1.4 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Sample Preparation

7.3.1 Water Samples

A 1000ml aliquot of sample is pH adjusted and extracted with methylene chloride utilizing separatory funnel extraction. The extract is concentrated to 1.0ml.

7.3.2 Solid Samples

A 30-gram aliquot of sample is extracted with methylene chloride and acetone utilizing pulse sonication. The extract is concentrated to 1.0ml.

7.4 Gas Chromatographic Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, the GC/MS system must be tuned, the injection port inertness must be verified, and the instrument must be calibrated.

7.4.1.1 Tune Verification (DFTPP)

The instrument should be hardware tuned per manufacturer's instructions. Verify the instrument tune by injecting 50ng of DFTPP solution onto the instrument. The resulting DFTPP spectra should meet the criteria in the following table.

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DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60 of mass 198
68	<2 % of mass 69
70	<2 % of mass 69
127	40-60 % of mass 198
197	<1 % of mass 198
198	Base peak, 100 % relative abundance
199	5-9 % of mass 198
275	10-30 % of mass 198
365	>1 % of mass 198
441	Present but less than mass 443
442	>40 % of mass 198
443	17-23 % of mass 442

Evaluate the tune spectrum using three mass scans from the chromatographic peak and a subtraction of instrument background. This procedure is performed automatically by the MS Chemstation software by running “autofind” on the DFTPP peak.

Select the scans at the peak apex and one to each side of the apex. Calculate an average of the mass abundances from the three scans.

Background subtraction is required. Select a single scan in the chromatogram that is absent of any interfering compound peak and no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tuning compound peak.

If the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are met.

Analysis must not begin until the tuning criteria are met. The injection time of the acceptable tune analysis is considered the start of the 12-hour clock. The same mass spec settings must be used for the calibration standards and samples that were used for the tune evaluation standard.

7.4.1.2 Injection Port Inertness Verification

DDT, pentachlorophenol, and benzidine must also be evaluated in the tune standard. These compounds are used to assess injection port inertness and column performance.

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Pentachlorophenol and benzidine should be present at their normal responses and, no peak tailing should be visible. The tailing factor for Benzidine must be less than 3 and the tailing factor for pentachlorophenol must be less than 5.

DDT breakdown should not exceed 20%. Breakdown is calculated as follows:

$$\%DDT_{BREAKDOWN} = \frac{(\text{DDE Area} + \text{DDD Area}) \times 100}{(\text{DDE Area} + \text{DDD Area} + \text{DDT Area})}$$

If degradation is excessive or peak tailing is noticed, injection port maintenance is required.

This performance test must be passed before any samples or standards are analyzed.

7.4.1.3 Internal Standard Calibration

A minimum 5-point calibration curve is created for the semivolatile organic compounds and surrogates using an internal standard technique. Accutest Laboratories routinely performs a 6-point calibration to maximize the calibration range.

The low point may be omitted from the calibration table for any compound with an RL set at the level two standard. Additionally the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

Response factors (RF) for each analyte are determined as follows:

$$RF = (A_{anayte} \mathbf{X} C_{istd})/(A_{istd} \mathbf{X} C_{analyte})$$

A_{anayte} = area of the analyte
A_{istd} = area of the internal standard
$C_{analyte}$ = concentration of the analyte
C_{istd} = concentration of the internal standard.

The mean RF and standard deviation of the RF are determined for each analyte. The percent relative standard deviation (%RSD) of the response factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of RF X 100) / Mean RF

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If the %RSD ≤ 15%, linearity through the origin can be assumed and the mean RF can be used to quantitate target analytes in the samples. Alternatively if the %RSD > 15% a calibration curve of response vs. amount can be plotted. If the correlation coefficient (r) is ≥0.995 (r^2 ≥0.990) then the curve can be used to quantitate target analytes in the samples.

Benzidine and benzaldehyde are extremely reactive in the calibration mix and will quickly break down. The initial calibration criteria for these two analytes has been increased to a %RSD ≤ 30% or a correlation coefficient (r) is ≥0.95 (r^2 ≥0.90) for determining non-detects. However, if either analyte is detected in the samples, the instrument must be recalibrated and the sample must be reanalyzed. It may be beneficial to recalibrate the instrument using the individual analyte, and then reanalyze the sample.

The method also employs a series of Calibration Check Compounds (CCC) and System Performance Check Compounds (SPCC). The %RSD for any CCC must be ≤ 30% and the average relative response factor for any SPCC must be at least 0.05. If the %RSD for any CCC is ≤ 30% but > 15% the compound should be evaluated by curve fit.

Calibration Check Compounds (CCC)

Base Neutral	**Acid**
1,4-Dichlorobenzene	Phenol
Hexachlorobutadiene	2,4-Dichlorophenol
Acenaphthene	2-Nitrophenol
Fluoranthene	p-Chloro-m-cresol
N-Nitrosodiphenylamine	2,4,6-Trichlorophenol
Di-n-octyl phthalate	Pentachlorophenol
Benzo (a) pyrene	

System Performance Check Compounds (SPCC)

Base Neutral	**Acid**
N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

7.4.1.4 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV should be prepared from a second source

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at a mid-range concentration. Because of compound interactions, the ICV for this method is prepared as two separate standards that cover the entire list of compounds.

The %D for all analytes of interest should be ≤ 25%. Benzidine and benzaldehyde must be ≤ 30%. If the %D > 25%, the analysis of samples may still proceed if the analyte failed high and the analyte is not expected to be present in the samples. However, if a reportable analyte is detected in a sample and the %D for that analyte was greater than 25% in the ICV, the sample will need to be reanalyzed on a system with a passing ICV for that analyte.

If the ICV does not meet this criteria, a second standard should be prepared. If the ICV still does not meet criteria, analyze an ICV prepared from a third source. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.4.2 Continuing Calibration Verification (CCV)

7.4.2.1 Inject 1ul of the tune evaluation mix at the beginning of each 12-hour shift. Evaluate the resultant peaks against the criteria in sections 7.4.1.1 and 7.4.1.2. The injection time of this standard starts the 12-hour window.

7.4.2.2. Analyze a continuing calibration check standard. Since the method only requires one CCV per analytical batch, the level of the CCV should be varied throughout the week. At least one CCV must be below the mid-point of the calibration curve.

7.4.2.3. The minimum RF of check standard for SPCC compounds must be 0.05.

7.4.2.4. The %D for the CCC compounds must be ≤ 20%. If the CCCs are not part of the target list, then all target analytes must meet the 20 %D criteria.

7.4.2.5. The %D for all other analytes of interest should be ≤ 20%. Benzidine and benzaldehyde must be ≤ 25%. If the %D > 20%, the analysis of samples may still proceed if the analyte failed high and the analyte is not expected to be present in the samples. However, if a reportable analyte is detected in a sample and the %D for that analyte was greater than 20% in the CCV, the sample will need to be reanalyzed on a system with a passing CCV for that analyte, or the data must be qualified.

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7.4.2.6. The criteria in 7.4.2.3 and 7.4.2.4 must be met for the continuing calibration to be considered valid. Additionally, only analytes that are being reported for a given sample must meet the criteria in 7.4.2.5. If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated.

7.4.2.7. If any of the internal standard area change by a factor of two (-50% to +100%) or retention time changes by more than 30 seconds from the midpoint standard of the last initial calibration, the mass spectrometer must be inspected for malfunctions and corrections made, as appropriate. Corrective action may include re-calibration (initial Calibration) of the instrument.

7.4.3 Sample Extract Analysis

7.4.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Tune Evaluation Mix
Initial Calibration Standards (or CCV)
QC Extracts
Sample Extracts

7.4.3.2 Four microliters of internal standard solution is added to every 100ul of extract in the autosampler vial. Generally, 400ul of extract are transferred to the autosampler vial with a gas tight syringe.

7.4.3.3 One or two microliters (same amount as standards) of extract is injected into the GC by the autosampler. The data system then records the resultant peak responses and retention times.

7.4.3.4 Qualitative identification

The target compounds shall be identified by analysts with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. The criteria required for a positive identification is:

The sample component must elute at the same relative retention time (RRT) as the daily standard. The RRT of sample component must be within ± 0.06 RRT units of the standard.

All ions present in the standard mass spectra at a relative intensity greater than 10% (major abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

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The relative intensities of these ions must agree within ± 30% between the daily standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%.)

Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

If peak identification is prevented by the presence of interferences, further cleanup may be required or the extract must be diluted so that the interference does not mask any analytes.

7.4.3.5 Quantitative analysis

When a target compound has been identified, concentration will be based on the integrated area of the quantitation ion, which is normally the base peak.

The sample matrix may produce an interference with the primary ion. This may be characterized by an excessive background signal of the same ion, which distorts the peak shape beyond a definitive integration. The interference could also, severely inhibit the response of the internal standard ion. If an interference is apparent the secondary ion can be used to generate a new calibration factor.

If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.3.6 Library search for tentatively identified compounds

If a library search is requested, the analyst should perform a forward library search of the NIST mass spectral library to tentatively identify 10 to 20 non-target compounds.

Guidelines for making tentative identification are:

These compounds should have a response greater than 12.5% of the nearest internal standard. The response is obtained from the Total Ion Chromatogram.

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The search is to include a spectral printout of the best library match for a particular substance. The results are to be interpreted by the analyst.

Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

The relative intensities the major ions should agree within ±20%.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be verified by performing further manual background subtraction to eliminate the interference created by coeluting peaks and/or matrix interference.

Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 from the nearest internal standard and is to be tabulated on the library search summary data sheet.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

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9.1 Internal Standards

1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12 and Perylene-d12 are used as internal standards for this method. The response of the internal standard in all subsequent runs should be within a factor of two (-50% to +100%) of the internal standard response in the opening CCV for each sequence. On days that an initial calibration is performed, the internal standard responses should be compared to the internal standard responses for the mid-point standard.

9.1.1 If the internal standard responses are not within limits, the following are required.

9.1.1.1 Check to be sure that there are no errors in calculations, integrations, or internal standards solutions. If errors are found, recalculate the data accordingly.

9.1.1.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.1.3 If no problem is found, prepare a second aliquot of extract and reanalyze the sample.

9.1.1.4 If upon reanalysis, the responses are still not within limits, the problem is considered matrix interference. The extract may need to be diluted or the results qualified.

9.2 Surrogates

9.2.1 Nitrobenzene-d5, 2-Fluorobiphenyl, and p-Terphenyl are used as the base neutral surrogate standards and Phenol-d5, 2-Fluorophenol, and 2,4,6-Tribromophenol are used as the acid surrogate standards to monitor the efficiency of the extraction.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for all surrogates for the results to be acceptable.

9.2.2 If any surrogate recovery is not within the established control limits, the following are required. Note: If the samples are being analyzed for only

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base neutral compounds or only acid compounds, then only the relative surrogates need to be monitored.

9.2.2.1 Check to be sure that there are no errors in calculations, integrations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly.

9.2.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.2.2.3 If no problem is found, re-extract and reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary. If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report

9.2.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis should be reported on the final report.

9.3 Method Blank

9.3.1 The method blank is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high level samples. The method blank must be free of any analytes of interest or interferences at ½ the required reporting level to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.3.2 If the MB is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

9.3.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination should be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

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9.3.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination should be investigated and documented. The samples should be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4 Blank Spike

9.4.1 The blank spike is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable. The large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of the established control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary.

Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A marginal exceedance in the Blank Spike is defined as a recovery being outside of 3 standard deviations but within 4 standard deviations of the mean.

The number of allowable marginal exceedances is based on the number of analytes in the Blank Spike. Marginal Exceedances must be random. If the same analyte exceeds the BS control limits repeatedly, it is an indication of a systematic problem and corrective action must be taken.

The number of allowable marginal exceedances is as follows:

1) > 90 analytes in BS, 5 analytes allowed in ME range;

2) 71-90 analytes in BS, 4 analytes allowed in ME range;

3) 51-70 analytes in BS, 3 analytes allowed in ME range;

4) 31-50 analytes in BS, 2 analytes allowed in ME range;

5) 11-30 analytes in BS, 1 analyte allowed in ME range;

6) < 11 analytes in BS, no analytes allowed in ME range

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9.4.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.4.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.4.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary and the sample results can be reported without qualification.

9.4.2.4 If the recovery of an analyte in the BS is high and the associated sample is non-detect, then the sample results can be reported without qualification.

9.4.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.4.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4.2.7 Because of their problematic nature, benzidine, benzaldehyde, and benzoic acid are generally not evaluated in the blank spike unless they are of specific concern for a given project.

9.5 Matrix Spike and Matrix Spike Duplicate

9.5.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

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The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.5.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.5.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.5.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.5.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extraction but are an indication of the sample matrix effects.

9.5.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

$$RPD = [|\text{MS Result} - \text{MSD Result}| \ / \ \text{Average Result}] \times 100$$

The RPD for each analyte should fall within the established control limits. If more than 33% of the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

10.0 CALCULATIONS

The concentration of each analyte in the original sample is calculated as follows:

Water (ug/l) = $(CONC_{inst}) \times (V_F / V_I) \times DF$

Soil (ug/kg) = $[(CONC_{inst}) \times (V_F / W_I) \times DF] / \%solids$

$CONC_{inst}$ = Instrument concentration calculated from the initial calibration using mean RF, linear curve, or quadratic curve

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DF	=	Dilution Factor
V_F	=	Volume of final extract (ul)
V_I	=	Volume of sample extracted (ml)
W_I	=	Weight of sample extracted (g)
%solids	=	Dry weight determination in decimal form

All soils are reported on a dry weight basis.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst should follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment should be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis and standards preparation are collected in waste storage bottles and are eventually transferred to the chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 REFERENCES

SW846 Method 8000B Revision 2, December 1996

SW846 Method 8000C Revision 3, March 2003

SW846 Method 8270C Revision 3, December 1996

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TABLE 1

Routine Target Analytes

1,4-Dioxane
N-nitrosodimethylamine
Pyridine
Benzaldehyde
Aniline
bis(2-Chloroethyl)ether
Phenol
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
Benzyl alcohol
bis(2-chloroisopropyl)ether
2-Methylphenol
Acetophenone
Hexachloroethane
N-Nitroso-di-n-propylamine
3&4-Methylphenol
Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
bis(2-Chloroethoxy)methane
Benzoic Acid
2,4-Dichlorophenol
1,2,4-Trichlorobenzene
Naphthalene
4-Chloroaniline
2,6-Dichlorophenol
Hexachlorobutadiene
Caprolactam
4-Chloro-3-methylphenol
2-Methylnaphthalene
1-Methylnaphthalene
1,2,4,5-Tetrachlorobenzene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
1,1'-Biphenyl
2-Chloronaphthalene
2-Nitroaniline
Acenaphthylene
Dimethylphthalate
2,6-Dinitrotoluene
Acenaphthene
3-Nitroaniline
2,4-Dinitrophenol
Dibenzofuran
2,4-Dinitrotoluene
4-Nitrophenol
2,3,4,6-Tetrachlorophenol
Fluorene
4-Chlorophenyl-phenylether
Diethylphthalate
4-Nitroaniline
4,6-Dinitro-2-methylphenol
n-Nitrosodiphenylamine
1,2-Diphenylhydrazine
4-Bromophenyl-phenylether
Hexachlorobenzene
Atrazine
Pentachlorophenol
Phenanthrene
Anthracene
Carbazole
Di-n-butylphthalate
Fluoranthene
Benzidine
Pyrene
Butylbenzylphthalate
3,3'-Dichlorobenzidine
Benzo[a]anthracene
Chrysene
bis(2-Ethylhexyl)phthalate
Di-n-octylphthalate
Benzo[b]fluoranthene
Benzo[k]fluoranthene
Benzo[a]pyrene
Indeno[1,2,3-cd]pyrene
Dibenz[a,h]anthracene
Benzo[g,h,i]perylene

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STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF BASE-NEUTRAL AND ACID (BNAs) EXTRACTABLES FROM SOLID SAMPLES

Prepared by: Norm Farmer Date: 03/10/06

Reviewed by: Stephanie Weber Date: 03/10/06

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Document Control

Issued to: QA Department Date: _____

Issued to: Organics Prep Department Date: _____*

Issued to: _____ Date: _____

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TEST NAME: **STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF BASE-NEUTRAL AND ACID (BNAs) EXTRACTABLES FROM SOLID SAMPLES**

Method: SW846 3550B/8270C

Dept: OP

1.0 Summary, Scope and Application

1.1 Summary

Solid samples are serially extracted by pulse sonication, concentrated by Kuderna-Danish apparatus, and stored in glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to solid samples, including soils and sediments, submitted for BNA analysis by GC/MS method SW-846 8270C.

2.0 Discussion and Comments

This procedure is adapted from SW-846 methods 3500B and 3550B. The method outlined in this SOP is designed for low concentration samples (concentration of the individual organic components is expected to be less than 20mg/kg).

Although the mass spectrometer identifies compounds by specific ions, it will respond to most organic compounds. It is important to minimize extraneous contaminants by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass, stainless steel, wood, or Teflon. Plastic items must be avoided because they can lead to phthalate contamination.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.0 Samples shall be collected in amber glass jars with Teflon lined caps. 250ml jars are recommended for solid samples.

3.1.1 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be refrigerated at –10°C to –20°C until analysis.

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3.2 Holding Time

3.2.0 Solid samples must be extracted within 14 days of collection. A sample is considered extracted after it has been sonicated three times and the solvent has been transferred to an Erlenmeyer flask.

3.2.1 Extracts must be analyzed within 40 days of extraction.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in

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environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Acetone – pesticide grade or equivalent

5.2 Methylene chloride – pesticide grade or equivalent

5.3 Anhydrous sodium sulfate – precleaned to remove phthalates

5.4 BNA Surrogate Solution – prepared in acetone or methanol at a concentration specified by the GC/MS analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.5 BNA Spike Solution – prepared in acetone or methanol at a concentration specified by the GC/MS analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

6.0 Glassware and Apparatus

6.1 400ml thick walled beaker

6.2 Glass funnels (large enough to support the filters)

6.3 500ml Erlenmeyer flask

6.4 Spatula – stainless steel, wood, or Teflon

6.5 0.5ml or 1.0ml syringes

6.6 1.0ml volumetric flask

6.7 500ml Kuderna-Danish (K-D) flask

6.8 10ml graduated concentrator tube

6.9 3-ball macro Snyder column

6.10 Disposable transfer pipettes

6.11 Teflon boiling chips

6.12 glass wool – precleaned

6.13 Fisher P8 filters – or equivalent

6.14 2.0ml glass screw cap vials – caps must have Teflon lined septa

6.15 Water bath – adjustable temperature control

6.16 Nitrogen evaporator

6.17 Ultrasonic disrupter – minimum power of 300 watts with pulse capability

6.18 Disrupter horns – ¾" solid titanium tip

6.19 Top loading balance – capable of weighing samples to +/- 0.1 grams

7.0 Sonic Disrupter

Follow the manufacturer's instructions for tuning and operating the sonic disrupter. Tuning should be verified at least weekly and should be documented in the "Instrument Repair and Maintenance" logbook. The sonic disrupter should be set in the pulse mode with a 50% duty cycle (energy on 50% of the time and off 50% of the time). Because the Fisher Scientific 550 Sonic Dismembrator and Branson 450 Digital Sonifier have maximum power outputs of 475 watts and 400 watts respectively, the power control levels can be set at 70% and 80% output instead of 100% output.

8.0 Procedure

8.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, and dates. The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the GC/MS analyst with the extracts.

8.2 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. Discard the top layer or soil and thoroughly mix the sample with a wooden spatula. Samples with obvious layers may need to be transferred to a larger container for proper mixing. Refer to SOP QA035 for more information on sample homogenization.

8.3 Transfer approximately 30 grams of each sample to the appropriately labeled beakers. Use a clean spatula for each sample. Record the weight to the nearest 0.1gram on the prep sheet.

8.4 Add approximately 30 grams of sodium sulfate to each sample and mix until each sample has a free flowing sandy texture. Wetter soils will require more sodium sulfate.

8.5 Transfer approximately 30 grams of each of the QC samples to the appropriately labeled beakers. This includes the method blank (MB), blank spike (BS), matrix

spike (MS), and matrix spike duplicate (MSD). Use 30 grams of sodium sulfate and/or clean sand for the MB and BS. Use additional 30 gram aliquots of a sample for the MS and MSD. If there is insufficient sample amount, a lesser amount may be used. Record the sample ID, bottle number and weight on the prep sheet.

8.6 Using the dedicated surrogate syringe add 0.5ml of surrogate solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

8.7 Using the dedicated spike syringe add 0.5ml of spike solution to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

8.8 Immediately add 100ml of 50:50 methylene chloride and acetone to each of the beakers. This will minimize the loss of the more volatile analytes.

NOTE: Crushed concrete samples should be extracted with 100ml aliquots of methylene chloride. Concrete reacts with the acetone to form excessive Aldol Condensation byproducts that interfere with the early eluting acid target compounds and surrogates.

8.9 Assemble and label the K-D flasks and concentrator tubes. Using forceps, place a Teflon boiling chip in each concentrator tube and set each K-D flask in a support stand. Use an Erlenmeyer flask (with cover) for storage, if the extracts will not be concentrated the same day.

8.10 Place a glass filter funnel containing a Fisher P8 filter on the top of each K-D.

8.11 Place the disrupter horn in the beaker such that the tip of the horn is approximately ½ inch into to solvent but not touching the beaker or the sample.

8.12 Sonicate the sample for 3 minutes.

8.13 Remove the beaker and decant the solvent through the filter funnel into the K-D.

8.14 Repeat steps 8.11 to 8.13 two more times with additional 100ml solvent aliquots. All three solvent aliquots will be combined in the K-D flask.

8.15 After the final extraction, transfer the entire sample to the funnel, rinse thoroughly, and allow the solvent to drain through the sample.

NOTE: It is important to clean the disrupter horn between samples. This is done by rinsing the horn with the extraction solvent and wiping it dry with a Kim-wipe or paper towel.

8.16 Remove the filter funnel and attach a Snyder column to the K-D flask.

8.17 Pre-wet the Snyder column with a few drops of methylene chloride and place the K-D assembly in a hot (75 to 85 ^{o}C) water bath. **NOTE:** If the bath is too hot, the

more volatile compounds may be lost during this step. Concentrate the extract to approximately 5ml.

8.18 Remove the K-D assembly from the bath and allow it to cool. Remove the Snyder columns and rinse them with methylene chloride and then with acetone before placing them back in their storage rack.

8.19 Wipe the water from the joint area between the K-D flask and the concentrator tube. Be sure that the sample ID is still on the concentrator tube. Remove the concentrator tube from the K-D flask, **rinse the joint with solvent**, and place the concentrator tube in the rack for the nitrogen evaporator.

8.20 Use a steady stream of nitrogen to concentrate the extract to 0.5ml.

8.21 If the extract is cloudy or contains water droplets, run the extract through a micro column of glass wool and sodium sulfate.

8.22 Transfer the extract to a 1.0ml volumetric flask. Rinse the concentrator tube with a few drops of methylene chloride and transfer it to the volumetric flask. Adjust the final volume to 1.0ml. **NOTE:** If the extract will not concentrate to 1.0ml, choose the next appropriate final volume. Be sure to record the final volume on the prep sheet.

8.23 Transfer the extract to an appropriately labeled 2.0ml screw cap vial. Store the extracts in the “extract freezer” until they are needed for analysis.

9.0 Quality Assurance and Quality Control

9.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

9.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

10.0 Safety and Waste Disposal

10.1 Safety

10.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

10.1.2 **Hearing protection** must be worn while operating the sonic disrupters. The high frequency could cause permanent hearing loss.

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10.1.3 Avoid touching the disrupter horns while they are active. Contact may cause burns or tissue damage.

10.1.4 Material Safety Data Sheets (MSDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS prior to using any new reagents or solvents.

10.1.5 Methylene chloride is an inhalation hazard and a suspected carcinogen. Fume hoods must be used to minimize exposure to vapors.

10.2 Waste Disposal

10.2.1 Waste methylene chloride is placed in the “chlorinated waste” container.

10.2.2 Waste acetone is placed in the “non-chlorinated waste” container.

10.2.3 Extracted soil samples are placed in a waste container after the solvent has drained or evaporated.

10.2.4 Waste soil from the homogenizing process should be place in the “soil waste” container. **NOTE:** Waste soil from foreign soils must follow “foreign soil” disposal requirements.

11.0 References

11.1 SW-846 Method 3500B, Rev. 2, 12/96

11.2 SW-846 Method 3550B, Rev. 2, 12/96

11.3 SW-846 Method 8270C, Rev. 3, 12/96

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ANALYSIS OF POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

Prepared by: Norm Farmer _____ Date: 03/06/06

Reviewed by: Stephanie Weber _____ Date: 03/06/06

Annual Review

Reviewed by: _____ Date: _____

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Document Control

Issued to: QA Department _____ Date: _____

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Effective 7 days after "*" date

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TEST NAME: ANALYSIS OF POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

METHOD REFERENCE: SW846 8082

DEPT: GC

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the polychlorinated biphenyl (PCB) concentrations in water, solid, and waste matrices utilizing a gas chromatograph equipped with an electron capture detector.

1.1.2 PCBs for this method are reported as Aroclors. Aroclors are multi-component mixtures consisting of various chlorinated biphenyl congeners. Quantitation is based on individual Aroclor standards. For the purpose of this SOP, the term Aroclor and PCB may be used interchangeably. The following compounds can be reported by this method:

Aroclor 1016
Aroclor 1221
Aroclor 1232
Aroclor 1242
Aroclor 1248
Aroclor 1254
Aroclor 1260
Aroclor 1262 (optional)
Aroclor 1268 (optional)

1.1.3 Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. Reporting limits may vary depending on matrix complications and volumes. Reporting limits for this method are in the range of 0.5 ug/l for aqueous samples, 33 ug/kg for solid samples, 1ug/wipe for surface wipe samples, and 10 mg/kg for oil samples. Solid matrices are reported on a dry weight basis.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.1.5 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a

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"J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8082.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with Accutest SOP OP008, OP009, or OP033.

1.2.4 The extracts are analyzed on a gas chromatograph equipped with an electron capture detector.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples.

2.1.2 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be stored at 4°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Interferences from phthalate esters can be eliminated by using plastic-free solvent containers and solvent rinsed glassware.

3.3 Other organic compounds, including chlorinated pesticides, chlorinated hydrocarbons, phenols, and phthalate esters may be coextracted by this method. Many of these interferences can be removed by sulfuric acid cleanup.

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3.4 Interferences from sulfur compounds can be eliminated using either a tetrabutyl ammonium sulfite (TBA) cleanup or an activated copper powder cleanup. Regional samples are generally high in sulfur content; therefore, all aqueous and solid sample extracts should be sulfur cleaned prior to analysis.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the

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analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Hexane – pesticide grade or equivalent

5.2 PCB Stock standards – Traceable to Certificate of Analysis

5.3 Surrogate standard – TCMX and Decachlorobiphenyl

6.0 APPARATUS

6.1 Gas Chromatograph – HP 5890 or Agilent Technologies 6890

Suitable gas chromatograph equipped with a split-splitless injection port and electron capture detectors.

Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Data System – Agilent Technologies MS Chemstation rev. BA 02.00 and DA 02.00

6.2.1 A computer system interfaced to the gas chromatograph that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.2.2 Data is archived to magnetic tape for long term storage.

6.3 Dual DB-17/DB-1701 Column or equivalent: 30m X 0.32mm X 0.25um

6.4 Dual DB-17/DB-5 Column or equivalent: 30m X 0.32mm X 0.25um

6.5 Suitable syringes and glassware for dilutions of standards and extracts.

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7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards should be stored at 4 °C, or as recommended by the manufacturer.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to one year or the vendor's expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with hexane. The hold time for intermediate standards is six months or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicate analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for PCB 1016 and 1260 are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the RL and the remaining standards define the working range of the detector.

Standards for the remaining five PCBs are made at a single level near the low to mid-point of the curve.

Calibration standard concentrations for PCB 1016 and PCB 1260 are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Gas Chromatograph Conditions

1-2ul autosampler injection

Carrier gas – UHP Helium (~2.5 ml/min)

Detector gas – P-5 (5% methane in argon) (30-90 ml/min)

Injection port temperature – 190 °C

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Detector temperature – 300 °C

6890
Oven program – 150 °C for 0.5 minutes
30 °C/min to 150 °C for 0 minutes
30 °C/min to 280 °C for 5.7 minutes

OR

5890
Oven program – 150 °C for 0.5 minutes
25 °C/min to 150 °C for 0 minutes
25 °C/min to 280 °C for 7.3 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Sample Preparation

7.3.1 Water Samples

A 1000ml aliquot of sample is extracted with methylene chloride utilizing separatory funnel extraction. The extract is solvent exchanged into hexane, concentrated to 10.0ml, sulfur cleaned, and acid cleaned.

7.3.2 Solid Samples

A 30-gram aliquot of sample is extracted with methylene chloride utilizing pulse sonication. The extract is solvent exchanged into hexane, concentrated to 10.0ml, sulfur cleaned, and acid cleaned.

7.3.3 Surface Wipe Samples

A wipe sample is extracted with hexane utilizing a wrist shaker. The extract may then be sulfur cleaned or acid cleaned.

7.3.4 Oil Samples

A one-gram aliquot of sample is diluted in hexane to 10ml. The extract may then be sulfur cleaned or acid cleaned.

7.4 Gas Chromatographic Analysis

Instrument calibration consists of three major sections:

Initial Calibration Procedures
Daily Calibration Procedures
Continuing Calibration Verification

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7.4.1 Initial Calibration Procedures

Before samples can be run, the chromatographic system must be calibrated, retention time windows must be determined, and pattern recognition standards must be analyzed.

7.4.1.1 External Standard Calibration

Prime the GC by injecting a conditioning standard, this is a high level PCB standard. This will aid in deactivating the injection port and column.

Quantitation for this method is based on the response of individual peaks in each individual PCB standard. Three to six major peaks from each Aroclor are used for quantitation purposes. Each individual PCB peak is assigned the concentration of the total PCB standard.

A minimum 5-point calibration curve is created for PCB 1016, PCB 1260, TCMX, and Decachlorobiphenyl. PCB 1016 and 1260 are used to establish linearity for all of the PCBs.

The remaining PCBs are calibrated at a single level in the low to mid-point range of the PCB 1016/1260 curve. The single point standards are also used for pattern recognition.

Calibration factors (CF) for PCBs are determined at each concentration by dividing the total area (or height) of the PCB (3 to 6 major peaks) by the concentration of the standard. Calibration factors for the surrogate are determined at each concentration by dividing the area (or height) of the peak by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the calibration factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively if the %RSD > 20% a calibration curve of response vs. amount can be plotted. If the correlation coefficient (r) is $\geq$0.995 ($r^2 \geq$0.990) then the curve can be used to quantitate target analytes in the samples.

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7.4.1.2. Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV should be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be ≤ 15%. If the ICV does not meet this criteria, a second standard should be prepared. If the ICV still does not meet criteria, analyze an ICV prepared from a third source. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.4.1.3 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Individual PCB peaks must fall within the appropriate retention time windows; however, Aroclor identification is based primarily on individual peak ratios and pattern recognition.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the

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retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations.

The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. The analyst should monitor the retention times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential "hits". The analyst will then review these "hits" and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

7.4.2 Daily Calibration Procedures

Prime the GC by injecting a conditioning standard, this is a high level PCB standard. This will aid in deactivating the injection port and column.

Analyze a continuing calibration verification standard (section 7.4.3) for PCB 1016 and PCB 1260. Then analyze the PCBs that are calibrated at a single concentration level. The single point standards must be analyzed every 24 hours; they do not have to be analyzed at the same frequency as the CCV standard.

7.4.3 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for PCB 1016 and PCB 1260 are prepared at various concentrations (CCV standards for other PCBs may be required); at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally a CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| should be ≤ 15% for each analyte. If the first continuing calibration verification does not meet criteria, a second standard may be

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injected. If the second standard does not meet criteria, the system must be recalibrated.

If the |%D| is greater than 15%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported without qualification. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed or the data must be qualified.

7.4.4 Sample Extract Analysis

7.4.4.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Conditioning Standard
Initial Calibration Standards (or Initial CCV)
Single Point Standards
QC Extracts
Sample Extracts
CCV Standards

7.4.4.2 One or two microliters (same amount as standards) of extract is injected into the GC by the autosampler. A splitless injection technique is used. The data system then records the resultant peak responses and retention times.

7.4.4.3 Tentative identification of an analyte occurs when the peaks from the sample extract fall within the established retention time windows for a calibrated compound on the primary column.

7.4.4.4 If the peaks of interest fall within the retention time windows on the confirmation column, the identification is confirmed. Quantitation of the analyte on the primary and confirmation column should agree within 40%. If the difference is greater than 40% and no obvious reason can be found, the higher result should be reported and flagged as “estimated”; otherwise, the result from the primary column should be reported.

7.4.4.5 Pattern comparisons should also be used to aid in the identification of the multipeak analytes.

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7.4.4.6 If the compound identification does not confirm on a dissimilar column, then the result should be reported as ND or “U”.

7.4.4.7 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.4.8 If peak or pattern identification is prevented by the presence of interferences, further cleanup may be required or the extract must be diluted so that the interference does not mask any analytes.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Surrogates

9.1.1 Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl are used as the surrogate standards to monitor the efficiency of the extraction and clean-up procedures.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for both surrogates for the results to be acceptable.

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9.1.2 If the surrogate recoveries are not within the established control limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, integrations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly.

9.1.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, re-extract and reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary. If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis should be reported on the final report.

9.2 Method Blank

9.2.1 The method blank is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high level samples. The method blank must be free of any analytes of interest or interferences at ½ the required reporting level to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination should be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

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9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination should be investigated and documented. The samples should be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable.

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.3.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary and the sample results can be reported without qualification.

9.3.2.4 If the recovery of an analyte in the BS is high and the associated sample is non-detect, then the sample results can be reported without qualification.

9.3.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

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9.3.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4 Matrix Spike and Matrix Spike Duplicate

9.4.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.4.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.4.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.4.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are an indication of the sample matrix effects.

9.4.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the

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department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

10.0 CALCULATIONS

The amount of the PCB in the samples is determined by averaging the concentration of the major peaks for each individual PCB. The HP Enviroquant software will automatically sum and average the peaks used for each individual Aroclor.

The concentration of each individual PCB peak in the original sample is calculated as follows:

PCB Water (ug/l) = $(CONC_{inst}) \times (V_F / V_I) \times DF$

PCB Soil (ug/kg) = $[(CONC_{inst}) \times (V_F / W_I) \times DF] / \%solids$

$CONC_{inst}$	=	Instrument concentration calculated from the initial calibration using mean CF, linear curve, quadratic curve or single point calibration
DF	=	Dilution Factor
V_F	=	Volume of final extract (ml)
V_I	=	Volume of sample extracted (ml)
W_I	=	Weight of sample extracted (g)
%solids	=	Dry weight determination in decimal form

All soils are reported on a dry weight basis.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst should follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment should be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis and standards preparation are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

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Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 REFERENCES

SW846 Method 8000B Revision 2, December 1996

SW846 Method 8000C Revision 3, March 2003

SW846 Method 8082 Revision 0, December 1996

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ANALYSIS OF ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

Prepared by: Norm Farmer Date: 03/06/06

Reviewed by: Stephanie Weber Date: 03/06/06

Annual Review

Reviewed by: _____ Date: _____

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Reviewed by: _____ Date: _____

Document Control

Issued to: QA Department Date: _____

Issued to: GC Semivolatile Department Date: _____*

Issued to: _____ Date: _____

Issued to: _____ Date: _____

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Effective 7 days after "*" date

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TEST NAME: ANALYSIS OF ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

METHOD REFERENCE: SW846 8081A

DEPT: GC

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of specific organochlorine pesticides in water and solid matrices utilizing a gas chromatograph equipped with an electron capture detector.

1.1.2 The following compounds can be reported by this method:

Aldrin	Dieldrin
Alpha-BHC	Endosulfan I
Beta-BHC	Endosulfan II
Delta-BHC	Endosulfan Sulfate
Gamma-BHC (Lindane)	Endrin
Chlordane (Technical)	Endrin Ketone
Alpha-chlordane	Endrin Aldehyde
Gamma-chlordane	Heptachlor
4,4'-DDD	Heptachlor Epoxide
4,4'-DDE	Methoxychlor
4,4'-DDT	Toxaphene

1.1.3 Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. Reporting limits may vary depending on matrix complications and volumes. Reporting limits for the single peak pesticides in this method are in the range of 0.05 to 0.10 ug/l for aqueous samples and 1.7 to 3.3 ug/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.1.5 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a

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"J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8081A.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with Accutest SOP OP008 and OP009.

1.2.4 The extracts are analyzed on a gas chromatograph equipped with an electron capture detector.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples.

2.1.2 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be stored at 4°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Interferences from phthalate esters can be eliminated by using plastic-free solvent containers and solvent rinsed glassware.

3.3 Other organic compounds, including PCBs, chlorinated hydrocarbons, phenols, and phthalate esters may be coextracted by this method.

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3.4 Interferences from sulfur compounds can be eliminated using a tetrabutyl ammonium sulfite (TBA) cleanup. Regional samples are generally high in sulfur content; therefore, all aqueous and solid sample extracts should be sulfur cleaned prior to analysis.

Sulfur cleanup will reduce endrin aldehyde recoveries to less than 20%. If endrin aldehyde is detected, the non-sulfur cleaned aliquot shall be analyzed. If possible, endrin aldehyde will be reported from the non-sulfur cleaned aliquot.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

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4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Hexane – pesticide grade or equivalent

5.2 Pesticide stock standards – Traceable to Certificate of Analysis

5.3 Surrogate standard – TCMX and Decachlorobiphenyl

6.0 APPARATUS

6.1 Gas Chromatograph – Agilent Technologies 6890 with 7683 Autosampler

Suitable gas chromatograph equipped with a split-splitless injection port and electron capture detectors.

Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Data System – Agilent Technologies MS Chemstation rev. BA 02.00 and DA 02.00

6.2.1 A computer system interfaced to the gas chromatograph that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.2.2 Data is archived to magnetic tape for long term storage.

6.3 Dual DB-17/DB-1701 Column or equivalent: 30m X 0.32mm X 0.25um

6.4 Suitable syringes and glassware for dilutions of standards and extracts.

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7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards should be stored at 4 °C, or as recommended by the manufacturer.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to one year or the vendor's expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with hexane. The hold time for intermediate standards is six months or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicate analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the single peak pesticides are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the RL and the remaining standards define the working range of the detector.

Standards for the multipeak pesticides (Toxaphene and Technical Chlordane) are made at a single level near the RL. **NOTE:** Some projects may require that samples with positive detects for either toxaphene or technical chlordane be reanalyzed and quantitated using a 5-point calibration.

Calibration standard concentrations for the single peak pesticides are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Gas Chromatograph Conditions

1-2ul autosampler injection

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Carrier gas – UHP Helium (2.5 to 3.0 ml/min ramped flow or pulsed injection)

Detector gas – P-5 (5% methane in argon) (45 - 90 ml/min)

Injection port temperature – 190 °C

Detector temperature – 300 °C

Oven program – 110 °C for 0.5 minutes
25 °C/min to 150 °C for 0 minutes
12.5 °C/min to 260 °C for 0 minutes
30 °C/min to 280 °C for 6.4 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Sample Preparation

7.3.1 Water Samples

A 1000ml aliquot of sample is extracted with methylene chloride utilizing separatory funnel extraction. The extract is solvent exchanged into hexane, concentrated to 10.0ml, and sulfur cleaned.

7.3.2 Solid Samples

A 30-gram aliquot of sample is extracted with methylene chloride utilizing pulse sonication. The extract is solvent exchanged into hexane, concentrated to 10.0ml, and sulfur cleaned.

7.4 Gas Chromatographic Analysis

Instrument calibration consists of three major sections:

Initial Calibration Procedures
Daily Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, injection port inertness must be verified, the chromatographic system must be calibrated, and retention time windows must be determined.

7.4.1.1 Breakdown Check

Prime the GC by injecting a conditioning standard; this is a pesticide standard that is approximately 20 times the

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concentration of the mid-level standard. This will aid in deactivating the injection port and column.

Endrin and DDT breakdown must be verified at the start of each 12-hour shift. Inject a DDT/Endrin standard that is at a concentration near the mid-point of the curve. Calculate the percent breakdown of each analyte as follows (height may be used instead of area):

$$\%DDT_{BREAKDOWN} = \frac{(\text{DDE Area} + \text{DDD Area}) \times 100}{(\text{DDE Area} + \text{DDD Area} + \text{DDT Area})}$$

$$\%Endrin_{BREAKDOWN} = \frac{(\text{Endrin ketone Area} + \text{Endrin aldehyde Area}) \times 100}{(\text{Endrin ketone Area} + \text{Endrin aldehyde Area} + \text{Endrin Area})}$$

The percent breakdown for both DDT and endrin must not exceed 15%. If breakdown exceeds 15%, instrument maintenance must be performed before any samples can be analyzed.

7.4.1.2 External Standard Calibration

A minimum 5-point calibration curve is created for the single peak pesticides, TCMX, and Decachlorobiphenyl. Accutest Laboratories routinely performs a 6-point calibration to maximize the calibration range.

The low point may be omitted from the calibration table for any compound with an RL set at the level two standard. Additionally the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

Toxaphene and chlordane (technical) are calibrated at a single level near the RL. **NOTE:** Some projects may require that samples with positive detects for either toxaphene or technical chlordane be reanalyzed and quantitated using a 5-point calibration.

Calibration factors (CF) for the single peak pesticides and surrogates are determined at each concentration by dividing the area (or height) of each compound by the concentration of the standard.

Quantitation for chlordane is based on the individual peaks in the chlordane standard. Three to six major peaks are used for quantitation purposes. Each individual chlordane peak is assigned the concentration of the total chlordane standard. Toxaphene may be quantitated by total area or by the

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individual peaks. See method 8081A for additional information.

Calibration factor (CF) for chlordane is determined by dividing the total area (or height) of chlordane (3 to 6 major peaks) by the concentration of the standard. The calibration factor for the toxaphene is determined by dividing the total area (height can not be used) of toxaphene by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the calibration factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively if the %RSD > 20% a calibration curve of response vs. amount can be plotted. If the correlation coefficient (r) is $\geq$0.995 ($r^2 \geq$0.990) then the curve can be used to quantitate target analytes in the samples.

7.4.1.3 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV should be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be $\leq$ 15%. If the ICV does not meet this criteria, a second standard should be prepared. If the ICV still does not meet criteria, analyze an ICV prepared from a third source. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.4.1.4 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do

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not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations.

The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. The analyst should monitor the retention times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential “hits”. The analyst will then review these “hits” and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

7.4.2 Daily Calibration Procedures

Prime the GC by injecting a conditioning standard; this is a pesticide standard that is approximately 20 times the concentration of the mid-level standard. This will aid in deactivating the injection port and column.

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Analyze a DDT/Endrin breakdown standard as described in section 7.4.1.1. This standard must be analyzed at the start of each 12-hour shift.

Analyze a continuing calibration verification standard (section 7.4.3) for the single peak pesticides. Then analyze the multipeak pesticide standards that are calibrated at a single concentration level. The single point standards must be analyzed every 12 hours; they do not have to be analyzed at the same frequency as the CCV standard.

7.4.3 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the single peak pesticides are prepared at various concentrations; at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally a CCV must be analyzed after every 10 samples. If toxaphene or technical chlordane is known to be present at the site, the analyst should analyze additional CCVs for these analytes.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| should be $\leq$ 15% for each analyte. If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated.

If the |%D| is greater than 15%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported without qualification. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed or the data must be qualified.

7.4.4 Sample Extract Analysis

7.4.4.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Conditioning Standard
DDT/Endrin Breakdown Standard
Initial Calibration Standards (or Initial CCV)
Single Point Standards

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QC Extracts
Sample Extracts
CCV Standards

7.4.4.2 One or two microliters (same amount as standards) of extract is injected into the GC by the autosampler. A splitless injection technique is used. The data system then records the resultant peak responses and retention times.

7.4.4.3 Tentative identification of an analyte occurs when the peaks from the sample extract fall within the established retention time windows for a calibrated compound on the primary column.

7.4.4.4 If the peaks of interest fall within the retention time windows on the confirmation column, the identification is confirmed. Quantitation of the analyte on the primary and confirmation column should agree within 40%. If the difference is greater than 40% and no obvious reason can be found, the higher result should be reported and flagged as “estimated”; otherwise, the result from the primary column should be reported.

7.4.4.5 Pattern comparisons should also be used to aid in the identification of the multipeak analytes.

7.4.4.6 If the compound identification does not confirm on a dissimilar column, then the result should be reported as ND or “U”.

7.4.4.7 The sulfur cleaning procedure used for this method will lower the Endrin Aldehyde recoveries to 10-20%. If Endrin, Endrin Ketone or Endrin Aldehyde is detected in the samples, the analyst should analyze a “non-sulfur cleaned” aliquot (and associated QC samples). If possible, Endrin Aldehyde should be reported from this analysis.

7.4.4.8 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.4.9 If peak identification is prevented by the presence of interferences, further cleanup may be required or the extract must be diluted so that the interference does not mask any analytes.

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8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Surrogates

9.1.1 Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl are used as the surrogate standards to monitor the efficiency of the extraction and clean-up procedures.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for both surrogates for the results to be acceptable

9.1.2 If the surrogate recoveries are not within the established control limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, integrations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly.

9.1.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

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9.1.2.3 If no problem is found, re-extract and reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary. If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis should be reported on the final report.

9.2 Method Blank

9.2.1 The method blank is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high level samples. The method blank must be free of any analytes of interest or interferences at ½ the required reporting level to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination should be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination should be investigated and documented. The samples should be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard and spike standard

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have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable. The large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of the established control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary.

Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A marginal exceedance in the Blank Spike is defined as a recovery being outside of 3 standard deviations but within 4 standard deviations of the mean.

The number of allowable marginal exceedances is based on the number of analytes in the Blank Spike. Marginal Exceedances must be random. If the same analyte exceeds the BS control limits repeatedly, it is an indication of a systematic problem and corrective action must be taken.

The number of allowable marginal exceedances is as follows:

1) 11-30 analytes in BS, 1 analyte allowed in ME range;

2) < 11 analytes in BS, no analytes allowed in ME range

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.3.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary and the sample results can be reported without qualification.

9.3.2.4 If the recovery of an analyte in the BS is high and the associated sample is non-detect, then the sample results can be reported without qualification.

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9.3.2.5 Excessive exposure to the sulfur cleaning reagents may adversely impact the recoveries of Delta BHC, Endrin Aldehyde, and Endosulfan Sulfate. Therefore, a non-sulfur cleaned fraction is always retained. If recoveries for these analytes are low in the sulfur cleaned blank spike, analyze the non-sulfur cleaned aliquot.

If the recoveries are within control limits then the entire "non-sulfur cleaned" batch should be analyzed and results reported from that analysis. Under NO circumstances can just the blank spike be reanalyzed and reported.

9.3.2.6 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.3.2.7 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4 Matrix Spike and Matrix Spike Duplicate

9.4.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.4.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.4.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may

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be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.4.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are an indication of the sample matrix effects.

9.4.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If more than 33% of the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

10.0 CALCULATIONS

The concentration of each single peak pesticide or toxaphene (if using the total area technique) in the original sample is calculated as follows:

Water (ug/l) = $(CONC_{inst}) \times (V_F / V_I) \times DF$

Soil (ug/kg) = $[(CONC_{inst}) \times (V_F / W_I) \times DF] / \%solids$

$CONC_{inst}$	=	Instrument concentration calculated from the initial calibration using mean CF, linear or quadratic curve, or single point standard
DF	=	Dilution Factor
V_F	=	Volume of final extract (ml)
V_I	=	Volume of sample extracted (ml)
W_I	=	Weight of sample extracted (g)
%solids	=	Dry weight determination in decimal form

All soils are reported on a dry weight basis.

The amount of chlordane or toxaphene (if using the multipeak technique) in the samples is determined by averaging the concentration of the major peaks for each multipeak pesticide. The MS Chemstation software will automatically sum and average the peaks used for each multipeak pesticide.

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11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst should follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment should be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis and standards preparation are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 REFERENCES

SW846 Method 8000B Revision 2, December 1996

SW846 Method 8000C Revision 3, March 2003

SW846 Method 8081A Revision 1, December 1996

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STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF PESTICIDES AND/OR PCBs FROM WATER SAMPLES

Prepared by: Norm Farmer Date: 03/10/06

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Annual Review

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TEST NAME: STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF PESTICIDES AND/OR PCBs FROM WATER SAMPLES

Method: EPA 608, SW846 3510C/8081A, and SW846 3510C/8082

Dept: OP

1.0 Summary, Scope and Application

1.1 Summary

Aqueous samples are serially extracted with methylene chloride, solvent exchanged into hexane, concentrated by TurboVap, and stored in glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to aqueous samples submitted for Pesticide and/or PCB analysis by GC/ECD methods, including EPA 608, SW-846 8081A, and SW-846 8082.

2.0 Discussion and Comments

This procedure is adapted from SW-846 methods 3500B and 3510C. The method utilizes “Separatory Funnel Liquid-Liquid Extraction”; it is not applicable to samples requiring “Continuous Liquid-Liquid Extraction”.

The ECD is an extremely sensitive detector that will respond to many organic and some inorganic compounds that exhibit a strong electronegativity. This includes phthalates and sulfur compounds. It is important to minimize extraneous contaminants by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass or Teflon.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.0 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples.

3.1.1 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be refrigerated at 4°C until analysis.

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3.2 Holding Time

3.2.0 Aqueous samples must be extracted within 7 days of collection. A sample is considered extracted after the methylene chloride aliquots have been combined in the Erlenmeyer flask.

3.2.1 Extracts must be analyzed within 40 days of extraction.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in

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environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Acetone – pesticide grade or equivalent

5.2 Methylene chloride – pesticide grade or equivalent

5.3 Hexane – pesticide grade or equivalent

5.4 Isopropyl Alcohol – pesticide grade or equivalent

5.5 Anhydrous sodium sulfate – precleaned to remove phthalates

5.6 Tetrabutylammonium (TBA) sulfite reagent

5.7 Reagent water – distilled or deionized - free of interferences

5.8 10 Normal NaOH

5.9 1:1 H_2SO_4

5.10 Concentrated H_2SO_4

5.11 Pesticide/PCB Surrogate Solution – prepared in acetone or methanol at a concentration specified by the GC analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.12 **Pesticide** Spike Solution – prepared in acetone or methanol at a concentration specified by the GC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.13 **PCB** Spike Solution – prepared in acetone or methanol at a concentration specified by the GC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

6.0 Glassware and Apparatus

6.1 1000ml graduated cylinder

6.2 2 liter separatory funnel (glass or Teflon)

6.3 250ml separatory funnel

6.4 250ml or 500ml Erlenmeyer flasks

6.5 0.5ml or 1.0ml syringes

6.6 200ml graduated TurboVap tube

6.7 10ml graduated concentrator tube

6.8 Disposable transfer pipettes

6.9 pH paper

6.10 glass wool – precleaned

6.11 Glass filter funnel

6.12 Fisher P8 filters, or equivalent

6.13 2.0ml glass screw cap vials – caps must have Teflon lined septa

6.14 8ml and 16ml glass screw cap vials – caps must have Teflon lined septa

6.15 Zymark TurboVap II or equivalent

7.0 Procedure

7.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: sample ID, bottle number, initial volume, final volume, pHs, solvent lot numbers, spike and surrogate lot numbers, batch numbers, and dates. The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the GC analyst with the extracts.

7.2 Mark the level of the sample on the bottle. Transfer the entire sample directly into the appropriately labeled separatory funnel. Rinse the sample bottle with a 50-60ml aliquot of methylene chloride and transfer it to the appropriate separatory funnel. Fill the bottle to the sample mark with tap water. Transfer the water to a 1000ml graduated cylinder and record the sample volume. Discard the tap water.

Alternative procedure for samples with high silt or sediment content.

The entire contents of the sample bottle should be analyzed for aqueous samples, including any solids that may have been collected. However, high levels of solids in the sample can create heavy emulsions that can not be broken down by the various mechanical means.

The solids will normally settle out during storage. If the sample bottle contains more than an inch of solids, it may be necessary to decant the water phase rather than extracting the entire sample. The decision to decant a water sample

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should be based on the experience and judgement of the extraction technician or the department supervisor. If the sample is decanted, it must be noted on the prep sheet.

Using a 1000ml-graduated cylinder, transfer the sample to the appropriately labeled separatory funnel taking care to minimize the amount of solids that are transferred. Record the actual sample volume that is transferred. Rinse the graduated cylinder with a 50-60ml aliquot of methylene chloride and transfer it to the appropriate separatory funnel.

The graduated cylinder must be rinsed with hot tap water, reagent water, and methylene chloride between samples in order to prevent cross contamination.

7.3 Using a 1000ml-graduated cylinder, transfer 1000ml of each of the QC samples to the appropriately labeled separatory funnels. This includes the reagent blank (MB), reagent blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD). Use 1000mls of reagent water for the MB and BS. Use additional 1000ml aliquots of a sample for the MS and MSD. If there is insufficient sample volume, a lesser volume may be used. Record the sample ID, bottle number, and volume on the prep sheet.

7.4 Using the dedicated surrogate syringe add 0.5ml of surrogate solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

7.5 Two spiking solutions are used in this SOP. If the samples are to be analyzed for "pesticides" only, use the **Pesticide** spiking solution. If the samples are to be analyzed for "PCBs" only, use the **PCB** spiking solution. If the samples are to be analyzed for both "pesticides" and "PCBs" two separate sets of QC (BS, MS, and MSD) must be prepared, one using the **Pesticide** spiking solution and one using the **PCB** spiking solution**.**

7.6 Using the dedicated spike syringe add 0.5ml of the appropriate spike solution to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

7.7 Check the pH of each sample by dipping a clean glass-stirring rod into the sample and touching it to the pH paper. Record the pH on the prep sheet. If necessary, adjust the pH to between 5 and 9 by adding a few drops of 10 N NaOH or 1:1 H_2SO_4.

7.8 Cap and shake each separatory funnel for two minutes. **CAUTION:** The separatory funnels must be vented frequently to avoid an excessive buildup in pressure.

7.9 After shaking, allow the layers to separate for at least 10 minutes. Collect the solvent layer (bottom) in a labeled 250ml Erlenmeyer flask.

NOTE: Some samples may form emulsions. If emulsions are present, the technician must take steps to breakdown the emulsion. This may include filtering

the emulsion through a smaller separatory funnel, centrifuging, or filtering through sodium sulfate.

7.10 Repeat steps 7.8 and 7.9 two additional times using 50 to 60ml of methylene chloride. Combine the extract in the Erlenmeyer flask.

7.11 Label the TurboVap tubes and place them in the metal support rack.

7.12 This step is **optional**, but it must be performed if the extracts appear cloudy or contain water droplets. Place a glass filter funnel containing sodium sulfate supported on a Fisher P8 filter on the TurboVap tube. Pour the extracts through the sodium sulfate into the TurboVap tube and rinse with methylene chloride. Care must be taken not to overfill the TurboVap tube.

7.13 If the extract appears to be dry, simply transfer it to an appropriately labeled TurboVap tube. Rinse the Erlenmeyer flask with methylene chloride and transfer to the appropriate TurboVap tube. Care must be taken not to overfill the TurboVap tube.

7.14 Set the water bath temperature for the TurboVap to 40 ^{o}C. Place the tube in the TurboVap. Concentrate the extract to less than 5ml.

7.15 Add approximately 50ml of hexane to the each of the TurboVap tubes. Increase the TurboVap water bath temperature to 55 ^{o}C and concentrate the extract to less than 5ml.

7.16 Remove the TurboVap tube and place it in the metal support rack to allow it to cool.

7.17 If the extract is cloudy or contains water droplets, run the extract through a micro column of glass wool and sodium sulfate.

7.18 Transfer the extract to a 10ml concentrator tube. Rinse the TurboVap tube with a few drops of hexane and transfer it to the concentrator tube. Adjust the final volume to 10.0ml. Use a transfer pipet to thoroughly mix the extract. Be sure to record the final volume on the prep sheet.

7.19 If the extract is to be analyzed for pesticides, transfer some of the extract to an appropriately labeled 2.0ml screw cap vial. This is kept as a backup.

7.20 Proceed to section 8.0 for acid cleaning procedure and section 9.0 for sulfur cleaning procedure. The PCB fraction undergoes both the acid and sulfur cleanup procedures. The pesticide fraction undergoes only the sulfur cleanup procedure.

8.0 Acid Cleaning Procedure (PCB only)

NOTE: This cleaning procedure can be used on the PCB fraction only. It will destroy the pesticides.

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8.1 Label the 8ml screw cap vials for each sample and associated QC.

8.2 Add 2ml of concentrated H_2SO_4 to each of the vials.

8.3 Using a transfer pipet, transfer approximately 2ml of extract to the vial. Cap the vial and shake for one minute.

8.4 Allow the extracts to stand for 10 minutes. Transfer the hexane layer (top) to an appropriately labeled 2.0ml screw cap vial.

8.5 Store the extracts in the "extract refrigerator" until they are needed for analysis.

9.0 Sulfur Cleaning Procedure (Pesticide and PCB)

9.1 Label the 16ml screw cap vials for each sample and associated QC.

9.2 Add 1ml of TBA-sulfite reagent and 2ml of isopropyl alcohol to each of the vials.

9.3 Using a transfer pipet, transfer approximately 2ml of extract to the vial. Cap the vial and shake for one minute.

9.4 Add 5ml of reagent water to each vial. Cap the vial and shake for one minute.

9.5 Allow the extracts to stand for 10 minutes. Transfer the hexane layer (top) to an appropriately labeled 2.0ml screw cap vial.

9.6 Store the extracts in the "extract refrigerator" until they are needed for analysis.

10.0 Quality Assurance and Quality Control

10.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

10.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

11.0 Safety and Waste Disposal

11.1 Safety

11.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

11.1.2 Material Safety Data Sheets (MSDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS prior to using any new reagents or solvents.

11.1.3 Methylene chloride is an inhalation hazard and a suspected carcinogen. Fume hoods must be used to minimize exposure to vapors.

11.2 Waste Disposal

11.2.1 Waste methylene chloride is placed in the “chlorinated waste” container.

11.2.2 Waste acetone is placed in the “non-chlorinated waste” container.

11.2.3 Waste sodium sulfate is placed in a waste container after the solvent has drained or evaporated.

11.2.4 Extracted water samples are rinsed down the drain with large amounts of water.

12.0 References

12.1 SW-846 Method 3500B, Rev. 2, 12/9

12.2 SW-846 Method 3510C, Rev. 3, 12/96

12.3 SW-846 Method 8081A, Rev. 1, 12/96

12.4 SW-846 Method 8082, Rev. 0, 12/96

12.5 SW-846 Method 3660B, Rev. 2, 12/96

12.6 SW-846 Method 3665A, Rev. 1, 12/96

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STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF PESTICIDES AND/OR PCBs FROM SOLID SAMPLES

Prepared by: Norm Farmer Date: 03/10/06

Reviewed by: Stephanie Weber Date: 03/10/06

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TEST NAME: **STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF PESTICIDES AND/OR PCBs FROM SOLID SAMPLES**

Method: SW846 3550B/8081A and SW846 3550B/8082

Dept: OP

1.0 Summary, Scope and Application

1.1 Summary

Solid samples are serially extracted by pulse sonication, solvent exchanged into hexane, concentrated by TurboVap, and stored in glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to solid samples submitted for Pesticide and/or PCB analysis by GC/ECD methods, including SW-846 8081A and SW-846 8082.

2.0 Discussion and Comments

This procedure is adapted from SW-846 methods 3500B and 3550B. The method outlined in this SOP is designed for low concentration samples (concentration of the individual organic components is expected to be less than 20mg/kg).

The ECD is an extremely sensitive detector that will respond to many organic and some inorganic compounds that exhibit a strong electronegativity. This includes phthalates and sulfur compounds. It is important to minimize extraneous contaminants by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass, stainless steel, wood, or Teflon.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.0 Samples shall be collected in amber glass jars with Teflon lined caps. 250ml jars are recommended for solid samples.

3.1.1 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be refrigerated at 4°C until analysis.

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3.2 Holding Time

3.2.0 Solid samples must be extracted within 14 days of collection. A sample is considered extracted after it has been sonicated three times and the solvent has been transferred to an Erlenmeyer flask.

3.2.1 Extracts must be analyzed within 40 days of extraction.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in

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environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Acetone – pesticide grade or equivalent

5.2 Methylene chloride – pesticide grade or equivalent

5.3 Hexane – pesticide grade or equivalent

5.4 Isopropyl Alcohol – pesticide grade or equivalent

5.5 Anhydrous sodium sulfate – precleaned to remove phthalates

5.6 Tetrabutylammonium (TBA) sulfite reagent

5.7 Concentrated H_2SO_4

5.8 Reagent water – distilled or deionized - free of interferences

5.9 Pesticide/PCB Surrogate Solution – prepared in acetone or methanol at a concentration specified by the GC analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.10 **Pesticide** Spike Solution – prepared in acetone or methanol at a concentration specified by the GC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.11 **PCB** Spike Solution – prepared in acetone or methanol at a concentration specified by the GC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

6.0 Glassware and Apparatus

6.1 400ml thick walled beaker

6.2 Glass funnels (large enough to support the filters)

6.3 500ml Erlenmeyer flask

6.4 Spatula – stainless steel, wood, or Teflon

6.5 0.5 or 1.0ml syringes

6.6 200ml graduated TurboVap tubes

6.7 10ml graduated concentrator tube

6.8 Disposable transfer pipettes

6.9 glass wool – precleaned

6.10 Fisher P8 filters – or equivalent

6.11 2.0ml glass screw cap vials – caps must have Teflon lined septa

6.12 8ml and 16ml glass screw cap vials – caps must have Teflon lined septa

6.13 Zymark TurboVap II or equivalent

6.14 Ultrasonic disrupter – minimum power of 300 watts with pulse capability

6.15 Disrupter horns – ¾" solid titanium tip

6.16 Top loading balance – capable of weighing samples to +/- 0.1 grams

7.0 Sonic Disrupter

Follow the manufacturer's instructions for tuning and operating the sonic disrupter. Tuning should be verified at least weekly and should be documented in the "Instrument Repair and Maintenance" logbook. The sonic disrupter should be set in the pulse mode with a 50% duty cycle (energy on 50% of the time and off 50% of the time). Because the Fisher Scientific 550 Sonic Dismembrator and Branson 450 Digital Sonifier have maximum power outputs of 475 watts and 400 watts respectively, the power control levels can be set at 70% and 80% output instead of 100% output.

8.0 Procedure

8.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, and dates. The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the GC analyst with the extracts.

8.2 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. Discard the top layer of soil and thoroughly mix the sample with a wooden spatula. Samples with obvious layers may need to be transferred to a larger container for proper mixing. Refer to SOP QA035 for more information on sample homogenization.

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8.3 Transfer approximately 30 grams of each sample to the appropriately labeled beakers. Use a clean spatula for each sample. Record the weight to the nearest 0.1gram on the prep sheet.

8.4 **Note:** A lesser amount, 3 to 10 grams of soil may be used for certain high level PCB projects, check with the department supervisor before reducing the sample amount.

8.5 Add approximately 30 grams of sodium sulfate to each sample and mix until each sample has a free flowing sandy texture. Wetter soils will require more sodium sulfate.

8.6 Transfer approximately 30 grams of each of the QC samples to the appropriately labeled beakers. This includes the method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD). Use 30 grams of sodium sulfate and/or clean sand for the MB and BS. Use additional 30 gram aliquots of a sample for the MS and MSD. If there is insufficient sample amount, a lesser amount may be used. Record the sample ID, bottle number, and weight on the prep sheet.

8.7 Using the dedicated surrogate syringe add 0.5ml of surrogate solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

8.8 Two spiking solutions are used in this SOP. If the samples are to be analyzed for "pesticides" only, use the **Pesticide** spiking solution. If the samples are to be analyzed for "PCBs" only, use the **PCB** spiking solution. If the samples are to be analyzed for both "pesticides" and "PCBs" two separate sets of QC (BS, MS, and MSD) must be prepared, one using the **Pesticide** spiking solution and one using the **PCB** spiking solution**.**

8.9 Using the dedicated spike syringe add 0.5ml of the appropriate spike solution to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

8.10 Immediately add 100ml of methylene chloride (50:50 methylene chloride and acetone or 50:50 hexane and acetone may also be used) to each of the beakers. This will minimize the loss of the more volatile analytes.

8.11 Label the Erlenmeyer flasks and place a glass filter funnel containing a Fisher P8 filter on the top of each flask.

8.12 Place the disrupter horn in the beaker such that the tip of the horn is approximately ½ inch into the solvent but not touching the beaker or the sample.

8.13 Sonicate the sample for 3 minutes.

8.14 Remove the beaker and decant the solvent through the filter funnel into the K-D.

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8.15 Repeat steps 8.12 to 8.14 two more times with additional 100ml solvent aliquots. All three solvent aliquots will be combined in the flask.

8.16 After the final extraction, transfer the entire sample to the funnel, rinse thoroughly, and allow the solvent to drain through the sample.

NOTE: It is important to clean the disrupter horn between samples. This is done by rinsing the horn with the extraction solvent and wiping it dry with a Kim-wipe or paper towel.

8.17 Label the TurboVap tubes and place them in the metal support rack. Because the total extract volume exceeds the capacity of the TurboVap tube, transfer approximately 200ml each extract to the appropriately labeled tube. The remaining extract will be transferred later.

8.18 Set the water bath temperature for the TurboVap to 40 ^{o}C. Place the tube in the TurboVap. **NOTE:** If the bath is too hot, the more volatile compounds may be lost during this step. Concentrate the extract to approximately 50ml.

8.19 Remove the TurboVap tube and transfer the remaining extract from the Erlenmeyer flask to the appropriate tube. Rinse each Erlenmeyer flask with methylene chloride and transfer it to the appropriate tube.

8.20 Return the tube to the TurboVap and concentrate the extract to between 1 and 5ml.

8.21 Add approximately 50ml of hexane to the each of the TurboVap tubes. Increase the TurboVap water bath temperature to 55 ^{o}C and concentrate the extract to less than 5ml.

8.22 Remove the TurboVap tube and place it in the metal support rack to allow it to cool.

8.23 If the extract is cloudy or contains water droplets, run the extract through a micro column of glass wool and sodium sulfate.

8.24 Transfer the extract to a 10ml concentrator tube. Rinse the TurboVap tube with a few drops of hexane and transfer it to the concentrator tube. Adjust the final volume to 10.0ml. Use a transfer pipet to thoroughly mix the extract. Be sure to record the final volume on the prep sheet.

8.25 If the extract is to be analyzed for pesticides, transfer some of the extract to an appropriately labeled 2.0ml screw cap vial. This is kept as a backup.

8.26 Proceed to section 9.0 for acid cleaning procedure and section 10.0 for sulfur cleaning procedure. The PCB fraction undergoes both the acid and sulfur cleanup procedures. The pesticide fraction undergoes only the sulfur cleanup procedure.

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9.0 Acid Cleaning Procedure (PCB only)

NOTE: This cleaning procedure can be used on the PCB fraction only. It will destroy the pesticides.

9.1 Label the 8ml screw cap vials for each sample and associated QC.

9.2 Add 2ml of concentrated H_2SO_4 to each of the vials.

9.3 Using a transfer pipet, transfer approximately 2ml of extract to the vial. Cap the vial and shake for one minute.

9.4 Allow the extracts to stand for 10 minutes. Transfer the hexane layer (top) to an appropriately labeled 2.0ml screw cap vial.

9.5 Store the extracts in the “extract refrigerator” until they are needed for analysis.

10.0 Sulfur Cleaning Procedure (Pesticide and PCB)

10.1 Label the 16ml screw cap vials for each sample and associated QC.

10.2 Add 1ml of TBA-sulfite reagent and 2ml of isopropyl alcohol to each of the vials.

10.3 Using a transfer pipet, transfer approximately 2ml of extract to the vial. Cap the vial and shake for one minute.

10.4 Add 5ml of reagent water to each vial. Cap the vial and shake for one minute.

10.5 Allow the extracts to stand for 10 minutes. Transfer the hexane layer (top) to an appropriately labeled 2.0ml screw cap vial.

10.6 Store the extracts in the “extract refrigerator” until they are needed for analysis.

11.0 Quality Assurance and Quality Control

11.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

11.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

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12.0 Safety and Waste Disposal

12.1 Safety

12.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

12.1.2 **Hearing protection** must be worn while operating the sonic disrupters. The high frequency could cause permanent hearing loss.

12.1.3 Avoid touching the disrupter horns while they are active. Contact may cause burns or tissue damage.

12.1.4 Material Safety Data Sheets (MSDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS prior to using any new reagents or solvents.

12.1.5 Methylene chloride is an inhalation hazard and a suspected carcinogen. Fume hoods must be used to minimize exposure to vapors.

12.2 Waste Disposal

12.2.1 Waste methylene chloride is placed in the “chlorinated waste” container.

12.2.2 Waste acetone is placed in the “non-chlorinated waste” container.

12.2.3 Extracted soil samples are placed in a waste container after the solvent has drained or evaporated.

12.2.4 Waste soil from the homogenizing process should be place in the “soil waste” container. **Note:** Waste soil from foreign soils must follow “foreign soil” disposal requirements.

13.0 References

13.1 SW-846 Method 3500B, Rev. 2, 12/96

13.2 SW-846 Method 3550B, Rev. 2, 12/96

13.3 SW-846 Method 8081A, Rev. 1, 12/96

13.4 SW-846 Method 8082, Rev. 0, 12/96

13.5 SW-846 Method 3660B, Rev. 2, 12/96

13.6 SW-846 Method 3665A, Rev. 1, 12/96

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METALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

Prepared by: ____Dave Metzgar_____ Date: __4-4-06__

Reviewed by: _____Norm Farmer_____ Date: __4-4-06__

Annual Review

Reviewed by:_____ Date: _____

Reviewed by:_____ Date: _____

Reviewed by:_____ Date: _____

Document Control

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TEST NAME: METALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

INSTRUMENT: TJA TRACE 61E, SERIAL # 470790 – TRACE 1
INSTRUMENT: TJA TRACE 61E, SERIAL # 348490 – TRACE 2
AUTOSAMPLER: TJA 300 POSITION, SERIAL #C2389-TRACE 1
AUTOSAMPLER: TJA 300 POSITION, SERIAL # G7245-TRACE 2

METHOD REFERENCES: SW846 6010B, EPA 200.7 Rev 4.4 1994

DEPARTMENT: Metals

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is applicable for the determination of metals in water, sludges, sediments, and soils. Elements that can be reported by this method include: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Tin, Vanadium, and Zinc.

1.2 Sample matrices are pretreated following SW846 methods for digestion of soil, sediment, sludge or water samples. Refer to specific digestion SOP's for more information on digestion techniques.

1.3 Digested samples are introduced into the TJA Trace ICP, passed through a nebulizer and transported to a plasma torch. The element-specific emission spectra generated are dispersed by a grating spectrophotometer and photosensitive devices measure the intensities of the emission lines.

1.4 Reporting limits (RL) are based on the extraction procedure. Reporting limits may vary depending on matrix complications and volumes. Solid matrices are reported on a dry weight basis.

1.5 MDLs should be established for all analytes, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year for each matrix and instrument. Please refer to Accutest QA SOP QA020, current version.

1.6 An MDL check standard will be analyzed at the time of the annual MDL study for verification. The concentration of the MDL check standard should be 2x-3x the statistical MDL. This is a qualitative check; therfore, the analyte needs to be detected only. If the analyte is not detected, the

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concentration of the MDL check standard must be increased to a level where the analyte is detected. This then becomes the current MDL.

1.7 MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.8 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

2.0 PRESERVATION

All water samples must be preserved with nitric acid to a pH of less than 2. All soil samples must be stored in a refrigerator at 4°C before digestion.

3.0 HOLDING TIME

All samples must be analyzed within 6 months of the date of collection.

4.0 INTERFERENCES

Several types of interferences can cause inaccuracies in trace metals determinations by ICP. These interferences are discussed below.

4.1 Spectral interferences are caused by overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, and background contribution from stray light from the line emission of high concentration elements. Corrections for these interferences can be made by using interfering element corrections, by choosing an alternate analytical line, and/or by applying background correction points.

4.2 Physical interferences can be caused by changes in sample viscosity or surface tension, by high acid content in a sample, or by high dissolved solids in a sample. These interferences can be reduced by making sample dilutions or by analyzing a sample using the method of standard additions.

4.3 Matrix interferences in high solid samples can be overcome by using an internal standard. Yttrium is used for the TJA Trace 61E. The concentration should be sufficient for optimum precision but not so high as to alter the salt concentration of the matrix. The element intensity is used

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by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation.

4.4 Chemical interferences are not pronounced with ICP due to the high temperature of the plasma, however if they are present, they can be reduced by optimizing the analytical conditions (i.e. power level, torch height, etc.).

5.0 APPARATUS

Currently there are two Thermo Jarrell Ash Trace simultaneous ICP's available for use in the lab. The Trace unit has been optimized to obtain lower detection limits for Arsenic, Selenium, Antimony, Lead, and Thallium. This optimization was done using a horizontal torch, a purge optics system, optimized photomultiplier tubes, and Crawford-Kunselman Noise Reduction techniques. The lines programmed into the Trace system are shown in Table 2. Additional elements and lines may be analyzed using an off-peak analysis function.

6.0 REAGENTS

All chemicals listed below are trace metal grade unless otherwise specified. Distilled, deionized water must be used whenever water is required.

6.1 5 ppm Yttrium internal standard, made from ICP quality standard.

6.2 Hydrochloric acid, trace metals grade.

6.3 Nitric Acid, trace metals grade.

6.4 Standard stock solutions are available from Inorganic Ventures, Spex, Plasma Pure, Ultra, Environmental Express, or equivalent.

Note: All standards must be ICP quality standards.

6.5 Calibration Standards. These can be made up by diluting the stock solutions to the appropriate concentrations.

Note: Standards and prepared reagents must be prepared every 6 months or before stock standard expiration date, whichever comes first.

6.5.1 For calibration and quantitation an internal standard (Yttrium) is used to limit nebulization problems. If it is known that the samples contain a significantly different acid matrix, the samples should be

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diluted so that they are in a similar matrix to the curve. If sample concentrations are greater than the linear range, the samples will be diluted and re-analyzed. All sample results are referenced to the calibration blank Yttrium counts. The criteria is 60-125 percent of the calibration blank counts. If the Yttrium counts fall outside these criteria matrix effects must be suspected and the sample diluted until it meets the criteria or footnoted in LIMS as suspected matrix interference.

6.5.2 Standards must be prepared so that there is minimal spectral interference between analytes.

6.5.3 Refer to the standards logbook for the make-up and concentrations of standards and stock solutions being used to calibrate the ICP. Refer to tables 2 through 7 for concentration levels of standards used. Unless otherwise approved, the calibration curve must contain 3 points determined by a blank and a series of standards (mid and high) representing the elements of interest.

Note: All Ag stock and intermediate solutions must be stored away from direct sunlight.

6.6 Analytical Quality Control Solutions.

All of the solutions below are prepared by adding either mixed or single element metals solutions to a solution containing 3 percent nitric acid and 5 percent hydrochloric acid and diluting to a fixed final volume with this acid mixture.

6.6.1 Blank (Calibration, ICB, CCB)

This reagent contains Nitric Acid at 3 percent, and Hydrochloric Acid at 5 percent.

6.6.2 Initial Calibration Verification solution.

This standard solution must be made from a different source than the calibration curve. The values for each element must be within the range of the calibration curve. This solution is used to verify the accuracy of the initial calibration. Levels for the ICV standard are shown in Table 4.

6.6.3 Continuing Calibration Verification solution.

The metals concentrations for this standard must be at the mid-point of the calibration curve for each element. Levels for the CCV standard are shown in Table 5.

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6.6.4 Interference Element Check Solutions.

These solutions must be analyzed to check the interfering element corrections on the instruments.

Note: If interferences from different elements than those listed below are a problem, the interfering element solutions may be modified.

6.6.4.1 ICSA Solution.

The ICSA solution contains only the interfering elements. Concentrations are shown below.

Al	**500 mg/L**
Ca	**500 mg/L**
Fe	**200 mg/L**
Mg	**500 mg/L**

6.6.4.2 ICSAB Solution.

The ICSAB solution contains both the interferents and the analytes of interest. Concentrations are shown below.

Ag	**1.0 mg/L**	**Al**	**500 mg/L**
Ba	**0.50 mg/L**	**Ca**	**500 mg/L**
Be	**0.50 mg/L**	**Fe**	**200 mg/L**
Cd	**1.0 mg/L**	**Mg**	**500 mg/L**
Co	**0.50 mg/L**	**V**	**0.50 mg/L**
Zn	**1.0 mg/L**	**Pb**	**1.0 mg/L**
Cr	**0.50 mg/L**		
Cu	**0.50 mg/L**		
Mn	**0.50 mg/L**		
Ni	**1.0 mg/L**		

6.7 CRIA and CRI Standards Solution

The CRIA standard contains the elements of interest at levels equal to our quantitation limits (RL). The CRI standard contains the elements of interest at levels two times our quantitation limits (RL). Levels for the CRI and CRIA standards are shown in Table 6.

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6.8 Matrix Spike, Matrix Spike duplicate, and Spike Blank Solution.

This solution is prepared by adding either mixed or single element metals solutions to a solution containing 3 percent nitric acid and 5 percent hydrochloric acid and diluting to a fixed final volume with this acid mixture. One milliliter of this stock solution should be added to the spike blank, matrix spike, and the matrix spike duplicate before they are digested and brought to a final volume of 50 ml. Levels for the MS and MSD and Spike Blank standard are shown in Table 7.

6.9 Liquid Argon or Argon Gas. (99.999% purity)

6.10 Yttrium Internal Standard.

For the TJA Trace, an ICP quality yttrium internal standard is used.

7.0 PROCEDURE

NOTE: Please refer to section 8 (QUALITY CONTROL) of this SOP for further detail on quality control standards.

General procedures on how to operate the TJA Trace are described below. The ThermoSpec software is used to run ICP instrument and it can be accessed either in a Windows environment or from a DOS environment.

IMPORTANT: When leaving most menus in the ThermoSpec software, an F9 command is required to save the changes. In order to avoid losing information, make sure that you exit screens by first using this Done/Keep command (F9).

7.1 Before bringing up the instrument, make sure that the lines, the nebulizer, and the spray chamber are clean and that there are no leaks in the torch area. Change the pump tubing if worn. Check Argon tank for adequate pressure to last a full day of analysis.

IMPORTANT: The purge TJA Trace requires that the argon be continuously flowing through optics even when the instrument is not being used. The waste reservoir should also be checked to ensure that it doesn't overflow. Empty reservoir only to level that would enable waste tubing to remain submerged.

7.2 Turn on the re-circulating cooler.

7.3 Tighten the clamps on the peristaltic pump.

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7.4 Put a new solution of acid rinse into the rinse reservoir. (**Note: the composition of the rinse solution may be periodically changed to minimize sample introduction problems and sample carryover**.)

7.5 Enter the Thermospec software and go to the SETUP menu. Then move to the Plasma Control Panel option. Ignite the instrument by entering Start-up (F1) and then enter F9 to continue. After the plasma is ignited wait about 15 seconds for plasma to stabilize. Hit Esc to go back to main menu and go into analysis to set plasma parameters. This allows plasma parameters (i.e.- aux. Pressure, pump rate, etc.) to be set according to levels listed in the method being used.

7.6 Make sure that the sample tip is in the rinse solution and then let the instrument warm up for at least 60 minutes. If a yttrium internal standard is being used, make sure that sufficient solution is in place to last the length of the run and that the standard is being properly introduced into the sample introduction system. Check with the lab supervisor for additional details.

NOTE: While the instrument is warming up, the samples that are to be analyzed should be collected together along with the required quality control. A copy of the metals prep batch should be included with each run.

7.7 After the instrument has warmed up, the nebulizer should be optimized. The procedures for the TJA Trace are given below.

7.7.1 For the TJA Trace, the nebulizer position and pressure should also be adjusted to achieve the highest signal to background ratio. To do this, go the method OPTNEB. Adjust the nebulizer pressure to the point between 25 and 35 PSI (normally approximately 28 psi) where the intensity is highest in relation to the lowest RSD. Then physically adjust the glass nebulizer in the spray chamber by rotating it or moving it in and out until the highest intensity is obtained for the 5 ug/ml As standard. This procedure is to be used only when the nebulizer is removed from the spray chamber.

7.7.2 For the TJA Trace, it is recommended that the torch placement optimization be checked whenever the torch is removed and replaced or whenever there is a decrease in sensitivity. This can be done using the TA program :\STATION\BIN>TA. Aspirate a blank into the instrument and measure the intensity using the TA program. Adjust the torch position using the manual thumbscrews in the compartment to the left of the nebulizer compartment. The torch should be positioned to obtain the lowest intensity reading for the blank.

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7.8 Next, profile the instrument by aspirating a 5 ppm As solution. Normally, the automatic profile program will properly profile the instrument. However, the manual profile program can be used if the instrument profile has drifted significantly. The profile can be done as an option from the SETUP menu or an option from the OPERATION menu. The peak width, peak intensity, peak position, analyst's initials, and date are recorded in a logbook after each profile has been accepted.

7.9 Move to the DEVELOPMENT menu and go to the Methods option. Open the analytical program that you will be using and move to the Method Info option (F3). Type in the file name that will be used to save the run data next to the space marked Analysis Data File. The name should be in the form of instrument ID, date in the order month/date, matrix identifier, and run number. For example, the first mixed (water and soil) analysis on Dec. 1 on the TJA Trace would be designated R1201M1.

7.10 Go to the OPERATION menu. If you are going to be running samples using the auto sampler, then set up the auto sampler table to be used for the analysis using the auto sampler setup option. Make sure to reference the proper analysis program in the auto sampler table. Also make sure to include all dilutions factors for serial dilutions or other required dilutions in the auto sampler table. **(Note: You can set up a new auto sampler table for another run while your current run is going using Windows. When the software is properly set up, Alt/Tab will take you from Thermospec into Windows.)**

7.11 Calibrate the instrument. Unless otherwise approved, the calibration curve must contain 3 points determined by a blank and a series of standards representing the elements of interest. Three exposures will be used with a %RSD <5%. The resulting correlation coefficient must be $\geq$0.995. If the calibration curves do not meet these criteria, analysis must be terminated, the problem corrected, and instrument re-calibrated. (**Note: the calibration standards may be included in the auto sampler program or they may be run separately.) Also see table 8 for typical run sequence.)**

7.12 After the instrument is properly calibrated, begin by reanalyzing the high standard(s) for each element. **(Note: The standards can be combined into one solution for this analysis)**. The analyzed value must be within 5 percent of the true value or that element must be re-calibrated. High standard shall be used for 200.7 only. After the high standards are analyzed, the ICV check standard shall be run. For the ICV, all elements to be reported must be within 5 percent for 200.7 and 10 percent for 6010B of the true values. If the ICV fails, analysis shall be terminated and the instrument re-calibrated.

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7.13 After analyzing the ICV, the ICB must be analyzed. The results of the ICB must be less than one half the reporting limit. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit for all samples to greater than two times the background concentration.

7.14 Before analyzing any real samples the CRI must be analyzed. The CRI contains the elements of interest at two times the reporting limit. For all elements the results must be within 30 percent of the true value. If the CRI fails, analysis should be terminated and the instrument re-calibrated. The only exception is if the CRI fails high and all sample results are less than the reporting limit. In this case the results can be reported and footnoted “possible positive bias, but all sample results are <RL.”.

7.15 A low level CRIA will also be analyzed at the reporting limit. The CRIA acceptance criteria are within 30 percent for all elements. If the CRIA fails high and all sample results are less than the reporting limit the results can be reported and footnoted “possible positive bias, but all sample results are <RL.” If the CRIA fails low, all sample and instrument RL’s will be elevated to the CRI level for the entire analytical run.

7.16 Before analyzing any real samples, the interference check standards (ICSA, ICSAB) must be analyzed. For all spiked elements, the analyzed results must be within 20 percent of the true results. For unspiked elements, the interfering element solutions must be +/- the reporting limit for each element.

7.17 After the initial analytical quality control has been analyzed, the samples and the preparation batch quality control shall be analyzed. Each sample analysis should be a minimum of 3 readings using at least a 5 second integration time. Between each sample, flush the nebulizer and the solution uptake system with a blank rinse solution for at least 90 seconds or for the required period of time to ensure that analyte memory effects are not occurring.

7.18 Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value, for method 6010B and 5 percent for method 200.7, the CCV shall be reanalyzed to confirm the initial value. If the CCV is not within criteria of the true value after the reanalysis, no samples can be reported in the area bracketed by the failing CCV. Immediately following the analysis of the CCV the CCB shall be analyzed. The results of the CCB must be less than one half the reporting limit for all elements. The instrument blank may be failing the

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criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit for all samples to greater than two times the background concentration.

7.19 One sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution should be prepared. For the serial dilution, a 1:5 dilution should be made on the sample. The results of the 1:5 dilution shall agree within 10 percent of the true value as long as the sample and the dilution result are greater than 10 times the method detection limit or greater than 50 times the IDL. If the results are outside these criteria then the proper footnote should be entered into LIMS.

7.20 For any readings that exceed the upper range limit for a given element, the sample shall be diluted and re-analyzed. After a high reading, the sample following the high one must be examined for possible carryover. Verification may be done by rinsing the lines with an acid solution and then reanalyzing the sample. A limit check table may be built into the autosampler file so that samples exceeding the standardization range are flagged on the raw data.

7.21 After the run is completed, convert the data file to an ASCII format using the Enable Program installed on the ICP computer. The data can be converted using the form EXTRACT.WPF. Save the converted file on a network drive(C:\FILENAME.ASC) using the file naming system described above, adding the designation “IR” at the beginning of the filename to designate ICP.

7.22 The data can be evaluated by running an automated data evaluation program, which will help to generate quality control summary pages. Each run should be evaluated as quickly as possible to make sure that all required quality control has been analyzed.

7.23 At the end of the analysis day the ICP must be shutdown using the following sequence.

a) Rinse tip in a solution of 3 percent nitric acid, 5 percent HCl for 10 minutes and in DI water for 20 minutes. **(Note: a stronger acid solution may be needed depending on the matrix of the samples that were analyzed.)**

b) Turn off the plasma using the Plasma Control Option under the SETUP menu. F7 initiates the automatic shutdown procedure.

c) Release the tension on the pump tubing.

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d) Turn off the cool flow.

e) Turn off the monitor.

8.0 QUALITY CONTROL

This section outlines the QA/QC operations necessary to satisfy the analytical requirements for method SW846 6010B.

8.1 High Standard Check.

After the instrument is properly calibrated, the high standard(s) shall be reanalyzed for each element. **(Note: The standards can be combined into one solution for this analysis)**. The analyzed value must be within 5 percent of the true value or that element should be re-calibrated. (Note: For method 200.7 only.)

8.2 Initial Calibration Verification Standard (ICV).

After each calibration, a standard from a different source than the calibration standard shall be analyzed. For the ICV, all elements to be reported must be within 10 percent of the true value for 6010B and within 5 percent for 200.7. If the ICV is outside these criteria then the analysis must be terminated and the instrument re-calibrated.

8.3 Continuing Calibration Blank/Initial Calibration Blank.

Analyze the Initial calibration blank solution at the beginning of each run and the continuing calibration blank after every tenth sample and at the end of the sample run. The ICB/CCB must be less than one half the reporting limit for each element. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit for all samples to greater than two times the background concentration.

8.4 Low Standard Check (CRI).

The CRI standards contain the elements of interest at two times the reporting detection limit. The CRI acceptance criteria are at 30 percent for all elements. If the CRI fails, analysis should be terminated and the instrument re-calibrated. The only exception is if the CRI fails high and all sample results are less than the reporting limit. In this case the results can

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be reported and footnoted "possible positive bias, but all sample results are <RL.". The CRI will be analyzed at the beginning and end of each analytical run. If the closing CRI fails the criteria, the samples associated with the CRI shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the CRI, or qualifying the results in LIMS.

8.5 Low Level Check (CRIA).

The CRIA contains elements of interest at the reporting limit. The CRIA acceptance criteria are at 30 percent for all elements. If the CRIA fails high and all sample results are less than the reporting limit the results can be reported and footnoted "possible positive bias, but all sample results are <RL." If the CRIA fails low, all sample and instrument RL's will be elevated to the CRI level for the entire analytical run. The CRIA will be analyzed at the beginning and end of each analytical run. If the closing CRIA fails the criteria, the samples associated with the CRIA shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the CRIA, or qualifying the results in LIMS.

NOTE: CRI does not need to be analyzed at the beginning of the run if the CRIA passes all criteria.

8.6 ICSA and ICSAB

Analyze the ICSA and ICSAB at the beginning and end of each run following the analysis of the CRI. For all spiked elements, the analyzed results must be within 20 percent of the true results. For unspiked elements, the interfering element solutions must be ± the reporting limit for each element. If the ICSA and/or the ICSAB falls outside this criteria the problem must be corrected and the instrument re-calibrated or data footnoted in LIMS system. If the closing ICSA/ICSAB fails the criteria, the samples associated with the ICSA/ICSAB shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the ICSA/ICSAB, or qualifying the results in LIMS.

8.7 Continuing Calibration Verification.

Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value, for method 6010B and 5 percent for method 200.7 the CCV must be reanalyzed to confirm the initial value. If the CCV is not within criteria of the true value after the reanalysis, no samples can be reported in the area bracketed by the failing CCV.

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8.8 Method Blank.

The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every batch. The method blank must contain elements at less than one half the reporting limit for each element. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit. Samples associated with the contaminated blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-digesting and reanalyzing the samples, qualifying the results with a "B" or "V" qualifier, or raising the reporting limit to greater than two times the background concentration,

8.9 Blank Spike Sample.

The laboratory must digest and analyze a spike blank sample with each set of samples. A minimum of one spike blank sample is required for every 20 samples. Blank Spikes must be within 20 percent of the true value for method SW846-6010B and within 15 percent for method EPA 200.7. If the lab control is outside of the control limits for a reportable element, all samples must be re-digested and reanalyzed for that element. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results may be reported with no flag.

8.10 Matrix Spike and Matrix Spike Duplicate.

The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below and should be within 20 percent of the true value for method SW846-6010B and within 30 percent for method EPA 200.7. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect.

Note: Both the matrix spike amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{Amount Spiked}} \times 100 = \text{matrix spike recovery}$$

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8.11 Matrix Duplicate.

The laboratory must digest a duplicate sample for a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed and should be $\leq$ 20 percent. If a duplicate is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of $\pm$ the reporting limit, then the duplicate is considered to be in control.

Note: Both the duplicate amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

8.12 Serial Dilution Analysis.

For one sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution should be prepared. For the serial dilution, a 1:5 dilution should be made on the sample. The results of the 1:5 dilution should agree within 10 percent of the true value as long as the sample and the dilution result are greater than 10 times the method detection limit and/or greater than 50 times the IDL. If the dilution does not agree, then the sample should be post –digestion spiked at a level no less than 10 times but no greater than 100 times the MDL concentration or flagged as interference. Serial dilution is performed instead of post digestion spike in accordance with method.

Alternatively, in cases where a sample has demonstrated physical or chemical interferences, which preclude the sample from being reported without a dilution, a serial dilution can be done from the diluted sample, and the sample can be reported from the dilution. For example, a sample that failed the serial dilution criteria using the undiluted sample and a 1:5 dilution, may pass the serial dilution criteria using a 1:2 dilution and a 1:10 dilution. In that case, the sample would be reported from the 1:2 dilution and the results would be footnoted that a dilution was required due to matrix interference. The calculation to be used for serial dilutions is shown below.

$$\frac{\text{(Sample Result - Serial Dil. Result)}}{\text{Sample Result}} \times 100 = \text{Serial Dilution RPD}$$

NOTE: An Alternate to using the method of standard additions is the internal standard technique. (SW846-6010B, Rev2, 12/96, page 15).

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8.13 Linear Calibration ranges.

The upper limit of the linear calibration ranges must be established for Aluminum, Calcium, Iron, and Magnesium by determining the signal responses from a minimum of three concentration standards, one of which is close to the upper limit of the linear range. The linear calibration range, which may be used for the analysis of samples should be judged by the analyst from the resulting data. Linear calibration ranges should be determined whenever there is a significant change in instrument response and every 12 months for those analytes that periodically approach their linear limit. The upper limit of the linear calibration range for all other elements shall be the highest calibration standard. Please refer to Table 3 for high calibration standard concentrations.

9.0 GLASSWARE CLEANING

All glassware must be washed with soap and tap water and then rinsed with 5 percent nitric acid. It must then be rinsed at least 3 times with deionized water.

10.0 DOCUMENTATION REQUIREMENTS

Refer to the Laboratory Quality Assurance Manual for documentation requirements.

11.0 SAFETY

The analyst must follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor. Follow proper safety precautions when working with gas cylinders.

12.0 CALCULATIONS

For water samples, the following calculations should be used. Refer to the QC section for the calculations to be used for the QC samples.

original sample concentration of metal (ug/l) =

$$\frac{\textbf{(conc. in the digestate (ug/l)) x (final digestate volume (ml))}}{\textbf{(initial sample volume (ml))}}$$

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For soil samples, the following calculations should be used.

concentration of the metal in the dry sample (mg/kg) =

$$\frac{\textbf{(conc. in the digestate (mg/l) x final digestate volume(L))}}{\textbf{(sample wt. (kg)) x (\% solids/100)}}$$

13.0 INSTRUMENT MAINTENANCE

Recommended periodic maintenance includes the items outlined below. All maintenance must be recorded in the instrument maintenance log.

13.1 Change the pump tubing weekly or as needed.

13.2 Clean the filter on the recirculating pump approximately once a month and dust off the power supply vents as needed.

13.3 Clean the nebulizer, torch, and injector tube as needed.

13.4 Change the sampler tip as needed .

13.5 Clean the re-circulating pump lines every 3 months or as needed.

13.6 Clean the slides on the autosampler with methanol and wipe them with a KimWipe saturated with Teflon spray as needed.

13.7 For the TJA Trace, check and clean the following filters as needed:

- 2 filters on the back of the polychromator controller compartment

- one filter on the back of the power source

- one filter below the torch compartment

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 14.2.

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14.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples. All ICP waste must be analyzed prior to disposal. The results must be under the TCLP Maximum Contamination Levels (MCL), if they are not then the waste must be labpacked and considered hazardous waste. After waste passes TCLP criteria it can be neutralized and disposed of.

15.0 DEFINITIONS

15.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours which ever comes first.

15.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

15.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run.

15.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

15.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

15.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

15.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

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15.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and bias of a method in a given sample matrix.

15.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

15.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

15.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

16.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

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TABLE 1: REPORTING LIMIT BY ELEMENT

Analyte	Water Reporting Limit (ug/L)	Soil Reporting Limit (mg/kg)	TCLP Reporting Limit (mg/L)
Tin	50	5	
Aluminum	200	20	
Antimony	5	6	
Arsenic	10	0.5	0.05
Barium	200	20	1.0
Beryllium	4	0.5	
Cadmium	5	0.4	0.005
Calcium	1000	500	
Chromium	10	1	0.010
Cobalt	50	5	
Copper	25	2.5	
Iron	300	10	
Lead	5	10	0.05
Magnesium	5000	500	
Manganese	15	1.5	
Nickel	40	4.0	
Potassium	5000	500	
Selenium	10	1	0.05
Silver	10	1	0.010
Sodium	5000	500	
Thallium	10	1	
Vanadium	50	5	
Zinc	20	2	
Molybdenum	50	5	

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TABLE 2. TJA TRACE ANALYSIS LINES

Element	Wavelength
Al	308.215
As	189.042
Ca	317.933
Fe	271.441
Mg	279.078
Mn	257.610
Pb	220.353 (a)
Se	196.026 (a)
Tl	190.864
V	292.402
Ag	328.068
Ba	493.409
Be	313.042
Cd	226.502
Co	228.616
Cr	267.716
Cu	324.753
K	766.491
Na	588.991
Ni	231.604
Sb	206.838
Zn	213.856
Mo	202.030
Sn	189.989

(a) This element has two lines at this wavelength so that Crawford-Kunselman Noise Reduction techniques can be applied.

TABLE 3: HIGH STANDARD LEVELS

NOTE: MID LEVEL STANDARD IS ½ THE CONCENTRATION OF THE HIGH STANDARD

Element	Conc. ug/l
Al	80000
As	4000
Ca	80000
Fe	80000
Mg	80000
Mn	4000
Pb	4000
Se	4000
Tl	4000
V	4000
Ag	500
Ba	4000
Be	4000
Cd	4000
Co	4000
Cr	4000
Cu	4000
K	80000
Na	80000
Ni	4000
Sb	4000
Zn	4000
Mo	4000
Sn	4000

TABLE 4: ICV STANDARD LEVELS

Element	Conc. ug/l
Al	40000
As	2000
Ca	40000
Fe	40000
Mg	40000
Mn	2000
Pb	2000
Se	2000
Tl	2000
V	2000
Ag	250
Ba	2000
Be	2000
Cd	2000
Co	2000
Cr	2000
Cu	2000
K	40000
Na	40000
Ni	2000
Sb	2000
Zn	2000
Mo	2000
Sn	2000

TABLE 5: CCV STANDARD LEVELS

Element	Conc.l ug/l
Al	40000
As	2000
Ca	40000
Fe	40000
Mg	40000
Mn	2000
Pb	2000
Se	2000
Tl	2000
V	2000
Ag	250
Ba	2000
Be	2000
Cd	2000
Co	2000
Cr	2000
Cu	2000
K	40000
Na	40000
Ni	2000
Sb	2000
Zn	2000
Mo	2000
Sn	2000

TABLE 6: CRI STANDARD LEVELS

NOTE: CRIA AT 1/2 THESE LEVELS

Element	Conc. ug/l
Al	400
As	20
Ca	2000
Fe	600
Mg	10000
Mn	30
Pb	10
Se	10
Tl	20
V	100
Ag	20
Ba	400
Be	10
Cd	10
Co	100
Cr	20
Cu	50
K	10000
Na	10000
Ni	80
Sb	10
Zn	40
Mo	100
Sn	100

TABLE 7: BLANK SPIKE, MATRIX SPIKE AND MATRIX SPIKE DUPLICATE LEVELS

Element	Conc. ug/l
Al	27000
As	2000
Ca	25000
Fe	26000
Mg	25000
Mn	500
Pb	500
Se	2000
Tl	2000
V	500
Ag	50
Ba	2000
Be	50
Cd	50
Co	500
Cr	200
Cu	250
K	25000
Na	25000
Ni	500
Sb	500
Zn	500
Mo	500
Sn	500

TABLE 8: TYPICAL RUN SEQUENCE

BLANK **NOTE: <5%RSD (3 EXPOSURES) AND CORRELATION COEFFICIENT ≥0.995**
MID
HIGH

HIGH STD
ICV
ICB
CRIA
CRI
ICSA
ICSAB
CCV
CCB
MB
SB
SAMPLE1
DUPLICATE
SERIAL DILUTION
MATRIX SPIKE
MATRIX SPIKE DUPLICATE
SAMPLE2
SAMPLE3
SAMPLE4
CCV
CCB
SAMPLE5
SAMPLE6
SAMPLE7
SAMPLE8
SAMPLE9
SAMPLE10
SAMPLE11
SAMPLE12
SAMPLE13
SAMPLE14
CRIA
CRI
ICSA
ICSAB
CCV
CCB
ETC.

COLD VAPOR ANALYSIS OF MERCURY FOR SOILS

Prepared by: ____Dave Metzgar_____ Date: __4-4-06__

Reviewed by: _____Norm Farmer_____ Date: __4-4-06__

Annual Review

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TEST NAME: COLD VAPOR ANALYSIS OF MERCURY FOR SOILS

METHOD REFERENCE: SW-846 7471A

INSTRUMENT SERIAL #: 2022

DEPARTMENT: METALS

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is outlined in this SOP is based on SW846 method 7471A for soils. The types of samples that can be analyzed include soils, sediments, bottom deposits and sludge type materials.

1.2 The mercury is reduced to the elemental state and aerated from the solution in a closed system. The mercury vapor passes through a cell in the light path of an atomic spectrophotometer, where the absorbance is measured as a function of mercury concentration.

2.0 PRESERVATION

All soils must be refrigerated between 1 - 4°C.

3.0 HOLDING TIME

All samples should be analyzed within 28 days of the date of collection.

4.0 REPORTING and METHOD DETECTION LIMIT

4.1 Reporting Limit. The reporting limit for this method has been established at 0.083mg/kg. Reporting limits (RL) are based on the extraction procedure. Reporting limits may vary depending on matrix complications and volumes. Solid matrices are reported on a dry weight basis.

4.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria. Experimental MDLs must be determined annually for this method.

4.3 MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit annually. To determine the MDL values, take seven replicate aliquots of the spiked

sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level.

4.4 An MDL check standard will be analyzed at the time of the annual MDL study. The concentration of the MDL check standard should be 2x-3x the statistical MDL. This is a qualitative check; therefore, the analyte needs to be detected only. If the analyte is not detected, the concentration of the MDL check standard must be increased until the analyte is detected. This then becomes the current MDL.

4.5 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

4.6 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

5.0 INTERFERENCES

Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations of sulfide as sodium sulfide as high as 20 mg/l do not interfere with mercury recoveries when following this method. High copper concentrations (> 10 mg/l) may also interfere with mercury recoveries. Samples that are high in chloride such as seawater, brine and industrial effluent may require as much as 12.5 ml of additional permanganate.

Note: When chloride concentrations are high, hydroxylamine sulfate and stannous sulfate should be used in place of corresponding chlorides. Finally, certain volatile organic materials will also absorb at this wavelength and can interfere. It can be determined if this type of interference is present by doing a preliminary run without reagents.

6.0 APPARATUS

6.1 A Leeman HYDRA AA automated analyzer is used for all analysis. Refer to the instrument manual for further details on this instrumentation.

6.2 Environmental Express Hot Block or equivalent.

6.3 Environmental Express digestion vessels or equivalent, 50ml capacity.

6.4 Class A volumetric pipettes and flasks

6.5 Thermometer

6.6 Fisherbrand wooden tongue depressors

6.7 Teflon boiling chips

7.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. De-ionized water should be used whenever water is required.

7.1 Sulfuric acid, concentrated

7.2 Hydrochloric acid, concentrated

7.3 Nitric acid concentrated. This acid must have low mercury content.

7.4 Stannous chloride. To 400 ml of DI water, add 14 ml of conc. sulfuric acid. Dilute to 500 ml and add 50 g of stannous chloride and dissolve. This compound does not dissolve well and should be stirred continuously when in use. Stannous sulfate may be used in place of stannous chloride. Stannous chloride is prepared daily. Lot number is recorded in Hg logbook.

7.5 Sodium chloride-hydroxylamine hydrochloride. Add 120 g of sodium chloride and 120 g of hydroxylamine hydrochloride to 1 liter of water. Mix well; hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.6 Potassium permanganate. Add 50 g of potassium permanganate to 1 liter of water and mix well.

Caution: Potassium permanganate is a strong oxidizing agent. Handle with care.

7.7 2 percent HCL Rinse Solution. Add 40ml concentrated HCL to a volumetric flask and dilute to 2 liters with DI water. 2 percent HCL is prepared daily. Lot number is recorded in Hg logbook.

7.8 Daily working standards. Mercury standard solutions are made from a purchased stock solution of 1000 ppm mercury.

7.8.1 10 ppm Hg solution intermediate 1. (Calibration curve, CCV's, Blank Spikes, MS, MSD's, CRI) Using a 10-ml volumetric pipette, add 10 ml of 1000-ppm stock (to be purchased from a vendor

such as Fisher) to a 1.00-liter volumetric flask containing approximately 750 ml of water and 10 ml of concentrated nitric acid. Dilute to volume with water and mix well. This 10ppm standard must be prepared every six months.

7.8.2 100 ppb Hg solution intermediate 2. (Calibration curve, CCV's, Blank Spikes, MS, MSD's, CRI) Using a 10-ml volumetric pipette, add 10 ml of 10 ppm Hg solution (intermediate 1) to a 1.00-liter volumetric flask containing approximately 750 ml of water and 10 ml of concentrated nitric acid. Dilute to volume with de-ionized water and mix well. This 100ppb standard must be prepared every month.

7.8.3 ICV working solutions are prepared at the same concentrations as the calibration standards listed above, except they must be from a second source.

7.8.4 Daily working standards are prepared and digested daily using 100ppb standard solutions.

Note: All standards and prepared reagents must be prepared every 6 months or before stock standard expiration date, whichever comes first, except as noted elsewhere in this SOP.

8.0 SOIL DIGESTION AND ANALYSIS PROCEDURE

Below is a step-by-step procedure for the digestion and analysis of soil samples for mercury.

8.1 Make up the standard curve as shown below. Clearly label each digestion vessel with a standard ID. The standard ID's should be recorded on the data summary sheet.

ml of 100 ppb Hg solution	ml of DI water	Total µg of Hg
0.0	50	0.0
0.10	50	0.02
0.5	50	0.10
1.5	50	0.30
2.5	50	0.50
3.0	50	0.60

Add enough DI water to bring the volume up to 10ml in each digestion vessel.

8.2 Make up the quality control samples as shown below. Make sure to clearly label each digestion vessel.

Sample ID	ml of 100 ppb Hg solution	ml of DI water	Total µg of Hg
Blank Spike	1.5	50	0.30
CCV	1.5	50	0.30
Low Check (CRI)	0.10	50	0.020
Method Blank	0.0	50	0.0
ICV	1.5	50	0.30

Sample ID	ml of 100 ppb Hg Solution	g of sample	Total µg of Hg
Matrix Spike	1.5	0.6	0.30 +sample
Matrix Spike Dup	1.5	0.6	0.30 +sample
Duplicate		0.6	sample

8.3 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. Discard the top layer of soil and thoroughly mix the sample with a tongue depressor. Samples with obvious layers may need to be transferred to a larger container for proper mixing. Refer to SOP QA035 for more information on sample homogenization. Using a tongue depressor, weigh out approximately 0.6 g of a homogeneous sample and place in the digestion vessel.

8.4 Preheat the Hot Block to 95 ± 1°C.

8.5 To all samples, QC and standards add the following reagents:

- 0.5 ml of concentrated nitric acid
- 1.5 ml of concentrated hydrochloric acid
- 2.5 ml of DI water

NOTE: the 2.5 ml of DI is not needed for the standards and QC samples already diluted to 10 ml

8.6 Swirl the samples well after each addition of reagent.

8.7 Place the vessels in the Hot Block and digest for 2 minutes at 95° C.

8.8 Cool the samples and then add 18ml of DI water and 7.5 ml of potassium permanganate. More potassium permanganate solution may be required for some samples. Enough should be added so that the purple color persists for at least 15 minutes.

8.9 Heat the samples for 30 minutes at 95 ± 1°C. Then remove and cool.

8.10 Add DI water to bring to a final volume of 50 ml.

8.11 While the samples are digesting, begin setting up the Leeman HYDRA AA mercury analyzer following the steps outlined below. Further details are available in the instrument manual.

8.12 Turn the Argon gas on. Make sure the vent line is connected to the exhaust hood.

8.13 Inspect all pump tubing and replace if necessary. Put the tubing on the cassettes and attach to the pump head, making sure the cassette adjusters are completely tightened. Place the fresh stannous chloride solution in the bottle. Fill the rinse bottle with 2 percent HCL solution.

8.14 Double click on the WINHG icon on the desktop. This will open the WinHg runner. Click on the control tab and click “turn on gas”, “turn on pump”, and “turn on lamp” options.

8.15 Go to file (pulldown menu) and choose new dataset. Save dataset as Hg-day-month-matrix-run no. (i.e., Hg0405S1).

8.16 Click on the **DB** ↓ button. This will open the WinHg Database window. Go to file and choose Hg7470 PROTOCOL. This will be the Protocol (method) used for water and soil analysis. Make sure “Dataset” match on WinHg Runner screen as well as the Database screen.

8.17 Click on the “STANDARD” tab in WinHgRunner window and click on **S1** through **S6.** Click on the “Rep1” button. Click on the “STAND AUTO” button and calibration will begin.

8.18 Calibration is performed by analyzing a series of 5 standards and a blank. While calibration is being performed, type the analysis sequence by clicking on the Rack Editor button. Enter the samples and the appropriate QC. ICV, ICB, CRI, CCV, CCB, 10 samples, CCV, and CCB, etc. Save the sequence as Hg-day-month-matrix-run no. (Hg0405S1). After calibration is performed and accepted **(Correlation Coeff. Of ≥0.995 must be achieved, if not, analysis must be terminated, problem corrected, and instrument re-calibrated.)**, click on the Sample Tab in WinHgRunner and click on “START NEW BATCH” button. Type the auto-sampler sequence filename as above (i.e., Hg0405S1).

8.19 In Autosampler Run rack name drop down window, choose the filename you are analyzing. Assign the location of the start and finish cups and click on “RUN AUTO”.

8.20 Add 3 ml of hydroxylamine hydrochloride solution to each standard and swirl until the solution has been completely decolorized before analyzing standards or samples.

9.0 QC REQUIREMENTS

All QC calculations should be done as outlined in the method. QC limits are compiled by Accutest quarterly and should be used to determine if a given analysis is valid.

9.1 Method Blank – An acceptable method blank or reagent blank must be analyzed with every batch of samples processed. The method blank must be less than one half the reporting limit. If the method blank is greater than one half the reporting limit, the samples associated with the contaminated blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-digesting and reanalyzing the samples, or qualifying the results with a “B” or “V” qualifier.

9.2 Initial Calibration Verification – An initial calibration verification (ICV) sample must be analyzed after the initial calibration has been performed. This sample must be prepared at or near the midpoint of the initial calibration from a reference material independent from the initial calibration solution. The results of the ICV must agree within 10 percent of the true value for the analysis to be valid. If the ICV fails, a new ICV or initial calibration must be performed and all samples must be re-digested and reanalyzed with an acceptable ICV.

9.3 Continuing Calibration Verification – If more than 10 samples are to be analyzed in a single day, a Continuing Calibration Verification sample prepared at or near the mid point of the initial calibration must be analyzed after every 10th sample and at the end of the analytical run. The results of this analysis must be within 20 percent of the initial calibration for the CCV analyses to be considered valid. If the CCV fails, all samples analyzed after the first passing CCV must be re-digested and reanalyzed.

9.4 Continuing Calibration Blank/Initial Calibration Blank – Analyze the Initial calibration blank solution at the beginning of each run and the continuing calibration blank after every tenth sample and at the end of the sample run. If the ICB/CCB is not less than one half the reporting limit for each element the reporting limit for those samples must be raised to greater than 2 times the background concentration. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank or qualifying the results with a “B” or “V” qualifier.

9.5 Low Level Check Standard (CRI) – A standard prepared at the low calibration concentration should be prepared and analyzed with each analytical run, at the beginning and end of each analytical run. The CRI should agree within 20 percent of the true value to be acceptable. If the CRI does not meet the acceptance criteria, the samples must be re-digested and reanalyzed, or footnoted in LIMS.

9.6 Blank Spike – A blank spike (BSP) or Laboratory Control Spike (LCS) should be prepared using de-ionized water spiked at the midpoint of the calibration curve. The blank spike must be within 20 percent of the true value for the analysis to be considered valid. If the blank spike exceeds the acceptance criteria, the samples must be re-digested and reanalyzed. A blank spike is required for every 20 field samples or for each analysis batch.

Note: Samples with Hg non-detected may be reported with Blank Spike recovery biased high.

9.7 Duplicate – A duplicate of a field sample should be processed and analyzed with each set of 20 field samples. The duplicate results should agree within 20 percent of one another for the analyses to be considered valid. If the duplicate fails the acceptance criteria, data must be footnoted in LIMS.

9.8 Matrix Spike/Matrix Spike Duplicate – At least on Matrix Spike/Matrix Spike Duplicate pair must be prepared and analyzed with every 20 field samples. The MS/MSD must agree within 20 percent of the true value of the MS/MSD (20 percent Relative Standard Deviation for the MSD. If the results of the MS/MSD are outside the acceptance criteria, the data should be flagged as possible matrix effect. Alternatively, the use of the Method of Standard Additions (MSA) may be used to calculate the concentration in matrix effected samples.

9.9 When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted and reanalyzed to bring them within the calibration curve.

10.0 DOCUMENTATION REQUIREMENTS

Record the start time, end time and temperature for all digestions on the analysis data sheet. All sample information should be clearly entered on these sheets. In addition, any unusual characteristics of the samples or the digestion procedure should be noted in the comment section. All dilutions must be clearly documented.

11.0 SAFETY

The analyst should follow normal safety procedures as outline in the Accutest Laboratory Safety Manual. Particular care should be observed in handling the strong acids and oxidizing agents. Safety glasses and lab coats should be worn at all times. Gloves should be worn when handling samples and hazardous chemicals.

12.0 CALCULATIONS

Below are the calculations, which should be used for soil samples. The concentration of the sample in µg should be obtained from the calibration curve.

$$\textbf{Final concentration in mg/kg} = \frac{\textbf{Concentration of sample in µg}}{\textbf{(Sample wt in g)(\%solids/100)}}$$

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

13.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids must be followed. All method users must be familiar with the waste management practices described in Section 13.2.

13.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples. Mercury waste must be analyzed prior to disposal. Result must be under TCLP MCL. If result is not under TCLP MCL waste must be considered hazardous and disposed of accordingly. After waste passes TCLP criteria it then can be neutralized and disposed of.

14.0 DEFINITIONS

14.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours which ever comes first.

14.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

14.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run.

14.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

14.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

14.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

14.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

14.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and bias of a method in a given sample matrix.

14.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

14.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

14.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

15.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

DIGESTION OF SOILS FOR ICP ANALYSIS

Prepared by: ____Dave Metzgar_____ Date: __4-4-06__

Reviewed by: _____Norm Farmer_____ Date: __4-4-06__

Annual Review

Reviewed by:_____ Date: _____

Reviewed by:_____ Date: _____

Reviewed by:_____ Date: _____

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Effective 7 days after "*" date

TEST NAME: DIGESTION OF SOILS FOR ICP ANALYSIS

METHOD REFERENCE: 3050B

DEPARTMENT: METALS

REPORTING LIMIT: Not applicable

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is applicable for the digestion of sediments, soils, sludges and solid wastes. After digestion, the samples can be analyzed by ICP. This digestion method is based upon SW846 method 3050B and the CLP soil digestion method.

1.2 An aliquot of a homogenized soil is digested with repeated additions of nitric acid and hydrogen peroxide. The volume is reduced to 5 ml and then hydrochloric acid is added and the sample is refluxed for 15 minutes. The sample is cooled to room temperature and diluted to 50 ml. If particulate matter is present, the sample is filtered.

2.0 PRESERVATION

Non-aqueous samples should be refrigerated at the time of receipt.

3.0 HOLDING TIME

All samples should be digested and analyzed within 6 months of the time of collection.

4.0 INTEREFRENCES

Sludge and soil samples can contain diverse matrix types, which may contain a variety of interference. Spiked samples can be used to determine if this interference is adequately treated in the digestion process. For discussion of other interference, refer to specific analytical methods.

5.0 APPARATUS

The apparatus needed for this digestion procedure are listed below.

5.1 Automatic pipette dispensing bottles

5.2 Filter paper, Whatman #41 or equivalent

5.3 Top loader balance- capable of accurately weighing 0.01g

5.4 Graduated cylinder

5.5 Thermometer- capable of measuring to at least 125^0C

5.6 Environmental Express Hot Block or equivalent capable of maintaining a temperature of 90-95°C.

5.7 Environmental Express digestion cups or equivalent, 50ml capacity.

5.8 Environmental Express Filtermate

5.9 Fisherbrand wooden tongue depressors

6.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. Deionized water should be used whenever water is required.

6.1 Hydrochloric acid, Fisher Trace grade or equivalent

6.2 Nitric acid, Fisher Trace grade or equivalent

6.3 Hydrogen peroxide, 30%

6.4 Metals spiking solutions. CPI Multi element or equivalent made with 5%HNO3 and a trace of HF; Inorganic Ventures 5000 mg/l Mineral solution; and 100 mg/l Mo and Sn solution.

7.0 PROCEDURE

7.1 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. Discard the top layer or soil and thoroughly mix the sample with a tongue depressor. Samples with obvious layers may need to be transferred to a larger container for proper mixing. Refer to SOP QA035 for more information on sample homogenization. Using a tongue depressor, weigh out approximately 0.5g wet, homogeneous

sample into a digestion vessel. The sample should be weighed out using a top loader balance.

7.2 The sample identification must be accurately recorded on the digestion vessel and sample digestion log. In addition to the samples, a matrix spike, matrix spike duplicate, blank spike, duplicate and a method blank should be set up with each batch of 20 samples. For batches of 10 samples and less no matrix spike duplicate is necessary. Refer to Table 1 for the spiking solution levels to use for each matrix spike and blank spike. For the method and blank spike, 0.5 g of Teflon chips should be used.

7.3 Add 2.5 ml of concentrated nitric acid to all quality control and samples.

7.4 Place the labeled digestion vessels into the heating apparatus. Heat the samples at a gentle reflux 10-15 minutes. Allow the samples to cool.

7.5 Add an additional 2.5 ml of concentrated nitric acid to all quality control and samples. Heat the samples at a gentle reflux for an additional 30 minutes. Cool.

7.5.1 If brown fumes are generated, which indicates oxidation of sample by HNO3, then repeat step 7.5 until no brown fumes are present.

7.5.2 Allow sample to evaporate to 5 ml without boiling or heat at 90 - 95°C without boiling for 2 hours.

Important: Do not allow sample to dry.

7.6 Cool. Add 2 ml of water and 3 ml of 30% hydrogen peroxide to each sample and reflux until effervescence subsides.

7.7 Continue to add 30% hydrogen peroxide in 1-ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

Important: Do not add more than a total of 10 ml of 30% hydrogen peroxide.

7.8 Allow sample to evaporate to 5 ml or heat at 90 - 95°C for 2 hours.

Important: Do not allow sample to dry.

7.9 Cool. Add 5 ml of concentrated HCl and reflux for an additional 15 minutes.

7.10 Allow the sample to cool. Dilute to final volume of 50 mls using DI water, cap and shake vessel. If particulate matter is present, uncap the vessel and filter using Environmental Express Filtermate apparatus or equivalent. The method blank and blank spike for the filtered sample's prep group must be filtered as well. The sample is now ready for analysis by ICAP.

8.0 QC REQUIREMENTS:

For each digestion batch of 20 samples, a matrix spike, a matrix spike duplicate, a duplicate, a blank spike (LCS), and a method blank should be prepared. Re-digestion is suggested for QC that does not meet the Accutest QC limits. The appropriate lab supervisor or lab manager will notify the analyst of samples that need re-digestion.

9.0 GLASSWARE CLEANING:

All glassware should be washed with soap and tap water and then soaked in a 5% nitric acid bath. It should then be rinsed at least 3 times with de-ionized water.

10.0 DOCUMENTATION REQUIREMENTS:

All digestion information should be completed in the Metals Digestion Log. The information required includes, the sample identification (including bottle number), the initial sample weight, the final sample volume, the acids (including the lot number and manufacturer), the spikes used, the temperature, the thermometer ID, the digestion vessel lot number, the filter lot number, and the digestion date. The analyst should write additional information such as unusual sample characteristics in the comment section.

11.0 SAFETY:

The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the uses of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact supervisor.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 12.2.

12.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples.

TABLE 1: ICP METALS SPIKING LEVELS

ELEMENT	INITIAL CONC (ppm)	VOLUME USED (ml)	FINAL CONC (mg/l)	FINAL VOL. (ml)
Ba	200	0.50	2.0	50
Be	5	0.50	.05	50
Cd	5	0.50	.05	50
Cr	20	0.50	.20	50
Cu	25	0.50	.25	50
Co	50	0.50	0.50	50
Mn	50	0.50	0.50	50
V	50	0.50	0.50	50
Zn	50	0.50	0.50	50
As	200	0.50	2.0	50
Se	200	0.50	2.0	50
Pb	50	0.50	0.50	50
Tl	200	0.50	2.0	50
Sb	50	0.50	0.50	50
Mo	100	0.25	0.50	50
Sn	100	0.25	0.50	50
Al	200/5000	0.5/0.25	27	50
Fe	200/5000	0.5/0.25	26	50
Mg	5000	0.25	25	50
Ca	5000	0.25	25	50
K	5000	0.25	25	50
Na	5000	0.25	25	50
Ag	5	0.50	0.05	50
Ni	50	0.50	0.50	50

DIGESTION OF WATER SAMPLES FOR METALS ANALYSIS BY ICP

Prepared by: ____Dave Metzgar_____ Date: __4-4-06__

Reviewed by: _____Norm Farmer_____ Date: __4-4-06__

Annual Review

Reviewed by:_____ Date: _____

Reviewed by:_____ Date: _____

Reviewed by:_____ Date: _____

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Effective 7 days after "*" date

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TEST NAME: DIGESTION OF WATER SAMPLES FOR METALS ANALYSIS BY ICP

METHOD REFERENCE: SW846 3010A, EPA 200.7, SM 3030C

DEPARTMENT: METALS

REPORTING LIMIT: NOT APPLICABLE

SUMMARY OF CHANGES: Added Document Control cover page, MDL sec, Definitions, expanded sec 8, 9, 11, 13; Expanded section 2.0, updated 200.7 criteria.

1.0 SCOPE AND APPLICATION, SUMMARY

This method is applicable for the digestion of aqueous samples, TCLP extracts and wastes that contain small amounts of suspended solids. After digestion, the samples can be analyzed by ICP. This digestion method is based upon SW846 method 3010A and the EPA water digestion method.

Reduced volume version of method 200.7, March 1983 is used for the 200.7 digestion procedure. This approach that use the same reagents and molar ratios is acceptable by the regulatory agents provided it meets the quality control and performance requirements stated in the method.

Acid Extractable Metals by method SM3030C 18th Edition 1992 modified volumes.

2.0 PRESERVATION

All samples should be preserved with nitric acid to a pH of <2 at the time of collection. All sample pH are checked in sample receiving. For method 200.7 if samples are received with a pH >2 they must be preserved to pH <2 and held for 16 hours prior to metals digestion. TCLP extracts are preserved to a pH < 2 upon receipt from Accutest Southeast Extractions Department. Please refer to sample receiving SOP for further instructions.

3.0 HOLDING TIME

All samples should be digested and analyzed within 6 months of the time collection.

Samples being digested by method 3030C must be prepared within 72 hours of collection.

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4.0 REPORTING and METHOD DETECTION LIMITS

See analytical SOP

5.0 INTERFERENCE

Organic substances in a matrix may cause interference if the sample is not digested rigorously enough. In addition, high levels of acids in the final digestate may cause interference in the analysis. This interference can be avoided by choosing the appropriate digestion method and by bringing the sample to an appropriate final volume. For a discussion of other interference, refer to specific analytical methods.

6.0 APPARATUS

The apparatus needed for this digestion procedure are listed below.

6.1 Automatic pipette bottles

6.2 Whatman #41 filter paper or equivalent

6.3 Watch glass

6.4 Thermometer- capable of measuring a temperature of at least 125^0C

6.5 Environmental Express Hot Block or equivalent capable of maintaining a temperature of 90-95°C.

6.6 Environmental Express digestion cups or equivalent, 50ml capacity.

6.7 Environmental Express Filtermate

7.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. De-ionized water should be used whenever water is required.

7.1 Hydrochloric acid. Fisher Trace grade or equivalent

7.2 Nitric acid. Fisher Trace grade or equivalent

7.3 Metals spiking solutions: CPI Multielement spiking solution or equivalent made with 5% HNO3 and a trace of HF Inorganic Ventures 5000 mg/l Mineral solution and 100 mg/l Mo and Sn solution.

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8.0 PROCEDURE

8.1 Measure out 50 ml of each sample into a labeled digestion vessel. The sample may be measured by using a graduated cylinder or by using the calibrated digestion tube. Make sure that the sample identifications are accurately recorded on the digestion vessels and in the sample digestion log. In addition to the samples, a matrix spike (MS), matrix spike duplicate (MSD), duplicate, blank spike and a method blank should be set up with each batch of 20 samples. For the method and blank spike, 50 ml of de-ionized water should be used. Refer to Table 1 for the spiking solution levels to use for each MS, MSD and blank spike.

8.1.1 When preparing TCLP samples, prepare an additional leachate blank and leachate blank spike from the extraction fluid used for the samples. See section 8.8 and 8.9 of current METSOP 100 for acceptance criteria.

8.1.2 When preparing filtered samples for dissolved metals, an additional method blank must be prepared. The method blank must be filtered through the same filter media as the samples. See section 8.8 of current METSOP 100 for acceptance criteria.

8.2 Add 1.5 ml of concentrated nitric acid to all quality control and samples

8.3 Place the labeled digestion vessels into the heating apparatus. Heat the block (90 to 95°C) until the samples are at a gentle reflux. Reduce the volume of each sample to approximately 5 ml. This should take approximately 1-2 hours. Cool.

8.4 Add an additional 1.5-ml of concentrated nitric acid to all quality control and samples. Heat the samples at gentle reflux until the volume is again at approximately 5.0 ml. Cool.

8.4.1 If a client requires method 200.7 for the digestion, add 2.5ml of concentrated nitric acid instead of 1.5 ml in step 8.4. Continue heating the samples at a gentle reflux until the sample is completely digested. Signs of a complete digestion are if the digestate is light in color and/or if the appearance of the sample does not change with continued refluxing. More acid may be added as necessary to complete the digestion.

8.5 Add 2.5 ml of concentrated HCL to each sample and reflux for an additional 15 minutes. Cool. Rinse digestion vessel walls with de-ionized water and then filter (if necessary) the sample to remove insoluble material.

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8.5.1 If a client requires method 200.7 for the digestion, add 7.5 ml of DI water along with the HCL.

8.6 Bring the samples to a final volume of 50.0 ml with de-ionized water. If the sample contains particulate, it should be filtered, along with the method blank and blank spike, through Whatman #41 filter paper or Filtermate apparatus before analysis. Filtering of samples should be documented in the prep logbook. The sample is now ready for analysis by ICP.

8.7 Standard Method 3030C

8.7.1 Thoroughly mix sample. Transfer 50 ml to a labeled digestion vessel, and add 2.5 ml of 1 + 1 HCL. Heat 15 minutes on 90-95°C hotblock. Filter through a membrane filter and adjust filtrate volume to 50 ml with DI water. Sample is now ready for analysis. Please refer to Standard Methods 18th edition section 3030C for further instruction.

9.0 QC REQUIREMENTS

For each digestion batch of 20 samples, a matrix spike, a matrix spike duplicate, a duplicate, a blank spike (LCS), and a method blank should be prepared. Re-digestion is suggested for QC that does not meet the Accutest QC limits. The appropriate lab supervisor or lab manager will notify the analyst of samples that need re-digestion.

10.0 GLASSWARE CLEANING

All glassware should be washed with soap and tap water, rinsed with 5% nitric acid solution, and then rinsed at least 3 times with de-ionized water.

11.0 DOCUMENTATION REQUIREMENTS

All digestion information should be completed on the form attached to this SOP. The information required includes the sample identification (including the sample bottle number), the initial sample volume, and the final sample volume, the acids used (including lot number and manufacturer), the spikes used, the digestion vessel lot number, the temperature and thermometer ID, and the date of digestion. The analyst should write additional information such as unusual sample characteristics and samples that need to be filtered in the comment section. Refer to the standards SOP for additional information on documentation requirements to follow when making spiking solutions.

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12.0 SAFETY

The analyst should follow safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acid contacts any part of the body, flush with water and contact the supervisor.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

13.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 13.2.

13.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples.

14.0 DEFINITIONS

14.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours which ever comes first.

14.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

14.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run.

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14.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

14.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

14.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

14.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

14.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and bias of a method in a given sample matrix.

14.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

14.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

14.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

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Table 1: ICP Metals Spiking Levels

ELEMENT	INITIAL CONC (ppm)	VOLUME USED (ml)	FINAL CONC (mg/l)	FINAL VOL. (ml)
Ba	200	0.50	2.0	50
Be	5	0.50	.05	50
Cd	5	0.50	.05	50
Cr	20	0.50	.20	50
Cu	25	0.50	.25	50
Co	50	0.50	0.50	50
Mn	50	0.50	0.50	50
V	50	0.50	0.50	50
Zn	50	0.50	0.50	50
As	200	0.50	2.0	50
Se	200	0.50	2.0	50
Pb	50	0.50	0.50	50
Tl	200	0.50	2.0	50
Sb	50	0.50	0.50	50
Mo	100	0.25	0.50	50
Sn	100	0.25	0.50	50
Al	200/5000	0.5/0.25	27	50
Fe	200/5000	0.5/0.25	26	50
Mg	5000	0.25	25	50
Ca	5000	0.25	25	50
K	5000	0.25	25	50
Na	5000	0.25	25	50
Ag	5	0.50	0.05	50
Ni	50	0.50	0.50	50

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COLD VAPOR ANALYSIS OF MERCURY FOR WATER SAMPLES

Prepared by: ____Dave Metzgar_____ Date: __4-4-06__

Reviewed by: _____Norm Farmer_____ Date: __4-4-06__

Annual Review

Reviewed by:_____ Date: _____

Reviewed by:_____ Date: _____

Reviewed by:_____ Date: _____

Document Control

Issued to: _____QA Department _____ Date: _____

Issued to: _____Inorganics Department_____ Date: _____*

Issued to: _____ Date: _____

Issued to: _____ Date: _____

Issued to: _____ Date: _____

Effective 7 days after "*" date

TEST NAME: COLD VAPOR ANALYSIS OF MERCURY FOR WATER SAMPLES

METHOD REFERENCE: EPA 245.1 Rev.3, 1994, SW-846 7470A

INSTRUMENT SERIAL #: 2022

DEPARTMENT: METALS

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 The method outlined in this SOP is based on EPA method 245.1 and SW846 7470A for waters. The types of samples that can be analyzed include drinking, surface and saline waters, as well as domestic and industrial wastes.

1.2 The mercury is reduced to the elemental state and aerated from the solution in a closed system. The mercury vapor passes through a cell in the light path of an atomic spectrophotometer, where the absorbance is measured as a function of mercury concentration.

2.0 PRESERVATION

All liquid samples should be preserved by acidification with nitric acid to a pH of 2 or lower.

3.0 HOLDING TIME

All samples should be analyzed within 28 days of the date of collection.

4.0 REPORTING and METHOD DETECTION LIMITS

4.1 Reporting Limit. The reporting limit for this method has been established at 0.001 mg/l.

4.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria. Experimental MDLs must be determined annually for this method.

4.3 MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit annually. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is

calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level.

4.4 An MDL check standard will be analyzed at the time of the annual MDL study. The concentration of the MDL check standard should be 2x-3x the statistical MDL. This is a qualitative check; therefore, the analyte needs to be detected only. If the analyte is not detected, the concentration of the MDL check standard must be increased until the analyte is detected. This then becomes the current MDL.

4.5 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

4.6 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

5.0 INTERFERENCES

Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations of sulfide as sodium sulfide as high as 20 mg/l do not interfere with mercury recoveries when following this method. High copper concentrations (> 10 mg/l) may also interfere with mercury recoveries. Samples that are high in chloride such as seawater, brine and industrial effluent may require as much as 12.5 ml of additional permanganate.

Note: When chloride concentrations are high, hydroxylamine sulfate and stannous sulfate should be used in place of corresponding chlorides.

Finally, certain volatile organic materials will also absorb at this wavelength and can interfere. It can be determined if this type of interference is present by doing a preliminary run without reagents.

6.0 APPARATUS

6.1 A Leeman HYDRA AA automated analyzer is used for all analysis. Refer to the instrument manual for further details on this instrumentation.

6.2 Environmental Express Hot Block or equivalent.

6.3 Environmental Express digestion vessels or equivalent, 50ml capacity.

6.4 Class A volumetric pipettes and flasks

6.5 Thermometer

7.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. De-ionized water should be used whenever water is required.

7.1 Sulfuric acid, concentrated

7.2 Nitric acid concentrated. This acid must have low mercury content.

7.3 Stannous chloride. To 400 ml of DI water, add 14 ml of conc. sulfuric acid. Dilute to 500 ml and add 50 g of stannous chloride and dissolve. This compound does not dissolve well and should be stirred continuously when in use. Stannous sulfate may be used in place of stannous chloride. Stannous chloride is prepared daily. Lot number is recorded in Hg logbook.

7.4 Sodium chloride-hydroxylamine hydrochloride. Add 120 g of sodium chloride and 120 g of hydroxylamine hydrochloride to 1 liter of water. Mix well, hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.5 Potassium permanganate. Add 50 g of potassium permanganate to 1 liter of water and mix well.

Caution: Potassium permanganate is a strong oxidizing agent. Handle with care.

7.6 Potassium persulfate. Add 50.0 g of potassium persulfate to 1 liter of water and mix well.

Caution: Potassium persulfate is a strong oxidizing agent. Handle with care.

7.7 2 percent HCl Rinse Solution. 40 mls concentrated HCl, diluted to 2 liters with DI water. 2 percent HCL solution is prepared daily. Lot number is recorded in Hg logbook.

7.8 Daily working standards. Mercury standard solutions are made from a purchased stock solution of 1000 ppm mercury.

7.8.1 10 ppm Hg solution intermediate 1. (Calibration curve, CCV's, Blank Spikes, MS, MSD's, CRI) Using a 10-ml volumetric pipette, add 10 ml of 1000-ppm stock (to be purchased from a vendor such as Fisher) to a 1.00-liter volumetric flask containing approximately 750 ml of water and 10 ml of concentrated nitric

acid. Dilute to volume with water and mix well. This 10ppm standard must be prepared every six months.

7.8.2 100 ppb Hg solution intermediate 2. (Calibration curve, CCV's, Blank Spikes, MS, MSD's, CRI) Using a 10-ml volumetric pipette, add 10 ml of 10 ppm Hg solution (intermediate 1) to a 1.00-liter volumetric flask containing approximately 750 ml of water and 10 ml of concentrated nitric acid. Dilute to volume with de-ionized water and mix well. This 100ppb standard must be prepared every month.

7.8.3 ICV working solutions are prepared at the same concentrations as the calibration standards listed above, except they must be from a second source.

7.8.4 Daily working standards are prepared and digested daily using 100ppb standard solutions.

Note: All standards and prepared reagents must be prepared every 6 months or before stock standard expiration date, whichever comes first, except as noted elsewhere in this SOP.

8.0 WATER DIGESTION AND ANALYSIS PROCEDURE

Below is a step-by-step procedure for the digestion and analysis of water samples for mercury.

8.1 Make up the standard curve as shown below. Clearly label each digestion vessel with the standard's ID. The standard ID's should be recorded on the data summary sheet.

ml of 100 ppb Hg solution	ml of DI water	Total µg of Hg
0.0	50	0.0
0.10	50	0.02
0.50	50	0.10
1.50	50	0.30
2.50	50	0.50
3.00	50	0.60

Dilute to the 50ml mark on the digestion vessel.

8.2 Make up the quality control samples as shown below. Make sure to clearly label each digestion vessel.

Sample ID	ml of 100ppb Hg solution	ml of DI water	Total µg of Hg
Spike Blank	1.5	50	0.30
CCV	1.5	50	0.30
Low Check (CRI)	0.10	50	0.020
Method Blank	0.0	50	0.0
ICV	1.5	50	0.30

Sample ID	ml of 100 ppb Hg solution	ml of sample	Total µg of Hg
Matrix Spike	1.5	50	0.30 + sample
Matrix Spike Dup	1.5	50	0.30 + sample
Duplicate		50	sample

8.3 If no information is available about the level of mercury in the samples to be analyzed set up a 50-ml sample size. If information is available, select a sample size that will result in an analysis value near the mid-range of the curve. For TCLP samples, use a 5-ml initial volume. Record the volume used on the sample analysis data sheet.

8.4 To all samples, QC and standards add the following reagents:

- 1.25 ml of concentrated nitric acid
- 2.5 ml of concentrated sulfuric acid
- 5 ml of permanganate solution
- 2 ml of persulfate solution

Swirl the samples well after each addition of reagent. More potassium permanganate solution may be required for some samples. Enough should be added so that the purple color persists for at least 15 minutes.

8.5 Pre-heat the Hot Block to 95 ± 1°C. Place the sample vessels in the Hot Block. Heat the samples for 2 hours. Then remove and cool to room temperature.

8.6 While the samples are digesting, begin setting up the Leeman HYDRA AA mercury analyzer following the steps outlined below. Further details are available in the instrument manual.

8.6.1 Turn the Argon gas on. Check that the vent line is connected to the exhaust hood.

8.6.2 Inspect all pump tubing and replace if necessary. Put the tubing on the cassettes and attach to the pump head, making sure the cassette adjusters are completely tightened. Place the fresh

stannous chloride solution in the bottle. Fill the rinse bottle with 2 percent HCL solution.

8.6.3 Double click on the WINHG icon on the desktop. This will open the WinHg runner. Click on the control tab and click "turn on gas" , "turn on pump", and "turn on lamp" options.

8.6.4 Go to file (pull down menu) and choose new dataset. Save dataset as Hg-day-month-matrix-run no. (i.e., Hg0405S1).

8.6.5 Click on the **DB ↓ button.** This will open the WinHg Database window. Go to file and choose Hg7470 PROTOCOL. This will be the Protocol (method) used for water and soil analysis. Make sure "Dataset" match on WinHg Runner screen as well as the Database screen.
.

8.6.6 Click on the "STANDARD" tab in WinHgRunner window and click on **S1** through **S6.** Click on the "Rep1" button. Click on the "STAND AUTO" button and calibration will begin.

8.6.6 Calibration is performed by analyzing a series of 5 standards and a blank. While calibration is being performed, type the analysis sequence by clicking on the Rack Editor button. Enter the samples and the appropriate QC. ICV, ICB, CRI, CCV, and CCB, 10 samples, CCV, and CCB, etc. Save the sequence as Hg-day-month-matrix-run number (Hg0405S1). After calibration is performed and accepted **(Correlation Coeff. Of ≥0.995 must be achieved, if not, analysis must be terminated, problem corrected, and instrument re-calibrated.)**, click on the Sample Tab in WinHgRunner and click on "START NEW BATCH" button. Type the auto-sampler sequence filename as above (ie, Hg0405S1).

8.6.7 In Autosampler Run rack name drop down window, choose the filename you are analyzing. Assign the location of the start and finish cups and click on "RUN AUTO".

9.0 QUALITY CONTROL

All QC calculations should be done as outlined in the method. QC limits are compiled by Accutest should be used to determine if a given analysis is valid.

9.1 Method Blank – An acceptable method blank or reagent blank must be analyzed with every batch of samples processed. The method blank must be less than one half the reporting limit. If the method blank is greater than one half the reporting limit, the samples associated with the contaminated blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-

digesting and reanalyzing the samples, or qualifying the results with a “B” or “V” qualifier.

9.2 High Standard Check – The high calibration standard must be analyzed for method 245.1. The results of the high standard check must agree within 5 percent of the true value for the analysis to be valid. If the high standard check fails criteria, the samples must be re-digested and re-analyzed.

9.3 Initial Calibration Verification – An initial calibration verification (ICV) sample must be analyzed after the initial calibration has been performed. This sample must be prepared at or near the midpoint of the initial calibration from a reference material independent from the initial calibration solution. The results of the ICV must agree within 10 percent of the true value for the analysis to be valid. For method 245.1 the ICV must agree within 5 percent of the true value for the analysis to be valid. If the ICV fails, a new ICV or initial calibration must be performed and all samples must be re-digested and reanalyzed with an acceptable ICV.

9.4 Continuing Calibration Verification – If more than 10 samples are to be analyzed in a single day, a Continuing Calibration Verification sample prepared at or near the mid point of the initial calibration must be analyzed after every 10th sample and at the end of the analytical run. The results of this analysis must be within 20 percent of the initial calibration for the CCV analyses to be considered valid. For method 245.1 the results of the CCV must be within 10 percent of the true value for the analysis to be valid. If the CCV fails, all samples analyzed after the first passing CCV must be re-digested and reanalyzed.

9.5 Continuing Calibration Blank/Initial Calibration Blank – Analyze the Initial calibration blank solution at the beginning of each run and the continuing calibration blank after every tenth sample and at the end of the sample run. If the ICB/CCB is not less than one half the reporting limit for each element the reporting limit for those samples must be raised to greater than 2 times the background concentration. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank or qualifying the results with a “B” or “V” qualifier.

9.6 Low Level Check Standard (CRI) – A standard prepared at the low calibration concentration should be prepared and analyzed with each analytical run, at the beginning and end of each analytical run. The CRI should agree within 20 percent of the true value to be acceptable. If the CRI does not meet the acceptance criteria, the samples must be re-digested and reanalyzed, or footnoted in LIMS.

9.7 Blank Spike – A blank spike (BSP) or Laboratory Control Spike (LCS) should be prepared using de-ionized water spiked at the midpoint of the calibration curve. The blank spike must be within 20 percent of the true value for the analysis to be considered valid. For method 245.1 the results of the BSP must be within 15 percent of the true value for the analysis to be valid. If the blank spike exceeds the acceptance criteria, the samples must be re-digested and reanalyzed. A blank spike is required for every 20 field samples or for each analysis batch.

Note: Samples with Hg non-detected may be reported with Blank Spike recovery biased high.

9.8 Duplicate – A duplicate of a field sample should be processed and analyzed with each set of 20 field samples. The duplicate results should agree within 20 percent of one another for the analyses to be considered valid. If the duplicate fails the acceptance criteria, the samples should be re-digested and reanalyzed.

9.9 Matrix Spike/Matrix Spike Duplicate – At least on Matrix Spike/Matrix Spike Duplicate pair must be prepared and analyzed with every 20 field samples. The MS/MSD must agree within 30 percent of the true value for method 245.1 and within 20 percent of the true value for method 7470A. (20 percent Relative Standard Deviation for the MSD). If the results of the MS/MSD are outside the acceptance criteria, the data should be flagged as possible matrix effect. Alternatively, the use of the Method of Standard Additions (MSA) may be used to calculate the concentration in matrix effected samples.

9.10 When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted and reanalyzed to bring them within the calibration curve.

9.11 When preparing TCLP/SPLP samples, prepare an additional leachate blank and leachate blank spike from the extraction fluid used to extract the samples. See section 9.1 and 9.7 for acceptance criteria.

9.12 When preparing dissolved metals, an additional method blank must be prepared. The method blank must be filtered through the same filter media as the samples and then digested as usual. See section 9.1 for acceptance criteria.

10.0 DOCUMENTATION REQUIREMENTS

Record the start time, end time and temperature for all digestions on the analysis data sheet. All sample information should be clearly entered on these sheets. In addition, any unusual characteristics of the samples or the digestion procedure should be noted in the comment section. All dilutions must be clearly documented.

11.0 SAFETY

The analyst should follow normal safety procedures as outline in the Accutest Laboratory Safety Manual. Particular care should be observed in handling the strong acids and oxidizing agents. Safety glasses and lab coats should be worn at all times in the lab. Gloves should be worn when handling samples.

12.0 CALCULATIONS

Below are the calculations, which should be used for water samples. The concentration of the sample in ug should be obtained from the calibration curve.

$$\textbf{Final concentration in mg/l} = \frac{\textbf{Concentration of sample in ug}}{\textbf{Sample volume in ml}}$$

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

13.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 13.2.

13.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples. Mercury waste must be analyzed prior to disposal. Result must be under TCLP MCL. If result is not under TCLP MCL waste must be considered hazardous and disposed of accordingly. If waste passes TCLP criteria in can then be neutralized and disposed of.

14.0 DEFINITIONS

14.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours which ever comes first.

14.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

14.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run.

14.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

14.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

14.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

14.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

14.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and bias of a method in a given sample matrix.

14.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

14.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

14.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

15.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors

ANALYSIS OF DIESEL RANGE ORGANICS BY GAS CHROMATOGRAPHY USING FLAME IONIZATION DETECTOR

Prepared by: Norm Farmer Date: 03/06/06

Reviewed by: Stephanie Weber Date: 03/08/06

Annual Review

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TEST NAME: ANALYSIS OF DIESEL RANGE ORGANICS BY GAS CHROMATOGRAPHY USING FLAME IONIZATION DETECTOR

METHOD REFERENCE: SW846 8015B

DEPT: GC

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the total petroleum hydrocarbon (TPH) concentration in water, solid, and waste matrices utilizing a gas chromatograph equipped with a flame ionization detector.

1.1.2 TPH for this method is defined as any peaks, including any unresolved hydrocarbon mixtures eluting between Decane (nC10) and Octacosane (nC28). Quantitation is based on a diesel fuel standard.

1.1.3 Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. Reporting limits may vary depending on matrix complications and volumes. Reporting limits for this method are in the range of 0.25 mg/l for aqueous samples and 8.0 mg/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.1.5 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with a “J” qualifier. Some program or project specifications may require that no values below the RL be reported.

1.2 Summary

1.2.1 This method is adapted from SW846 Method 8015B for Diesel Range Organics.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with Accutest SOP OP010 and OP011.

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1.2.4 The extracts are analyzed on a gas chromatograph equipped with a flame ionization detector.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples.

2.1.2 Aqueous samples may be preserved with hydrochloric or sulfuric acid to minimize biological degradation.

2.1.3 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be stored at 4°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Interferences from phthalate esters can be eliminated by using plastic-free solvent containers and solvent rinsed glassware.

3.3 Other organic compounds, including chlorinated hydrocarbons, phenols, and phthalate esters may be coextracted by this method. By definition, these compounds are included as TPH.

3.4 Animal and vegetable oils and fats may be removed by silica cleanup.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

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4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

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5.0 REAGENTS

5.1 Methylene chloride – pesticide grade or equivalent

5.2 Diesel Stock standard – Traceable to Certificate of Analysis

5.3 Retention Time Standard – Containing nC10 and nC28

5.4 Surrogate standard – O-terphenyl

6.0 APPARATUS

6.1 Gas Chromatograph – HP 5890 or Agilent Technologies 6890

Suitable gas chromatograph equipped with a split-splitless injection port and flame ionization detector.

Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Data System – Agilent Technologies MS Chemstation rev. BA 02.00 and DA 02.00

6.2.1 A computer system interfaced to the gas chromatograph that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.2.2 A data collection and processing system. System must be capable of forced baseline integrations and quantitations using multipoint calibrations.

6.2.3 Data is archived to magnetic tape for long term storage.

6.3 DB-5 Column or equivalent: 30m X 0.32mm X 0.25um

6.4 DB-17 Column or equivalent: 30m X 0.32mm X 0.25um

6.5 Suitable syringes and glassware for dilutions of standards and extracts.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards should be stored at 4 °C, or as recommended by the manufacturer.

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7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to one year or the vendor's expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with methylene chloride. The hold time for intermediate standards is six months or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicate analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for TPH are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the RL and the remaining standards define the working range of the detector.

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Gas Chromatograph Conditions

1-2ul autosampler injection

Carrier gas – UHP Hydrogen

Detector gas – UHP Air (~300 ml/min)

Detector gas – UHP Hydrogen (~30 ml/min)

Injection port temperature – 260 °C

Detector temperature – 320 °C

Oven program – 50 °C for 2.0 minutes
45 °C/min to 190 °C for 0.0 minutes
30 °C/min to 310 °C for 9.0 minutes

Pressure program – Constant Flow 4.0 ml/min

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Oven program – 60 °C for 2 minutes
45 °C/min to 120 °C for 0.0 minutes
30 °C/min to 270 °C for 0.0 minutes
25 °C/min to 310 °C for 6.1 minutes

Pressure program – 20.6 psi for 2 minutes
1 psi/min to 25 psi for 0 minutes
5 psi/min to 50 psi for 0 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Gas Chromatograph Conditions (GC Racer Equipped Instrument)

1-2ul autosampler injection

Carrier gas – UHP Helium

Detector gas – UHP Air (~300 ml/min)

Detector gas – UHP Hydrogen (~30 ml/min)

Injection port temperature – 275 to 300 °C

Detector temperature – 320 °C

Oven program – 50 °C for 2 minutes
65 °C/min to 310 °C for 6.9 minutes

Pressure program – 17.5 psi for 2 minutes
6 psi/min to 50 psi for 0 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.4 Sample Preparation

7.4.1 Water Samples

A 1000ml aliquot of sample is extracted with methylene chloride utilizing separatory funnel extraction. The extract is cleaned with silica gel and then concentrated to 1.0ml.

7.4.2 Solid Samples

A 30-gram aliquot of sample is extracted with methylene chloride utilizing pulse sonication. The extract is cleaned with silica gel and then concentrated to 1.0ml.

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7.4.3 Waste Samples

A one-gram aliquot of sample is diluted in methylene chloride to 10ml. The extract is then cleaned with silica gel.

7.5 Gas Chromatographic Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.5.1 Initial Calibration Procedures

Before samples can be run, the chromatographic system must be calibrated and retention time ranges and windows must be determined.

7.5.1.1 Retention Time Range

A nC10 to nC28 even numbered alkane standard is used to establish the retention time range. The diesel range is defined by the start of nC10 and the end of nC28. This range is used for all samples and standards. The range must be verified every 12 hours.

7.5.1.2 External Standard Calibration

Quantitation for this method is based on the response of diesel fuel. A minimum 5-point calibration curve is created for diesel fuel and o-terphenyl (surrogate).

The area of the diesel fuel standard is determined by integrating (baseline to baseline) from the time where nC10 starts to the time where nC28 ends. The MS Chemstation software will automatically correct for the area of the surrogate.

Calibration factors (CF) for diesel are determined at each concentration by dividing the total area of the diesel standard (corrected for the surrogate) by the concentration of the standard. Calibration factors for the surrogate are determined at each concentration by dividing the area of the peak by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the calibration factors is calculated for each analyte as follows:

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%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD ≤ 20%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively if the %RSD > 20% a calibration curve of response vs. amount can be plotted. If the correlation coefficient (r) is ≥0.995 (r^2 ≥0.990) then the curve can be used to quantitate target analytes in the samples.

7.5.1.3 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV should be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be ≤ 15%. If the ICV does not meet this criteria, a second standard should be prepared. If the ICV still does not meet criteria, analyze an ICV prepared from a third source. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.5.1.4 Solvent Blank

A solvent blank must be analyzed at the beginning of each 12hr shift to determine the normal baseline rise during a GC run. This amount must be less than the one half the RL for the initial solvent blank. Solvent blanks should also be analyzed after samples with high TPH concentrations to ensure that there is no carryover.

7.5.1.5 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Individual nC10, nC28, and surrogate peaks must fall within the appropriate retention time windows. However, the TPH range is defined as any peaks, including any unresolved hydrocarbon mixtures eluting between the start of nC10 and the end of nC28.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do

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not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. For this method, the retention time windows must be calculated for nC10, nC28, and o-terphenyl. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations.

The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. The analyst should monitor the retention times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential “hits”. The analyst will then review these “hits” and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

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7.5.2 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for TPH are prepared at various concentrations; at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally a CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| should be ≤ 15% for each analyte. If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated.

If the |%D| is greater than 15%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported without qualification. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed or the data must be qualified.

7.5.3 Sample Extract Analysis

7.5.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Retention Time Standard
Initial Calibration Standards (or Initial CCV)
Solvent Blank
QC Extracts
Sample Extracts
CCV Standards

7.5.3.2 One or two microliters (same amount as standards) of extract is injected into the GC by the autosampler. A splitless injection technique is used. The data system then records the resultant peak responses and retention times.

7.5.3.3 As defined by the method, any peaks (including any unresolved petroleum hump) falling within the retention time range are defined as TPH and must be quantitated. Confirmations are not required for this method.

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7.5.3.4 If the petroleum response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.5.3.5 Some petroleum products (gasoline, motor oil, etc) may not fall completely within the diesel range. If a significant amount of the product falls outside the diesel range, it should be noted on the report.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Surrogates

9.1.1 O-terphenyl is used as the surrogate standard to monitor the efficiency of the extraction and clean-up procedures.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for the results to be acceptable.

9.1.2 If the surrogate recoveries are not within the established control limits, the following are required.

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9.1.2.1 Check to be sure that there are no errors in calculations, integrations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly.

9.1.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, re-extract and reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary. If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis should be reported on the final report.

9.2 Method Blank

9.2.1 The method blank is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high level samples. The method blank must be free of any analytes of interest or interferences at ½ the required reporting level to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination should be investigated and documented. The samples results may be reported with the appropriate “B” qualifier. This must be approved by the department supervisor.

9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination should be

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investigated and documented. The samples should be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.3.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 If the recovery of an analyte in the BS is high and the associated sample is non-detect, then the sample results can be reported without qualification.

9.3.2.4 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.4 Matrix Spike and Matrix Spike Duplicate

9.4.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

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% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.4.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.4.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.4.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are an indication of the sample matrix effects.

9.4.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

10.0 CALCULATIONS

The area of the TPH in the samples is determined by integrating (baseline to baseline) from the time where nC10 starts to the time where nC28 ends. This integration must include any unresolved petroleum “hump”. The MS Chemstation software will automatically correct for the area of the surrogate.

The concentration of the analyte in the original sample is calculated as follows:

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TPH Water (mg/l) = ($CONC_{inst}$) X (V_F / V_I) X DF

TPH Soil (mg/kg) = [($CONC_{inst}$) X (V_F / W_I) X DF] / %solids

$CONC_{inst}$	=	Instrument concentration calculated from the initial calibration using mean RF, linear curve, or quadratic curve
DF	=	Dilution Factor
V_F	=	Volume of final extract (ml)
V_I	=	Volume of sample extracted (ml)
W_I	=	Weight of sample extracted (g)
%solids	=	Dry weight determination in decimal form

All soils are reported on a dry weight basis.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst should follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment should be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis and standards preparation are collected in waste storage bottles and are eventually transferred to the chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 References

SW846 Method 8000B Revision 2, December 1996

SW846 Method 8000C Revision 3, March 2003

SW846 Method 8015B Revision 2, December 1996

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Florida DEP Method FL-PRO, REV. 1, 11/95

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STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF DIESEL RANGE ORGANICS (DRO) FROM SOLID SAMPLES

Prepared by: Norm Farmer Date: 03/11/06

Reviewed by: Stephanie Weber Date: 03/11/06

Annual Review

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TEST NAME: **STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF DIESEL RANGE ORGANICS (DRO) FROM SOLID SAMPLES**

Method: SW846 3550B/8015B DRO

Dept: OP

1.0 Summary, Scope and Application

1.1 Summary

Solid samples are serially extracted by pulse sonication, concentrated by TurboVap apparatus, and stored in glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to solid samples, including soils and sediments, submitted for DRO analysis by GC/FID method SW-846 8015B.

2.0 Discussion and Comments

This procedure is adapted from SW-846 methods 3500B and 3550B. The method outlined in this SOP is designed for low concentration samples (concentration of the individual organic components is expected to be less than 20mg/kg).

The FID is a non-selective detector and will respond to most organic compounds. It is important to minimize extraneous contaminants by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass, stainless steel, wood, or Teflon.

Animal oils, vegetable oils, and biogenic terpenes can be removed by treating the extract with silica gel.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.0 Samples shall be collected in amber glass jars with Teflon lined caps. 250ml jars are recommended for solid samples.

3.1.1 The samples must be protected from light and refrigerated at 4°C from the time of collection until extraction. The extracts must be refrigerated at 4°C until analysis.

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3.2 Holding Time

3.2.0 Solid samples must be extracted within 14 days of collection. A sample is considered extracted after it has been sonicated three times and the solvent has been transferred to an Erlenmeyer flask.

3.2.1 Extracts must be analyzed within 40 days of extraction.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in

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environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Acetone – pesticide grade or equivalent

5.2 Methylene chloride – pesticide grade or equivalent

5.3 Anhydrous sodium sulfate – precleaned to remove phthalates

5.4 Silica gel – Davidson grade 950 or equivalent – 60 to 200 mesh

5.5 DRO Surrogate Solution – prepared in acetone or methanol at a concentration specified by the GC analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.6 DRO Spike Solution – prepared in acetone or methanol at a concentration specified by the GC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

6.0 Glassware and Apparatus

6.1 400ml thick walled beaker

6.2 Glass funnels (large enough to support the filters)

6.3 500ml Erlenmeyer flask

6.4 Spatula – stainless steel, wood, or Teflon

6.5 0.5ml or 1.0ml syringes

6.6 1.0ml volumetric flask

6.7 200ml graduated TurboVap tube

6.8 Disposable transfer pipettes

6.9 glass wool – precleaned

6.10 Fisher P8 filters – or equivalent

6.11 2.0ml glass screw cap vials – caps must have Teflon lined septa

6.12 Zymark TurboVap II or equivalent

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6.13 Ultrasonic disrupter – minimum power of 300 watts with pulse capability

6.14 Disrupter horns – ¾" solid titanium tip

6.15 Top loading balance – capable of weighing samples to +/- 0.1 grams

7.0 Sonic Disrupter

Follow the manufacturer's instructions for tuning and operating the sonic disrupter. Tuning should be verified at least weekly and should be documented in the "Instrument Repair and Maintenance" logbook. The sonic disrupter should be set in the pulse mode with a 50% duty cycle (energy on 50% of the time and off 50% of the time). Because the Fisher Scientific 550 Sonic Dismembrator and Branson 450 Digital Sonifier have maximum power outputs of 475 watts and 400 watts respectively, the power control levels can be set at 70% and 80% output instead of 100% output.

8.0 Procedure

8.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, and dates. The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the GC analyst with the extracts.

8.2 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. Discard the top layer of soil and thoroughly mix the sample with a wooden spatula. Samples with obvious layers may need to be transferred to a larger container for proper mixing. Refer to SOP QA035 for more information on sample homogenization.

8.3 Transfer approximately 30 grams of each sample to the appropriately labeled beakers. Use a clean spatula for each sample. Record the weight to the nearest 0.1gram on the prep sheet.

8.4 Add approximately 30 grams of sodium sulfate to each sample and mix until each sample has a free flowing sandy texture. Wetter soils will require more sodium sulfate.

8.5 Transfer approximately 30 grams of each of the QC samples to the appropriately labeled beakers. This includes the method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD). Use 30 grams of sodium sulfate and/or clean sand for the MB and BS. Use additional 30 gram aliquots of a sample for the MS and MSD. If there is insufficient sample amount, a lesser amount may be used. Record the sample ID, bottle number, and weight on the prep sheet.

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8.6 Using the dedicated surrogate syringe add 0.5ml of surrogate solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

8.7 Using the dedicated spike syringe add 0.5ml of spike solution to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

8.8 Immediately add 100ml of methylene chloride (50:50 methylene chloride and acetone may also be used) to each of the beakers. This will minimize the loss of the more volatile analytes.

8.9 Label the Erlenmeyer flasks and place a glass filter funnel containing a Fisher P8 filter on the top of each flask.

8.10 Place the disrupter horn in the beaker such that the tip of the horn is approximately ½ inch into the solvent but not touching the beaker or the sample.

8.11 Sonicate the sample for 3 minutes.

8.12 Remove the beaker and decant the solvent through the filter funnel into the flask.

8.13 Repeat steps 8.10 to 8.12 two more times with additional 100ml solvent aliquots. All three solvent aliquots will be combined in the K-D flask.

8.14 After the final extraction, transfer the entire sample to the funnel, rinse thoroughly, and allow the solvent to drain through the sample.

NOTE: It is important to clean the disrupter horn between samples. This is done by rinsing the horn with the extraction solvent and wiping it dry with a Kim-wipe or paper towel.

8.15 Label the TurboVap tubes and place them in the metal support rack. Because the total extract volume exceeds the capacity of the TurboVap tube, transfer approximately 200ml each extract to the appropriately labeled tube. The remaining extract will be transferred later.

8.16 Set the water bath temperature for the TurboVap to 40 ^{o}C. Place the tube in the TurboVap. **NOTE:** If the bath is too hot, the more volatile compounds may be lost during this step. Concentrate the extract to approximately 50ml.

8.17 Remove the TurboVap tube and transfer the remaining extract from the Erlenmeyer flask to the appropriate tube. Rinse each Erlenmeyer flask with methylene chloride and transfer it to the appropriate tube.

8.18 Return the tube to the TurboVap and concentrate the extract to 0.7ml.

8.19 Remove the TurboVap tube and place it in the metal support rack to allow it to cool.

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8.20 If the extract is cloudy or contains water droplets, run the extract through a micro column of glass wool and sodium sulfate.

8.21 Transfer the extract to a 1.0ml volumetric flask. Rinse the TurboVap tube with a few drops of methylene chloride and transfer it to the volumetric flask. Adjust the final volume to 1.0ml. **NOTE:** If the extract will not concentrate to 1.0ml, choose the next appropriate final volume. Be sure to record the final volume on the prep sheet.

8.22 Add 0.2 to 0.3 grams of silica gel to each extract. Use a transfer pipette to mix each extract.

8.23 Allow the silica gel to settle.

8.24 Transfer the extract to an appropriately labeled 2.0ml screw cap vial. Store the extracts in the "extract refrigerator" until they are needed for analysis.

9.0 Quality Assurance and Quality Control

9.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

9.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

10.0 Safety and Waste Disposal

10.1 Safety

10.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

10.1.2 **Hearing protection** must be worn while operating the sonic disrupters. The high frequency could cause permanent hearing loss.

10.1.3 Avoid touching the disrupter horns while they are active. Contact may cause burns or tissue damage.

10.1.4 Material Safety Data Sheets (MSDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS prior to using any new reagents or solvents.

10.1.5 Methylene chloride is an inhalation hazard and a suspected carcinogen. Fume hoods must be used to minimize exposure to vapors.

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10.2 Waste Disposal

10.2.1 Waste methylene chloride is placed in the “chlorinated waste” container.

10.2.2 Waste acetone is placed in the “non-chlorinated waste” container.

10.2.3 Extracted soil samples are placed in a waste container after the solvent has drained or evaporated.

10.2.4 Waste soil from the homogenizing process should be place in the “soil waste” container. **Note:** Waste soil from foreign soils must follow “foreign soil” disposal requirements.

11.0 References

11.1 SW-846 Method 3500B, Rev. 2, 12/96

11.2 SW-846 Method 3550B, Rev. 2, 12/96

11.3 SW-846 Method 8015B, Rev. 2, 12/96

11.4 Florida DEP Method FL-PRO, REV. 1, 11/95

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ANALYSIS OF GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY USING FLAME IONIZATION DETECTOR

Prepared by: Norm Farmer Date: 03/05/06

Reviewed by: Stephanie Weber Date: 03/06/06

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Effective 7 days after "*" date

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TEST NAME: ANALYSIS OF GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY USING FLAME IONIZATION DETECTOR

METHOD REFERENCE: SW846 8015B

DEPT: GC

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the total petroleum hydrocarbon (TPH) concentration in water, solid, and waste matrices utilizing a gas chromatograph equipped with a flame ionization detector. The method may be utilized to detect petroleum hydrocarbons in the Gasoline Range (GRO), which corresponds to the range of alkanes from C_6 to C_{10} and covers a boiling point range of approximately 60 ^oC - 170 ^oC.

1.1.2 Reporting limits (RL) are based on the sample amount and the lowest calibration standard. Reporting limits may vary depending on matrix complications and volumes. Reporting limits for this method are in the range of 0.1 mg/l for aqueous samples and 5.0 mg/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.3 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.1.4 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with a “J” qualifier. Some program or project specifications may require that no values below the RL be reported.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8015B for Gasoline Range Organics.

1.2.2 Samples are received, stored and analyzed within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with Accutest SOP OP020 and OP021.

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1.2.4 The samples are analyzed on a gas chromatograph equipped with a flame ionization detector (FID).

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

Aqueous Samples:

2.1.1 Samples shall be collected in standard 40mL vials with Teflon lined silicone septa. HCl is used to adjust the pH of the sample to <2.

2.1.2 The samples must be stored in capped vials, with minimum headspace, at 4°C in an area free of solvent fumes. The size of any bubble caused by degassing upon cooling should not exceed 5-6mm.

Solid Samples:

2.1.3 Special 40mL vials for purge-and-trap of solid samples, as well as the collection and preservation options are described in OP020. Samples collected in wide-mouth jars will be preserved in the lab and data footnoted accordingly.

2.1.4 Soil samples are preserved with methanol at a ratio of 1 gram of soil to 1ml of methanol. Soils prepared by Method 5035 are stored at –10°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be analyzed within 14 days of collection.

2.2.2 Solid and waste samples must be analyzed within 14 days of collection.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 High levels of heavier petroleum products may contain some volatile components within the range of retention times for gasoline. As defined by the method, the GRO results include these compounds.

3.3 Samples may become contaminated by diffusion of volatile organics through the container septum during shipment and storage. A trip blank may serve as a check on shipping and storage conditions.

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3.4 Contamination by carry-over can occur whenever high level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for carry-over.

3.5 Sparging tubes should be washed with detergent, rinsed with reagent water and dried at 105 C between use.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or samples loaded on an instrument within the same 12-hour shift, which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

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4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the method efficiency.

4.13 Trip Blank: A sample of analyte-free matrix taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organic samples.

5.0 REAGENTS

5.1 Reagent water - distilled or deionized - free of interferences.

5.2 Methanol - purge and trap grade or equivalent.

5.3 Gasoline stock standard - Traceable to Certificate of Analysis

5.4 Surrogate Solution - a,a,a-Trifluorotoluene and 4-Bromofluorobenzene

5.5 Spike Solution - Gasoline stock standard

5.6 GRO Defining Mix - a standard consisting of individual gasoline components, including 2-Methylpentane and 1,2,4-Trimethylbenzene. This is used to define the retention time window for GRO.

6.0 APPARATUS AND MATERIALS

6.1 Gas Chromatograph – HP 5890 or Agilent Technologies 6890

Suitable gas chromatograph equipped with a suitable injection port and flame ionization detector.

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6.2 Purge and Trap – OI Analytical 4560 or 4660 with OI Analytical 4552 or MPM16

6.2.1 The following autosampler models are used for purging, trapping and desorbing the sample onto GC column.

- O.I. Model 4560 sample concentrator with 4552 Water multisampler
- O.I. Model 4660 sample concentrator with 4552 Water multisampler
- O.I. Model 4560 sample concentrator with MPM16 Autosampler

6.2.2 The sample purge vial must be designed to accept 5 ml samples with a water column at least 3 cm deep.

6.2.3 The desorber should be capable of rapidly heating the trap to the manufacturer recommended desorb temperature.

6.3 Data System – Agilent Technologies MS Chemstation rev. BA02.00 and DA02.00

6.3.1 A computer system interfaced to the gas chromatograph that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.3.2 A data collection and processing system. System must be capable of forced baseline integrations and quantitations using multipoint calibrations.

6.3.3 Data is archived to magnetic tape for long term storage.

6.4 Trap – OI "7" or equivalent: Tenax
The trap should be conditioned according to the manufacturer's recommendations.

6.5 Column – RTX-502.2 or equivalent: 105m X 0.53mm X 3um
Column – RTX-VRX or equivalent: 75m X 0.45mm X 2.55um
The column chosen must resolve 2-methylpentane from the methanol solvent front.

6.6 Suitable syringes and glassware for dilutions of standards and samples.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Volatile Standards Logbook. All standards shall be traceable to their original source. The standards should be stored at temperatures between –10 °C and –20 °C, or as recommended by the manufacturer.

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7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to six months or the vendor's expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with methanol. The hold time for intermediate standards is one month or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicate analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the gasoline range organics are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration near the RL and the remaining standards define the working range of the detector. Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Gas Chromatograph Conditions

5mL sample purge

Carrier gas – UHP Helium (10-12 ml/min)

Detector gas – UHP Air (160 ml/min)

Detector gas – UHP Hydrogen (30 ml/min)

Make-up gas – UHP Helium (15-20 ml/min)

Injection port temperature – 220 °C

Detector temperature – 240 °C

5890
Oven program – 50 ^{o}C for 6 minutes
20 ^{o}C/min to 180 ^{o}C for 3.5 minutes
30 ^{o}C/min to 240 ^{o}C for 5.0 minutes

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6890
Oven program – 45 ^{o}C for 4 minutes
10 ^{o}C/min to 180 ^{o}C for 0 minutes
30 ^{o}C/min to 250 ^{o}C for 4 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Purge and Trap Device conditions

Purge Gas:	Helium at 35 ml/min
Sample Temp:	Ambient
Trap Temp:	<25°C
Purge Time:	12 min
Desorb:	2 min. at 180°C
Bake:	8 min. at 200-220°C

7.4 Sample Preparation

7.4.1 Water Samples

A 5ml aliquot of sample is loaded onto the purge-and-trap device and purged for 12minutes. Detailed procedures are described in SOP OP021.

7.4.2 Solid Samples

A methanol aliquot from the sample is loaded onto the purge-and-trap device and purged for 12 minutes. Detailed procedures are described in SOP OP021.

7.5 Gas Chromatographic Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.5.1 Initial Calibration Procedures

Before samples can be run, the chromatographic system must be calibrated, and retention time windows must be determined.

7.5.1.1 Retention Time Range

A gasoline component mix containing 2-Methylpentane and 1,2,4-Trimethylbenzene is used to establish the retention time range. The gasoline range is defined by the start of 2-Methylpentane and the end of 1,2,4-Trimethylbenzene. This

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range is used for all samples and standards. The range must be verified every 12 hours.

7.5.1.2 External Standard Calibration

Quantitation for this method is based on the response of gasoline. A minimum 5-point calibration curve is created for gasoline and the surrogates.

The area of the gasoline standard is determined by integrating (baseline to baseline) from the time where 2-Methylpentane starts to the time where 1,2,4-Trimethylbenzene ends. The MS Chemstation software will automatically correct for the area of the surrogates.

Calibration factors (CF) for gasoline are determined at each concentration by dividing the total area of the gasoline standard (corrected for the surrogate) by the concentration of the standard. Calibration factors for the surrogates are determined at each concentration by dividing the area of the peak by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the calibration factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively if the %RSD > 20% a calibration curve of response vs. amount can be plotted. If the correlation coefficient (r) is $\geq$0.995 ($r^2 \geq$0.990) then the curve can be used to quantitate target analytes in the samples.

7.5.1.3 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV should be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be $\leq$ 15%. If the ICV does not meet this criteria, a second standard should be prepared. If the ICV still does not meet criteria, analyze an ICV prepared from a third source. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet

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criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.5.1.4 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Individual 2-Methylpentane, 1,2,4-Trimethylbenzene, and surrogate peaks must fall within the appropriate retention time windows. However, the TPH range is defined as any peaks, including any unresolved hydrocarbon mixtures eluting between the start of 2-Methylpentane and the end of 1,2,4-Trimethylbenzene.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. For this method, the retention time windows must be calculated for 2-Methylpentane, 1,2,4-Trimethylbenzene, and surrogates. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations.

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The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. The analyst should monitor the retention times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential "hits". The analyst will then review these "hits" and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

7.5.2 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for TPH are prepared at various concentrations; at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally a CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| should be ≤ 15% for each analyte. If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated.

If the |%D| is greater than 15%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported without qualification. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed or the data must be qualified.

7.5.3 Sample Analysis

7.5.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

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GRO Window
Initial Calibration Standards (or Initial CCV)
Blank Spike (BS)
Method blank
Samples
Matrix spikes
CCV Standards

7.5.3.2 Each sequence shall begin with establishment of the RT range. Analyze 5ml of the window defining mix. The TPH range is defined as any peaks, including any unresolved hydrocarbon mixtures eluting between the start of 2-Methylpentane and the end of 1,2,4-Trimethylbenzene.

7.5.3.3 Ten microliters of surrogate solution is added to every 5mL of sample in the luer-lok syringe. If the Archon sampler is used, it is programmed to add 1uL of surrogate mix to each sample.

7.5.3.4 The automated sampler and concentrator are programmed to purge the sample for 12 minutes and then desorb the sample onto the GC column. After desorbing, the concentrator automatically bakes the trap for 8 minutes to remove any remaining volatiles.

7.5.3.5 If the analyte response exceeds the linear range of the system, the sample must be diluted and reanalyzed if sample is available. Samples should be diluted so that the response falls into the upper half of the calibration curve.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 Quality Assurance / Quality Control

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC

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set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Surrogates

9.1.1 Bromofluorobenzene and a,a,a-Trifluorotoluene are used as the surrogate standards to monitor the purge efficiency.

A known amount of surrogate standard is added to each sample including the QC set prior to analysis. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for the results to be acceptable.

9.1.2 If the surrogate recoveries are not within the established control limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, integrations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly.

9.1.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then reanalysis may not be necessary. If there is insufficient sample for reanalysis, footnote this on the report.

9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis should be reported on the final report.

9.2 Method Blank

9.2.2 The method blank is de-ionized water to which the surrogate standard has been added. The method blank is then purged along with the other samples to determine any contamination from the system or ambient sources. The method blank must be free of any analytes of interest or interferences at ½ the required reporting level to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the

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contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination should be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination should be investigated and documented. The samples should be for confirmation. If there is insufficient sample to reanalyze, or if the sample is reanalyzed beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is de-ionized water to which the surrogate standard and spike standard have been added. The blank spike is then purged along with the other samples to monitor the accuracy of the analytical procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable.

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.3.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported,

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additional corrective action is not necessary and the sample results can be reported without qualification.

9.3.2.4 If the recovery of an analyte in the BS is high and the associated sample is non-detect, then the sample results can be reported without qualification.

9.3.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples or qualifying the results as estimated.

9.3.2.6 If there is insufficient sample to reanalyze, or if the sample is reanalyzed beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4 Matrix Spike and Matrix Spike Duplicate

9.4.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then analyzed along with the other samples to monitor the precision and accuracy of the analytical procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.4.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.4.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.4.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for reanalysis but are an indication of the sample matrix effects.

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9.4.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If the RPD falls outside of the established control limits, the department supervisor shall review the data and determine if any corrective action is necessary. RPD failures are generally not grounds for batch reanalysis.

10.0 CALCULATIONS

10.1 The area of the TPH in the samples is determined by integrating (baseline to baseline) from the time where 2-Methylpentane starts to the time where 1,2,4-Trimethylbenzene ends. This integration must include any unresolved petroleum "hump". The MS Chemstation software will automatically correct for the area of the surrogate.

10.2 The concentration of each target compound in the original sample is calculated as follows:

Water (ug/l) = ($CONC_{inst}$) X DF

Soil (ug/kg) = [($CONC_{inst}$) X (V_F / V_A) X (5 / W_I) X DF] / %solids (*high level soils)*

$CONC_{inst}$	=	Instrument concentration calculated from the initial calibration using mean RF, linear curve, or quadratic curve
DF	=	Dilution Factor
V_F	=	Volume of methanol extract (ul)
V_A	=	Volume of methanol aliquot (ul)
W_I	=	Weight of sample (g)
%solids	=	Dry weight determination in decimal form

For high level soils, if %solids is < 90% then V_F is calculated as follows:

V_F = {ml of solvent + [(%moisture X W_I) / 100]} X 1000 ul/ml

All soils are reported on a dry weight basis.

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11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst should follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment should be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis, methanol extraction, and standards preparation are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Old stock standards are disposed of in the waste vial drum.

Samples are archived and stored for 30 days after analysis. Spent aqueous samples and those deemed non-hazardous are rinsed down the drain with large amounts of water.

Aqueous samples that are deemed hazardous are disposed of in the aqueous waste drum.

Soil samples are disposed of in the soil waste drum.
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12.0 REFERENCES

SW846 Method 8000B Revision 2, December 1996

SW846 Method 8000C Revision 3, March 2003

SW846 Method 8015B Revision 2, December 1996

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STANDARD OPERATING PROCEDURE FOR THE INTRODUCTION OF VOLATILE ORGANICS ANALYTES USING PURGE-AND-TRAP

Prepared by: Rick Watkins Date: 03/06/06

Reviewed by: Norm Farmer Date: 03/06/06

Annual Review

Reviewed by: _____ Date: _____

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TEST NAME: STANDARD OPERATING PROCEDURE FOR THE INTRODUCTION OF VOLATILE ORGANICS ANALYTES USING PURGE-AND-TRAP

Method: SW846 5030B

Dept: OP

1.0 Summary, Scope and Application

1.1 Summary

Aqueous samples: Helium is bubbled through a portion of the aqueous sample at ambient temperature; the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is rapidly heated and backflushed with helium to desorb the components onto a gas chromatographic column.

High-level extracts from Method 5035A: An aliquot of the extract is combined with organic free reagent water in the purging chamber. It is then analyzed following the procedure for aqueous samples.

1.2 Scope and Application

This method describes a purge-and-trap procedure for the analysis of volatile organic compounds (VOCs) in aqueous samples, water miscible liquids, and high-level extracts (prepared in Method 5035A).

2.0 Discussion and Comments

This procedure is adapted from SW-846 method 5030B. Cross contamination can occur whenever high concentration samples are analyzed. Any sample that contains analytes an order of magnitude higher than the highest level standard can be described as a possible source for cross contamination.

Two types of system contamination are possible in this method. A high-level sample followed by a low-level sample may show “carryover” of compounds with higher boiling points or water solubility. This type of contamination usually only persists for the analysis immediately following the high-level sample.

Extremely high-level samples may cause more persistent system contamination. These samples will cause “carryover” in one or more subsequent analysis. They may also result in “positional carryover”. The high-level compounds may linger in the autosampler position where the high-level sample was purged.

The analyst must use any available historical data or client supplied information to avoid contaminating the system. Volatile analysis systems employing the “Archon” are much

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less susceptible to carryover but are not immune altogether. Any doubt or suspicion is cause for reanalysis.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.1 Samples should be preserved to a pH < 2. The pH should be checked and recorded immediately after the sample analysis.

3.1.2 If the sample is not preserved to a pH < 2, it must be noted on the report.

3.1.3 Samples should be checked for the presence of residual chlorine. If residual chlorine present, it must be noted on the report.

3.2 Holding Times

3.2.1 All samples must be analyzed within fourteen days of collection.

3.2.2 Some agencies and project plans may require unpreserved samples to be analyzed within seven days of collection.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or samples loaded on an instrument within the same 12-hour shift, which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

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4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

4.10 Trip Blank: A sample of analyte-free matrix taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organic samples.

5.0 Reagents

5.1 Reagent water – distilled or deionized water free of interferences

5.2 Methanol – purge-and-trap grade or equivalent

5.3 Surrogate Solution – prepared in methanol at a concentration specified by the analyst.

5.4 Standard Solution – prepared in methanol or water at a concentration specified by the analyst.

5.5 Internal Standard Solution – prepared in methanol at a concentration specified by the analyst.

5.6 Spike Solution – prepared in methanol at a concentration specified by the analyst.

6.0 Glassware and Apparatus

6.1 Microsyringes – 10uL, 25uL, 50uL, 100uL, 250uL, 500uL, and 1000uL. These syringes are gastight with Teflon plunger tips used for spike and surrogate solutions. These may also be employed for sample dilutions.

6.2 Syringes – 5mL, and 10mL syringes with Luer-Lok tip.

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6.3 Volumetric flasks – 5mL, 10mL, 50mL, and 100mL class A flasks are used for standard and spike preparation and sample dilution.

6.4 Vials – 40mL vials with Teflon lined septa.

6.5 OI Analytical MPM 16 position samplers employing needle type spargers.

6.6 OI Analytical 4560 and 4660 sample concentrators.

6.7 OI Analytical 4552 (Archon) soil/water autosampler.

6.8 OI Analytical #10 trap or equivalent for 8021 or 8260 analysis, #7 trap for 8015 and 8021 Aromatics. Other traps may be used for 602 or 8015 analysis provided method criteria is met.

6.9 pH paper – narrow range (0.0-3.0) and wide range (1-12)

6.10 Potassium iodide – starch paper (residual chlorine check)

7.0 Procedure for Aqueous Samples

7.1 Prior to sample analysis, the GC system must be calibrated. The procedures for the determinative methods give details on preparation of standards for initial and daily calibrations. The GC-MS methods also require instrument tuning prior to proceeding with calibration.

7.2 Adjust the purge gas flow rate to 30-40mL/min on the purge-and-trap device. Once the flow is optimized, it is not necessary to set the flow daily, although periodic checking is recommended.

7.3 Traps shall be conditioned according to manufacturer's instructions upon installation. Prior to use, condition the trap daily by stepping the sample concentrator to the "bake" mode. Since bake temperature will vary according to method and trap type, ensure the proper concentrator method is loaded when performing the daily conditioning.

7.4 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.5 OI Analytical 4552 (Archon) Procedure

7.5.1 Prepare aqueous standards by injecting aliquots of standard solutions into a 50mL volumetric flask and inverting several times to mix. . The standard is then transferred to a 40mL vial with Teflon-lined septa cap.

7.5.2 Load standards, samples, and blanks into the auto-sampler tray. Program the auto-sampler methods to perform 5mL purges and dilutions when appropriate. Link auto-sampler methods together to build the run

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sequence. Without disturbing the vial seal, the Archon will transfer a 5mL volume from the vial and transfer it to the purge-and-trap device, along with a 1uL aliquot of internal standard/surrogate mix.

Caution: Care must be taken when programming the Archon. If the sample size exceeds the capacity of the purge vessel, catastrophic damage to the GC system may result! Consult the user manual if unfamiliar with use of the Archon.

7.6 Sixteen Position Auto-sampler (MPM-16) Procedure

7.6.1 For 16 position autosampler units, flush about 5mL of warmed reagent water through each sample valve that was used on the previous run. Then remove the glassware from those positions. Step the concentrator through each of those positions while holding a purge vessel under the sparger to remove any sample that may remain in the purge line. Replace the glassware with clean purge vessels that have been dried at 105C.

7.6.2 Remove the plunger from the 5mL luer-lok syringe to be used. Allow the sample to come to ambient temperature. Observe the sample for any headspace or signs of leakage and note it in the bench book if needed. Carefully pour the sample into the syringe barrel, replace the plunger, and adjust the sample volume.

7.6.3 Standards are prepared by injecting aliquots of standard solutions into a 5mL luer-lok syringe filled with DI water.

7.6.4 Sample dilutions, if needed, may be made up to 1000x directly in the luer-lok syringe by filling the syringe with reagent water and adding an aliquot of sample using a microsyringe. Dilutions greater than 1000x should be made in a volumetric flask and then transferred to the luer-lok syringe.

7.6.5 Add 10uL of surrogate spiking solution through the luer end of the syringe. Add matrix-spiking solutions as needed. Immediately, attach the syringe to the sample valve on the purging device. Open the valve and inject the sample into the purging chamber. Close the valve and remove the syringe.

7.7 Purge the sample for the time specified in the determinative method SOP.

7.8 Place the purge-and-trap system in the desorb mode and preheat the trap to the temperature specified in the method SOP. Start the flow of carrier gas, begin the GC temperature program, and start GC data acquisition. Continue the carrier flow for the time specified in the method SOP.

7.9 Recondition the trap by returning the purge-and-trap device to the purge mode. Maintain the trap temperature and bake time as specified in the method SOP.

7.10 Note: Modern purge-and-trap units are computerized and user programmable. The steps discussed in 7.7 to 7.9 are normally automatically controlled

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throughout an analysis sequence and are not necessarily manually performed by the analyst. Systems using an Archon headspace sampler also incorporate additional steps (such as hot water rinses of the purge chamber) between analysis that may reduce contamination.

7.11 Calculate response factors (RF) or calibration factors (CF) for each analyte of interest using the procedure described in Method 8000. Update the calibration levels in the MS Chemstation method and evaluate the response factors. Specific calibration requirements are described in the determinative methods and in Method 8000. The method must be saved to the hard drive after all levels are updated.

7.12 Any compound of interest that exceeds the initial calibration range should be diluted and reanalyzed so that the highest peak of interest falls in the upper half of the linear range of the curve.

7.13 After analyzing the sample, remove the sample cap. Using narrow range pH test paper (0.0 – 3.0) check the sample pH. If the pH is found to be > 3.0, use wide range pH test paper (1 – 12) to check the sample pH. Check for presence of residual chlorine with potassium iodide / starch test paper. Quickly recap the vial. Record sample pH and presence / absence of residual chlorine.

8.0 Analysis of water-miscible liquids

Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with reagent water.

9.0 Soil extracts from Method 5035

9.1 The methanol soils extracts prepared according to Method 5035 are diluted into reagent water at least 50-fold by addition of an aliquot of the methanol extract into a 5-mL syringe filled with reagent water. The volume of methanol extract added to 5-mL should not exceed 100-uL. The volume of methanol added to 5-mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of clean methanol is necessary to maintain a volume of 100-uL per 5-mL of water. Higher dilutions are made by introducing smaller aliquots of methanol extract (not less than 1uL into 5mL), or by using a volumetric flask.

9.2 The water/methanol sample is introduced as a water sample as previously described.

9.3 The purge temperature is either ambient or heated depending on the system and method used. All standards and spikes must be treated in the same manner as the samples.

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10.0 Quality Assurance and Quality Control

10.1 A sample batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours. **NOTE:** Some project plans may require different batch definitions.

10.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be analyzed with each new batch of samples.

11.0 Safety and Waste Disposal

11.1 Safety

11.1.1 The analyst should follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats.

11.1.2 The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment should be used by all analysts.

11.2 Waste Disposal

11.2.1 Expired and purged water samples are rinsed down the drain with large amounts of water.

11.2.2 Expired standards are disposed of in the waste vial drum.

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12.0 References

12.1 SW-846 Method 5030B, Rev. 2, 12/96

12.2 SW-846 Method 5030C, Rev. 3, 05/03

12.3 SW-846 Method 5035A, Rev. 1, 07/02

12.4 SW-846 Method 5000, Rev. 0, 12/96

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STANDARD OPERATING PROCEDURE FOR THE INTRODUCTION OF VOLATILE ORGANICS ANALYTES IN SOIL USING CLOSED SYSTEM PURGE-AND-TRAP

Prepared by: Rick Watkins Date: 03/21/06

Reviewed by: Norm Farmer Date: 03/21/06

Annual Review

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TEST NAME: **STANDARD OPERATING PROCEDURE FOR THE INTRODUCTION OF VOLATILE ORGANICS ANALYTES IN SOIL USING CLOSED SYSTEM PURGE-AND-TRAP**

Method: SW846 5035A

Dept: OP

1.0 Summary, Scope and Application

1.1 Summary

1.1.1 This method is performed in accordance with SW846 method 5035A.

1.1.2 Solid samples are collected in the field using techniques that are designed to minimize volatilization loses incurred during sampling, transportation and storage prior to analysis. Samples are collected using a modified syringe or Encore™ sampler designed to hold approximately 5 grams of material.

1.1.3 Samples for low concentration analysis, ≤200 ug/kg, collected in modified syringes are transferred to pre-weighed vials containing 5ml of reagent water and a magnetic stir bar. Low concentration samples collected in Encore™ samplers are transported to the laboratory and transferred to pre-weighed vials containing 5ml of reagent water and a magnetic stir bar.

1.1.4 Samples for high concentration analysis, >200 ug/kg, collected in modified syringes are transferred to pre-weighed vials containing 5ml of methanol. High concentration samples collected in Encore™ samplers are transported to the laboratory and transferred to pre-weighed vials containing 5ml of methanol. The soil must be completely covered by the methanol. In some cases, a measured addition of methanol may be required to cover the soil.

1.1.5 Unknown concentration samples, targeted for low level analysis may be screened to determine if analysis should be performed using low level or high level techniques.

1.2 Scope and Application

1.2.1 The following method describes the procedures used by Accutest Laboratories for the collection and preservation of solid samples for volatile organics analysis.

1.2.2 This procedure is designed for the sampling and preservation of sediments, soils, filter cakes, oily wastes, and tars.

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1.2.3 This method is used in conjunction with other SW846, 8000 series instrumental methods, including, but not limited to, Methods 8015, 8021, and 8260. This SOP describes the procedures employed for the sampling and sample preservation components of SW846 5035A. Sample introduction techniques are described in the SOP for the appropriate determinative method.

1.2.4 Techniques are described for collecting solid samples for analysis at two different target concentration ranges; low level concentration, ≤200 ug/kg and high level concentration, >200ug/kg.

2.0 Discussion and Comments

This method requires a heated purge for low level soil analysis. It is important to use a high quality vial with low bleed Teflon lined septa. Lower quality septa, which may work well for SW846 5030B samples, may produce very large interfering peaks due to the material used in the septa.

3.0 Preservation and Holding Times

3.1 Laboratory Preservation of Samples

3.1.1 Place a pre-tared 40ml vial containing preservative solution (water or methanol) on a calibrated top-loading balance.

3.1.2 Place the Encore™ sampler into the extruder tool.

3.1.3 Remove the cap from the Encore™ sampler. Record the condition of the vial and any odors noted in the sample prep logbook.

3.1.4 Carefully transfer the entire contents of the Encore™ sampler to the vial. Ensure the vial top is free of soil and tightly cap the vial. Read and record the sample weight in the logbook. Store the vials, if not immediately analyzed, at < –10 ^{0}C.

3.2 Holding Times

3.2.1 Samples collected in Encore™ units, or otherwise not preserved in the field, are transferred to preservative solution within 48 hours of collection.

3.2.2 Samples collected in low level vials containing reagent water must be stored at < –10 ^{0}C within 48 hours of collection.

3.2.3 All samples are analyzed within 14 days of sampling unless otherwise specified by the contract.

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4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or samples loaded on an instrument within the same 12-hour shift, which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

4.10 Trip Blank: A sample of analyte-free matrix taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organic samples.

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5.0 Reagents

5.1 Reagent water – distilled or de-ionized water free of interferences

5.2 Methanol – purge-and-trap grade or equivalent

5.3 Surrogate Solution – prepared in methanol at a concentration specified by the analyst.

5.4 Standard Solution – prepared in methanol or water at a concentration specified by the analyst.

5.5 Internal Standard Solution – prepared in methanol at a concentration specified by the analyst.

5.6 Spike Solution – prepared in methanol at a concentration specified by the analyst.

6.0 Glassware and Apparatus

6.1 Syringes

6.1.1 10cc graduated nylon syringe (Becton & Dickenson or equivalent).

6.1.2 Modify syringe by removing the end at the zero (0) cc mark. The modified device is an open cylinder with plunger.

6.2 Sampling Vials

Container - 40 ml glass screw-cap VOA vial with Teflon-faced silicone septum.

The vials are purchased as pre-cleaned and certified. These vials are used to prepare low-level and high-level soil vials with the appropriate preservative.

Note that for low level analysis, good quality low bleed septa are important. Septa that will work well for water samples may show excessive bleed when heated.

6.3 Balances - Top loading balance capable of weighing +/- 0.01 gram.

6.4 Magnetic Stirring Bar - (disposable) Teflon coated

6.5 Encore™ Samplers

6.5.1 Encore™ Sampler – En Novative Technologies, Green Bay, Wisconsin

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6.5.2 Encore™ Sample Extruder – En Novative Technologies, Green Bay, Wisconsin

7.0 PROCEDURE

7.1 Soil Vial Preparation

Note: An alternative to the following procedure for soil vial preparation, pre-made low-level and high level vials may be purchased and used for in-house preservation of samples or assembly of sampling kits to be used in the field.

7.1.1 Use the "Soil Vial Prep Log" to obtain the next lot number in sequence. Fill in the information required in the logbook and print ten sheets of labels to prepare one case of vials. Assign an expiration date for the lot that is three months from the date of preparation.

7.1.2 Apply a label to each vial in the lot, taking care to avoid large air bubbles or folds in the label that may interfere with the auto-sampler operation.

7.1.3 To prepare low-level soil vials, uncap and add small, Teflon coated magnetic stir bar and approximately 5ml of reagent water to each vial. Tightly re-cap each vial and place in the original box.

7.1.4 To prepare high-level soil vials, first check the calibration of the bottle-top methanol dispenser using a 5ml volumetric flask. Adjust the dispenser so that it dispenses exactly 5ml of methanol. Record the calibration check in the dispenser calibration log. Uncap and add 5ml of purge-and-trap grade methanol to each vial. Tightly re-cap each vial and place in the original box.

7.1.5 From each case of low-level or high-level vials, remove one vial for lot testing. Place the case of vials in the quarantine area until the results of the lot test have cleared the vials for use. Once cleared for use, the vials may be transferred to the storage area in the Volatiles Lab.

7.1.6 Prior to use (either in-house or as field kits), the vials must be weighed. Record the weigh of each vial to 0.01 gram on the appropriate label.

7.2 Selection of Analytical Level

7.2.1 Prior sample history, if available, can be used to determine analysis level. The sample-prep logbook can also help determine analysis level. If no data is available or high levels are expected, the sample should first be analyzed using high level procedures.

7.2.2 Any samples requiring dilution must be analyzed using high level techniques.

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7.3 Low Level Soil Method

7.3.1 Initial calibration of the system is accomplished using the appropriate analytical SOP as a guide. The standards are prepared at levels that range from 5ppb to 200ppb for most compounds of interest. Compounds that respond poorly or are highly water-soluble may be calibrated at higher levels.

7.3.2 The standards are prepared without the internal standard and surrogate added as they will be added by the auto-sampler.

7.3.3 Follow manufacturer's recommendations for trap conditioning and back-flushing. This method was developed using an OI Analytical #10 trap. Other trap packing materials may be used as long as method requirements are met.

7.3.4 Purge and trap operating parameters (#10 trap) :

Purge time: 11 min	Purge flow : 35 ml/min
Desorb time: 2 min	Bake time: 8 min
Desorb temp: 190 ^{0}C	Bake temp: 210 ^{0}C
Valves/Lines: 100 ^{0}C	

Auto-sampler conditions:

Method 1: Sample type, soil: Sample volume (water to be added to vial), 5ml: Standard 1, Yes: Standard 2, No: Soil preheat stir, Yes: Stir, Yes: Syringe flushes, 0: Preheat, Yes: Preheat temp, 40 ^{0}C: Preheat time, 3min

7.3.5 Perform qualitative and quantitative analysis following the guidance given in the determinative method and Method 8000. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample by the high level method.

7.4 High Level Soil Method

7.4.1 The high level method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending upon sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free reagent water, internal standards and/or surrogates are added, and the sample is purged and analyzed by the appropriate method.

7.4.2 When the high level sample is <u>not</u> preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Add the sample to a tared vial containing 5ml of methanol. Quickly cap and reweigh the vial. Record the weight to 0.01 gram.

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Note: The sample must be completely covered by the methanol. In some cases, additional methanol must be added. The amount of methanol added must be measured, and the final volume of methanol recorded in the prep-log and on the vial label.

7.4.3 The extracts are stored at < –10^{0}C in the dark, prior to analysis. Add an appropriate aliquot of the extract (not >100ul) to 5.0ml of organic-free reagent water and analyze by the appropriate determinative method.

7.4.4 The volume of methanol added to 5-mL of water being purged should be kept constant. Therefore, add to the 5-mL of water whatever volume of clean methanol is necessary to maintain a total volume of 100-uL of methanol.

7.4.5 Purge and trap operating parameters (#10 trap) :

Purge time: 11 min	Purge flow : 35 ml/min
Desorb time: 2 min	Bake time: 8 min
Desorb temp: 190 ^{0}C	Bake temp: 210 ^{0}C
Valves/Lines: 100 ^{0}C	

Auto-sampler conditions:

Method 1: Sample type, soil: Sample volume (water to be added to vial), 5ml: Standard 1, Yes: Standard 2, No: Soil preheat stir, Yes: Stir, Yes: Syringe flushes, 0: Preheat, Yes: Preheat temp, 40 ^{0}C: Preheat time, 3min

7.4.6 Purge and trap operating parameters (#7 trap) :

Purge time: 12 min	Purge flow : 35 ml/min
Desorb time: 2 min	Bake time: 8 min
Desorb temp: 180 ^{0}C	Bake temp: 200-220 ^{0}C
Valves/Lines: 100 ^{0}C	

Auto-sampler conditions:

Method 1: Sample type, soil: Sample volume (water to be added to vial), 5ml: Standard 1, Yes: Standard 2, No: Soil preheat stir, No: Stir, No: Syringe flushes, 0: Preheat, No: Preheat temp, ambient:

7.4.7 Perform qualitative and quantitative analysis following the guidance given in the determinative method and Method 8000. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample at a higher dilution.

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8.0 Quality Assurance and Quality Control

8.1 Field Blanks

Prepare soil field blanks for each field-sampling episode. Field blanks are analyzed in the laboratory to monitor for cross contamination during sampling.

8.2 Trip Blanks

Trip blanks are prepared to accompany every sample cooler. These blanks are used to determine if cross contamination occurred during sample transportation.

Soil trip blanks will be prepared for all soil samples.

8.3 Method Blanks

Method blanks are prepared and analyzed every analytical shift to demonstrate that the instrument and reagents are free from interfering contaminants. Method blanks are prepared in the same manner as samples with the exception of the addition of sample itself.

8.4 Matrix Spike and Matrix Spike Duplicate

A matrix spike duplicate pair (MS/MSD) is required to be analyzed for every analytical shift. The spike is prepared near the midpoint of the calibration range of the determinative method and is detailed in the SOP for that method.

8.4.1 MS/MSD for low concentration analysis. Select the Encore duplicate pair collected for quality control purposes and disperse the sample into a pre-weighed 40-ml purge vial containing preservative solution.

8.4.1.1 Re-weigh the sample vial and record the weight of the sample.

8.4.1.2 Spike with appropriate concentration of the matrix spike solution.

8.4.2 MS/MSD for high concentration analysis. Use methanol aliquots from the preserved sample to prepare the MS/MSD.

8.4.2.1 Add an appropriate aliquot of methanol solution to 5ml water contained in the purge and trap cell. Spike with surrogates and internal standards according to the requirements of the determinative method.

8.4.2.2 The volume of methanol added to 5-mL of water being purged should be kept constant. Therefore, add to the 5-mL of water whatever volume of clean methanol is necessary to maintain a total volume of 100-uL of methanol. The total volume of methanol added to 5-mL of water should be 100-uL, including the volume of spiking solutions.

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9.0 Safety and Waste Disposal

9.1 Safety

9.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

9.1.2 Material Safety Data Sheets (MSDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS prior to using any new reagents or solvents.

9.1.3 For samples containing high levels of VOCs, a fume hood is available in the prep area and should be used to minimize exposure to vapors.

9.2 Waste Disposal

9.2.1 Waste methanol is separated from the solid sample and is placed in the "non-chlorinated waste" container.

9.2.2 Expired soil samples are separated from the liquid portion and placed in a labeled 55-gallon drum for disposal.

10.0 References

10.1 SW-846 Method 5030C, Rev. 3, 05/03

10.2 SW-846 Method 5035A, Rev. 1, 07/03

10.3 SW-846 Method 5000, Rev. 0, 12/96

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ANALYSIS OF VOLATILE ORGANICS BY GC/MS

Prepared by: Norm Farmer Date: 03/20/06

Reviewed by: Rick Watkins Date: 03/20/06

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Document Control

Issued to: QA Department Date: _____

Issued to: MS Volatile Department Date: _____*

Issued to: _____ Date: _____

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Effective 7 days after "*" date

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TEST NAME: ANALYSIS OF VOLATILE ORGANICS BY GC/MS

METHOD REFERENCE: SW846 8260B

DEPT: MS

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of various volatile organic compounds in water and solid matrices utilizing a gas chromatograph equipped with a mass spectrometer detector. Routine compounds can be found in Table 1.

1.1.2 Reporting limits (RL) are based on the sample amount and the lowest calibration standard. Reporting limits may vary depending on matrix complications and sample volumes. Reporting limits for this method are in the range of 1.0 ug/l for aqueous samples and 5 ug/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.3 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.1.4 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I” qualifier. Some program or project specifications may require that no values below the RL be reported.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8260B.

1.2.2 Samples are received, stored and analyzed within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with Accutest SOP OP020 and OP021.

1.2.4 The samples are analyzed on a gas chromatograph equipped with mass spectrometer detector.

1.2.5 The peaks detected are identified by comparison to characteristic ions and retention times specific to the known target list of compounds.

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1.2.6 Additional unknown peaks with a response > 10% of the closest internal standard may be processed through a library search with comparison to a database of approximately 98,000 spectra. An estimated concentration is quantitated by assuming a response factor of 1.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

Aqueous Samples:

2.1.1 Samples shall be collected in standard 40mL vials with Teflon lined silicone septa. HCl is used to adjust the pH of the sample to <2.

2.1.2 The samples must be stored in capped vials, with minimum headspace, at 4°C in an area free of solvent fumes. The size of any bubble caused by degassing upon cooling should not exceed 5-6mm.

Solid Samples:

2.1.3 Special 40mL vials for purge-and-trap of solid samples, as well as the collection and preservation options are described in OP020.

2.1.4 Low level soil samples are preserved by storing them in sealed VOA vials at temperatures between –10 °C to –20 °C. High level soil samples are preserved by storing them in methanol at a ratio of 1 gram of soil to 1ml of methanol.

2.2 Holding Time

2.2.1 Aqueous samples are to be analyzed within 14 days of collection, unless otherwise specified by the contract. Samples that are not preserved should be analyzed within 7 days of collection; however, the preservation deficiency must be noted in the report.

2.2.2 Solid and waste samples must be analyzed within 14 days of collection.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory

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blanks. The use of non-TFE tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank can serve as a check on such contamination.

3.4 Contamination by carry-over can occur whenever high level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for carry-over.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or samples loaded on an instrument within the same 12-hour shift, which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Internal Standards: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Internal standards for Mass Spec methods are often deuterated forms of target analytes. Internal standards are used to compensate for retention time and response shifts during an analytical run.

4.6 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

4.7 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

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4.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.12 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.13 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

4.14 Trip Blank: A sample of analyte-free matrix taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organic samples.

5.0 REAGENTS

5.1 Methanol – purge-and-trap grade or equivalent

5.2 Volatile stock standards – Various mixes, traceable to Certificate of Analysis

5.3 4-Bromofluorobenzene (BFB) – instrument tuning mix

5.4 Surrogate standards –

Dibromofluoromethane
1,2-Dichloroethane-d4
Toluene-d8
4-Bromofluorobenzene

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5.5 Internal standards –

Fluorobenzene
Chlorobenzene-d5
1,4-Dichlorobenzene-d4
tert-Butyl alcohol-d10

6.0 APPARATUS

6.1 Gas Chromatograph – Agilent Technologies 6890 and HP 5890

6.1.1 Gas Chromatograph

The analytical system that is complete with a temperature programmable gas chromatograph and all required accessories, analytical columns, and gases.

6.1.2 The injection port is designed for split-splitless injection with capillary columns. The injection port must have an appropriate interface for sample introduction.

6.2 Mass Spectrometer– Agilent Technologies 5973 and HP 5970

The mass spectrometer must be capable of scanning from 35-300 amu every second or less utilizing 70-volt (nominal) electron energy in the electron impact ionization mode. It must also be capable of producing a mass spectrum that meets all the criteria in section 7.4.1.1 when injecting 50 ng of Bromofluorobenzene (BFB).

6.3 Purge and Trap – OI Analytical 4560 or 4660 with OI Analytical 4552

6.3.1 The following autosampler models are used for purging, trapping and desorbing the sample onto GC column.

- O.I. Model 4560 sample concentrator with 4552 Water/Soil multisampler
- O.I. Model 4660 sample concentrator with 4552 Water/Soil multisampler

6.3.2 The sample purge vial must be designed to accept 5 ml samples with a water column at least 3 cm deep.

6.3.3 The multisampler is equipped with a heater capable of maintaining the purge chamber at 40 °C to improve purging efficiency. The heater is to be used for soil and sediment analysis.

6.3.4 The desorber should be capable of rapidly heating the trap to the manufacturer recommended desorb temperature.

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6.4 Data System – Agilent Technologies MS Chemstation rev. AA03.02 and DA02.00

6.4.1 A computer system interfaced to the mass spectrometer that allows for the continuous acquisition and storage of all mass spectral data obtained throughout the duration of the chromatographic program.

6.4.2 The computer utilizes software that allows searching any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP).

6.4.3 The software should allow for integrating the abundances in any EICP between specific time or scan number limits.

6.4.4 The most recent version of the EPA/NIST mass spectral library should be available.

6.4.5 Data is archived to magnetic tape for long term storage.

6.5 Trap – OI #10 or equivalent: Tenax, Silica Gel, and Carbon Molecular Sieve. The trap should be conditioned according to the manufacturer‘s recommendations.

6.6 Columns – RTX-624 or equivalent: 60m X 0.25mm 1.4um.
– RTX-VMS or equivalent: 60m X 0.25mm 1.4um

6.7 Suitable syringes and glassware for dilutions of standards and samples.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Volatile Standards Logbook. All standards shall be traceable to their original source. The standards should be stored at temperatures between –10 °C and –20 °C, or as recommended by the manufacturer.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a “Certificate of Analysis” with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor’s expiration date. Once opened, the hold time is reduced to six months (one month for gases) or the vendor’s expiration date (whichever is shorter).

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7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with methanol. The hold time for intermediate standards is one month (one week for gases) or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicate analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the volatile organics are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the RL and the remaining standards define the working range of the detector.

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Instrument Conditions

<u>Gas Chromatograph/ Mass Spectrometer</u>

Carrier gas flow	1.2 mLs/min
Transfer line temperature	220 - 280 C
Analyzer temperature	150 C

Oven program – 45 °C for 2.0 minutes
10 °C/min to 80 °C for 0 minutes
14 °C/min to 210 °C for 0 minutes
16 °C/min to 240 °C for 4.0 minutes

OR

Oven program – 45 °C for 4.0 minutes
10 °C/min to 100 °C for 0 minutes
14 °C/min to 220 °C for 0 minutes
30 °C/min to 240 °C for 4.0 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

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7.3 Purge and Trap Device conditions

Purge Gas:	Helium at 30 ml/min
Sample Temp:	Aqueous (Ambient)
	Soils (40°C)
Trap Temp:	<25°C
Purge Time:	11 min
Desorb:	2 min. at 190°C
Bake:	8 min. at 210°C

7.4 Sample Preparation

7.4.1 Water Samples

A 5ml aliquot of sample is loaded onto the purge-and-trap device and purged for 11 minutes. Detailed procedures are described in SOP OP021.

7.4.2 Solid Samples

A 5-gram aliquot of sample is loaded onto the purge-and-trap device and purged for 11 minutes while heated to 40°C and mechanically agitated. Detailed procedures are described in SOP OP020.

7.5 Gas Chromatographic Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.5.1 Initial Calibration Procedures

Before samples can be run, the GC/MS system must be tuned, the injection port inertness must be verified, and the instrument must be calibrated.

7.5.1.1 Tune Verification (BFB)

The instrument should be hardware tuned per manufacturer's instructions. Verify the instrument tune by injecting 2ul (50ng) of BFB solution onto the instrument. The resulting BFB spectra should meet the criteria in the following table.

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BFB KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 69
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5-9 % of mass 174
176	>95% and <101% of mass 174
177	5-9% of mass 176

Evaluate the tune spectrum using three mass scans from the chromatographic peak and a subtraction of instrument background. This procedure is performed automatically by the MS Chemstation software by running “autofind” on the BFB peak.

Select the scans at the peak apex and one to each side of the apex. Calculate an average of the mass abundances from the three scans.

Background subtraction is required. Select a single scan in the chromatogram that is absent of any interfering compound peak and no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tuning compound peak.

Alternatively, the average spectra over the entire peak may be used. **All subsequent tune evaluations must use the same procedure that was used for the Initial Calibration.**

If the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are met.

Analysis must not begin until the tuning criteria are met. The injection time of the acceptable tune analysis is considered the start of the 12-hour clock. The same mass spec settings must be used for the calibration standards and samples that were used for the tune evaluation standard.

7.5.1.2 Internal Standard Calibration

A minimum 5-point calibration curve is created for the volatile organic compounds and surrogates using an internal standard

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technique. Accutest Laboratories routinely performs a 6-point calibration to maximize the calibration range.

The low point may be omitted from the calibration table for any compound with an RL set at the level two standard. Additionally the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

Response factors (RF) for each analyte are determined as follows:

$$RF = (A_{anayte} \mathbf{X} C_{istd})/(A_{istd} \mathbf{X} C_{analyte})$$

A_{anayte} = area of the analyte
A_{istd} = area of the internal standard
$C_{analyte}$ = concentration of the analyte
C_{istd} = concentration of the internal standard.

The mean RF and standard deviation of the RF are determined for each analyte. The percent relative standard deviation (%RSD) of the response factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of RF X 100) / Mean RF

If the %RSD $\leq$ 15%, linearity through the origin can be assumed and the mean RF can be used to quantitate target analytes in the samples. Alternatively if the %RSD > 15% a calibration curve of response vs. amount can be plotted. If the correlation coefficient (r) is $\geq$0.995 ($r^2 \geq$0.990) then the curve can be used to quantitate target analytes in the samples.

The method also employs a series of Calibration Check Compounds (CCC) and System Performance Check Compounds (SPCC). The %RSD for any CCC must be $\leq$ 30 % and the average relative response factor for any SPCC must be at least 0.10 or 0.30 (See tables below). If the %RSD for any CCC is $\leq$ 30% but > 15% the compound should be evaluated by curve fit.

Calibration Check Compounds (CCC)

Vinyl chloride	1,1-Dichloroethene
Chloroform	1,2-Dichloropropane
Toluene	Ethylbenzene

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System Performance Check Compounds (SPCC)

Compound	**Minimum RF**
Chloromethane	0.1
1,1-Dichloroethane	0.1
Bromoform	0.1
1,1,2,2-Tetrachloroethane	0.3
Chlorobenzene	0.3

7.5.1.3 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV should be prepared from a second source at a mid-range concentration.

The %D for all normal analytes of interest should be ≤ 25%, the %D for all poor performing (PP) analytes of interest should be ≤ 40%. (These analytes are identified in Table 1) If the %D > 25% (>40% for PP), the analysis of samples may still proceed if the analyte failed high and the analyte is not expected to be present in the samples. However, if a reportable analyte is detected in a sample and the %D for that analyte was greater than 25% (>40% for PP) in the ICV, the sample will need to be reanalyzed on a system with a passing ICV for that analyte.

If the ICV does not meet this criteria, a second standard should be prepared. If the ICV still does not meet criteria, analyze an ICV prepared from a third source. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.5.2 Continuing Calibration Verification (CCV)

7.5.2.1 Inject 2ul of the tune evaluation mix at the beginning of each 12-hour shift. Evaluate the resultant peaks against the criteria in sections 7.4.1.1 and 7.4.1.2. The injection time of this standard starts the 12-hour window.

7.4.2.2. Analyze a continuing calibration check standard. Since the method only requires one CCV per analytical batch, the level of the CCV should be varied throughout the week. At least one CCV must be below the mid-point of the calibration curve.

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7.4.2.3. The RF of check standard for SPCC compounds must meet the minimum RF requirement as listed in the table.

7.4.2.4. The %D for the CCC compounds must be ≤ 20%. If the CCCs are not part of the target list, then all target analytes must meet the 20 %D criteria.

7.4.2.5. The %D for all other analytes of interest should be ≤ 20%; however, the large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of this range. If the %D > 20%, the analysis of samples may still proceed if the analyte failed high and the analyte is not expected to be present in the samples. However, if a reportable analyte is detected in a sample and the %D for that analyte was greater than 20% in the CCV, the sample will need to be reanalyzed on a system with a passing CCV for that analyte, or the data must be qualified.

7.4.2.6. The criteria in 7.4.2.3 and 7.4.2.4 must be met for the continuing calibration to be considered valid. Additionally, only analytes that are being reported for a given sample must meet the criteria in 7.4.2.5. If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated.

7.4.2.7. If any of the internal standard area change by a factor of two (-50% to +100%) or retention time changes by more than 30 seconds from the midpoint standard of the last initial calibration, the mass spectrometer must be inspected for malfunctions and corrections made, as appropriate. Corrective action may include re-calibration (initial Calibration) of the instrument.

7.5.3 Sample Analysis

7.5.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Tune Evaluation Mix
Initial Calibration Standards (or CCV)
QC Samples
Samples

7.5.3.2 One microliter of internal standard/surrogate solution is added to every 5mL of sample in the sparge vessel. Generally, 5mL of sample are transferred to the sparge vessel.

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7.5.3.3 After purging, the system will automatically reverse flow and rapidly heat the trap to desorb the sample analytes onto the GC column.

7.5.3.4 Qualitative identification

The target compounds shall be identified by analysts with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. The criteria required for a positive identification is:

The sample component must elute at the same relative retention time (RRT) as the daily standard. The RRT of sample component must be within ± 0.06 RRT units of the standard.

All ions present in the standard mass spectra at a relative intensity greater than 10% (major abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

The relative intensities of these ions must agree within ± 30% between the daily standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%.)

Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

If peak identification is prevented by the presence of interferences, the sample must be diluted so that the interference does not mask any analytes.

7.5.3.5 Quantitative analysis

When a target compound has been identified, concentration will be based on the integrated area of the quantitation ion, which is normally the base peak.

The sample matrix may produce an interference with the primary ion. This may be characterized by an excessive background signal of the same ion, which distorts the peak shape beyond a definitive integration. The interference could also, severely inhibit the response of the internal standard ion. If an

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interference is apparent the secondary ion can be used to generate a new calibration factor.

If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that samples be diluted so that the response falls into the middle of the calibration curve.

7.5.3.6 Library search for tentatively identified compounds

If a library search is requested, the analyst should perform a forward library search of the NIST mass spectral library to tentatively identify 10 to 20 non-target compounds.

Guidelines for making tentative identification are:

These compounds should have a response greater than 10% of the nearest internal standard. The response is obtained from the Total Ion Chromatogram.

The search is to include a spectral printout of the best library match for a particular substance. The results are to be interpreted by the analyst.

Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

The relative intensities the major ions should agree within ±20%.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be verified by performing further manual background subtraction to eliminate the interference created by coeluting peaks and/or matrix interference.

Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 from the nearest internal standard and is to be tabulated on the library search summary data sheet.

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8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Internal Standards

Fluorobenzene, Chlorobenzene-d5, 1,4-Dichlorobenzene-d4, and Tert Butyl Alcohol-d10 are used as internal standards for this method. The response of the internal standard in all subsequent runs should be within a factor of two (-50% to +100%) of the internal standard response in the opening CCV for each sequence. On days that an initial calibration is performed, the internal standard responses should be compared to the internal standard responses for the mid-point standard. The response for Tert Butyl Alcohol-d10 need only be monitored if target analytes are being reported that reference that particular internal standard.

9.1.1 If the internal standard responses are not within limits, the following are required.

9.1.1.1 Check to be sure that there are no errors in calculations, integrations, or internal standards solutions. If errors are found, recalculate the data accordingly.

9.1.1.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.1.3 If no problem is found, reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then

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reanalysis may not be necessary. If there is insufficient sample for reanalysis, footnote this on the report.

9.1.1.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis should be reported on the final report.

9.2 Surrogates

9.2.1 Dibromofluoromethane, 1,2-Dichloroethane-d4, Toluene-d8, and 4-Bromofluorobenzene are used as the surrogate standards to monitor the efficiency of the purge-and-trap system.

A known amount of surrogate standard is added to each sample including the QC set prior to sparging. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for all surrogates for the results to be acceptable.

9.2.2 If any surrogate recovery is not within the established control limits, the following are required.

9.2.2.1 Check to be sure that there are no errors in calculations, integrations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly.

9.2.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.2.2.3 If no problem is found, reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then reanalysis may not be necessary. If there is insufficient sample for reanalysis, footnote this on the report

9.2.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis should be reported on the final report.

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9.3 Method Blank

9.3.1 The method blank is de-ionized water to which the surrogate standard has been added. The method blank is then purged along with the other samples to determine any contamination from the system or ambient sources. The method blank must be free of any analytes of interest or interferences at ½ the required reporting level to be acceptable. Common laboratory contaminants such as methylene chloride must be present below the RL. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.3.2 If the MB is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

9.3.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination should be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

9.3.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination should be investigated and documented. The samples should be reanalyzed for confirmation. If there is insufficient sample to reanalyze, or if the sample is reanalyzed beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4 Blank Spike

9.4.1 The blank spike is de-ionized water to which the surrogate standard and spike standard have been added. The blank spike is then processed along with the other samples to monitor the efficiency of the purge-and-trap procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable. The large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of the established control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary.

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Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A marginal exceedance in the Blank Spike is defined as a recovery being outside of 3 standard deviations but within 4 standard deviations of the mean.

The number of allowable marginal exceedances is based on the number of analytes in the Blank Spike. Marginal Exceedances must be random. If the same analyte exceeds the BS control limits repeatedly, it is an indication of a systematic problem and corrective action must be taken.

The number of allowable marginal exceedances is as follows:

1) > 90 analytes in BS, 5 analytes allowed in ME range;

2) 71-90 analytes in BS, 4 analytes allowed in ME range;

3) 51-70 analytes in BS, 3 analytes allowed in ME range;

4) 31-50 analytes in BS, 2 analytes allowed in ME range;

5) 11-30 analytes in BS, 1 analyte allowed in ME range;

6) < 11 analytes in BS, no analytes allowed in ME range

9.4.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.4.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.4.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary and the sample results can be reported without qualification.

9.4.2.4 If the recovery of an analyte in the BS is high and the associated sample is non-detect, then the sample results can be reported without qualification.

9.4.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best

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for each particular sample. That may include reanalyzing the samples or qualifying the results as estimated.

9.4.2.6 If there is insufficient sample to reanalyze, or if the sample is reanalyzed beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4.2.7 Because of their problematic nature, 2-Chloroethyl-vinyl-ether and acrolein are generally not evaluated in the blank spike unless they are of specific concern for a given project.

9.5 Matrix Spike and Matrix Spike Duplicate

9.5.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then processed along with the other samples to monitor the precision and accuracy of the purge-and-trap procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.5.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.5.2.1 Check to be sure that there are no errors in calculations, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly.

9.5.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.5.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for reanalysis, but are an indication of the sample matrix effects.

9.5.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

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RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If more than 33% of the RPDs fall outside of the established control limits, the department supervisor shall review the data and determine if any corrective action is necessary. RPD failures are generally not grounds for batch reanalysis.

10.0 CALCULATIONS

The concentration of each target compound in the original sample is calculated as follows:

Water (ug/l) = ($CONC_{inst}$) X DF

Soil (ug/kg) = [($CONC_{inst}$) X (5 / W_I)] / %solids (*low level soils)*

Soil (ug/kg) = [($CONC_{inst}$) X (V_F / V_A) X (5 / W_I) X DF] / %solids (*high level soils)*

$CONC_{inst}$	=	Instrument concentration calculated from the initial calibration using mean RF, linear curve, or quadratic curve
DF	=	Dilution Factor
V_F	=	Volume of methanol extract (ul)
V_A	=	Volume of methanol aliquot (ul)
W_I	=	Weight of sample (g)
%solids	=	Dry weight determination in decimal form

For high level soils, if %solids is < 90% then V_F is calculated as follows:

V_F = {ml of solvent + [(%moisture X W_I) / 100]} X 1000 ul/ml

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst should follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment should be used by all analysts.

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11.2 Pollution Prevention

Waste solvents from the sample analysis, methanol extraction, and standards preparation are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Old stock standards are disposed of in the waste vial drum.

Samples are archived and stored for 30 days after analysis. Spent aqueous samples and those deemed non-hazardous are rinsed down the drain with large amounts of water.

Aqueous samples that are deemed hazardous are disposed of in the aqueous waste drum.

Soil samples are disposed of in the soil waste drum.

12.0 REFERENCES

SW846 Method 8000B Revision 2, December 1996

SW846 Method 8000C Revision 3, March 2003

SW846 Method 8260B Revision 2, December 1996

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TABLE 1

Routine Target Analytes

Dichlorodifluoromethane	**2-Butanone**	n-Propylbenzene
Chloromethane	1,1-Dichloropropene	Bromobenzene
Vinyl Chloride	Propionitrile	1,1,2,2-Tetrachloroethane
Bromomethane	Methacrylonitrile	1,3,5-Trimethylbenzene
Chloroethane	Benzene	2-Chlorotoluene
Trichlorofluoromethane	TAME	**trans-1,4-Dichloro-2-Butene**
Ethyl Ether	1,2-Dichloroethane	1,2,3-Trichloropropane
1,2-Dichlorotrifluoroethane	Trichloroethene	**Cyclohexanone**
1,1-Dichloroethene	Methylcyclohexane	4-Chlorotoluene
Freon 113	Dibromomethane	tert-Butylbenzene
Carbon Disulfide	1,2-Dichloropropane	1,2,4-Trimethylbenzene
Iodomethane	Bromodichloromethane	**Pentachloroethane**
Acrolein	Methyl methacrylate	sec-Butylbenzene
Allyl chloride	**2-Chloroethyl vinyl ether**	4-Isopropyltoluene
Methylene Chloride	cis-1,3-Dichloropropene	1,3-Dichlorobenzene
Acetone	Toluene	1,4-Dichlorobenzene
Methyl acetate	2-Nitropropane	n-Butylbenzene
trans-1,2-Dichloroethene	4-Methyl-2-pentanone	**Benzyl Chloride**
Hexane	trans-1,3-Dichloropropene	1,2-Dichlorobenzene
Methyl Tert Butyl Ether	Tetrachloroethene	**1,2-Dibromo-3-Chloropropane**
Acetonitrile	Ethyl methacrylate	Hexachlorobutadiene
Di-isopropyl ether	1,1,2-Trichloroethane	1,2,4-Trichlorobenzene
Chloroprene	Dibromochloromethane	Naphthalene
1,1-Dichloroethane	1,3-Dichloropropane	1,2,3-Trichlorobenzene
Acrylonitrile	1,2-Dibromoethane	**Ethanol**
ETBE	**2-hexanone**	Tert Butyl Alcohol
Vinyl acetate	1-Chlorohexane	Isobutyl alcohol
cis-1,2-Dichloroethene	Ethylbenzene	Tert Amyl Alcohol
2,2-Dichloropropane	Chlorobenzene	**1,4-Dioxane**
Bromochloromethane	1,1,1,2-Tetrachloroethane	ETBA
Cyclohexane	m,p-Xylene	Tert Amy Alcohol
Chloroform	o-Xylene	Tert Butyl Formate
Ethyl acetate	Styrene	
Tetrahydrofuran	Bromoform	
Carbon Tetrachloride	Isopropylbenzene	
1,1,1-Trichloroethane	cis-1,4-Dichloro-2-butene	

Bolded analytes are considered “Poor Performing”

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STANDARD OPERATING PROCEDURE FOR THE TOXICITY CHARACTERISTIC LEACHING OF SEMIVOLATILE ORGANICS AND METALS (TCLP)

Prepared by: Norm Farmer Date: 02/20/06

Reviewed by: Stephanie Weber Date: 02/20/06

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Document Control

Issued to: QA Department Date: _____

Issued to: Organics Prep Department Date: _____*

Issued to: _____ Date: _____

Issued to: _____ Date: _____

Issued to: _____ Date: _____

Effective 7 days after "*" date

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TEST NAME: STANDARD OPERATING PROCEDURE FOR THE TOXICITY CHARACTERISTIC LEACHING OF SEMIVOLATILE ORGANICS AND METALS (TCLP)

Method: SW846 1311

Dept: OP

1.0 Summary, Scope and Application

1.1 Summary

For liquid and aqueous samples containing less than 0.5% solids, the sample is filtered through TCLP filter paper and the filtrate is defined as the TCLP leachate. The leachate can then be analyzed for semivolatile organics and metals.

For solid samples, the solid portion of the sample is extracted by adding extraction fluid equal to 20 times the weight of the sample and rotating the sample for 18 hours at 30 rpm. The extraction fluid used is based on the alkalinity of the solid portion of the sample. After leaching, the sample is filtered through TCLP filter paper. The leachate can then be analyzed for semivolatile organics and metals.

1.2 Scope and Application

This procedure is applicable to samples submitted for TCLP semivolatile analysis and/or TCLP metals analysis.

1.2.1 Metals by 6010

1.2.2 Mercury by 7470

1.2.3 Semivolatiles by 8270

1.2.4 Pesticides by 8081

1.2.5 Herbicides by 8151

1.2.6 Extractable TPH by 8015

2.0 Discussion and Comments

This procedure is adapted from SW-846 method 1311. The method utilizes an extraction bottle and rotary agitation device to evaluate the presence and mobility of semivolatile analytes and metals. It is not applicable for evaluating the mobility of volatile analytes.

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3.0 Preservation and Holding Times

3.1 Preservation

3.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples. Liquid samples for the analysis of metals only may be collected in HDPE bottles.

3.1.2 The samples must be protected from light and refrigerated at 4°C from the time of collection until leaching.

3.1.3 Samples for TCLP analysis should not be chemically preserved prior to leaching. After filtration, the TCLP leachate for metals should be preserved to a pH <2 with nitric acid unless the leachates will be digested immediately.

3.1.4 TCLP Leachates for semivolatile organics must be protected from light and stored at 4°C from the time of filtration until extraction.

3.2 Holding Time

3.2.1 Samples submitted for the analysis of semivolatile organics including pesticides and herbicides must be leached within 14 days of collection.

3.2.2 Samples submitted for the analysis of mercury must be leached within 28 days of collection.

3.2.3 Samples submitted for the analysis of metals (except mercury) must be leached within 180 days of collection.

3.2.4 Leachates for semivolatile organics must be extracted by the appropriate procedure within 7 days of filtration.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

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4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Leachate Blank Spike (LBS): An aliquot of TCLP fluid spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Leachate blank spike recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.8 Leachate Blank (LB): An aliquot of TCLP fluid to which all reagents are added in the same volumes or proportions as used in sample processing. The leachate blank is processed simultaneously with the samples through all the steps of the analytical procedure. The Leachate blank is used to document contamination resulting from the analytical process.

4.9 Leachate Spike (LS): A sample leachate aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The leachate spike recoveries are used to document the bias of a method in a given sample matrix.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

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5.0 Reagents

5.1 Reagent water – distilled or deionized - free of interferences

5.2 Hydrochloric Acid, 1.0N Fisher brand or equivalent

5.3 Sodium Hydroxide, 10.0N Fisher brand or equivalent

5.4 Glacial Acetic Acid, Reagent Grade

5.5 Extraction Fluid #1: Add 57 ml of glacial acetic acid and 64.3 ml of 10.0N sodium hydroxide to a 1000ml graduated cylinder and dilute to 1 liter with reagent water. Transfer to a PTFE lined carboy. Using a 1000ml graduated cylinder, transfer an additional 9 liters of reagent water to the carboy. Mix thoroughly. This will prepare 10 liters of Fluid #1. The preparation of all TCLP fluids must be logged in the Organics Reagent Logbook and the pH must be verified prior to use. The pH of this solution should be 4.93 $\pm$ 0.05.

NOTE: If the pH of the fluid is out of range, remake the fluid.

5.6 Extraction Fluid #2: Add 57 ml of glacial acetic acid to a 1000ml graduated cylinder and dilute to 1 liter with reagent water. Transfer to a PTFE lined carboy. Using a 1000ml graduated cylinder, transfer an additional 9 liters of reagent water to the carboy. Mix thoroughly. This will prepare 10 liters of Fluid #2. The preparation of all TCLP fluids must be logged in the Organics Reagent Logbook and the pH must be verified prior to use. The pH of this solution should be 2.88 $\pm$ 0.05.

NOTE: If the pH of the fluid is out of range, remake the fluid.

6.0 Glassware and Apparatus

6.1 Agitation apparatus – Environmental Express, Millipore Corp., or equivalent. Must be capable of rotating the extraction vessels in an end-over-end fashion at 30 $\pm$ 2 rpm.

6.2 Extraction Vessels – 2 liter PTFE coated HDPE bottles

6.3 Filtration device - Millipore Corp.142 mm, or equivalent, capable of exerting pressures of up to 50 psi

6.4 Filters – Environmental Express or equivalent, 0.7um glass fiber, 142 mm diameter. Filters are acid washed by the manufacturer.

6.5 pH meter - capable of reading $\pm$ 0.05 pH units

6.6 Balance - capable of weighing $\pm$ 0.01 g

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6.7 Graduated cylinders – 100ml and 1000ml

6.8 Beakers – 250 ml glass or plastic

6.9 Watchglass - appropriate to cover beaker

6.10 Magnetic Stirrer and stir bars

6.11 PTFE lined 20 liter carboy

6.12 Thermometer, calibrated against an NIST traceable thermometer

6.13 Water bath – adjustable temperature control

7.0 Procedure

7.1 The preparation of all samples must be documented. See Section 8.1 for the various logbooks and prep sheets that are required for this method. The prep sheet will include such items as: sample ID, bottle number, initial volume, final volume, pHs, lot numbers, batch numbers, and dates. The extraction technician is responsible for filling out all the required information. A copy of the prep sheet will be submitted to the analyst with the leachates.

7.2 Determination of Percent Solids

7.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration, proceed to Section 7.5.

7.2.2 If the sample is liquid or mixed-phase (solid and liquid), proceed as follows. **NOTE:** If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as “totals” instead of TCLP.

7.2.3 Pre-weigh the filter and container that will receive the filtrate. Document all weights in the TCLP_SPLP Description Log.

7.2.4 Assemble the filtering apparatus as per the manufacturer's instructions.

7.2.5 Transfer a 100 gram aliquot of the sample to a beaker and record the weight. If a 100 gram aliquot is not available, inform the Department Supervisor.

7.2.6 Quantitatively transfer the sample aliquot to the filter apparatus. Slurries may be allowed to settle and the liquid portion filtered prior to transferring the solid portion of the sample. **NOTE**: If sample material has adhered to the sample container, obtain the weight of this residue and subtract from the total weight of the sample.

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7.2.7 Complete the assembly of the filtration device, and gradually apply pressure until fluid is expelled or 10 psi is obtained. If no fluid is expelled, gradually increase the pressure in 10 psi increments to a maximum of 50 psi. If no fluid is expelled in any 2 minute period, stop the filtration. Shut off the pressurizing gas and vent the filtration system using the top vent.

CAUTION: Do not remove flange clamps while system is pressurized! Serious injury may result.

NOTE: Instantaneous application of high pressure can cause the filter to clog prematurely.

7.2.8 The material in the filtration apparatus is defined as the solid phase.

NOTE: Some high viscosity liquids (oils, paints) will not filter under these circumstances. The material remaining within the filtration device is defined as the solid phase.

7.2.9 Remove the solid phase of the sample and the filter from the filtration apparatus. If there is a noticeable amount of filtrate entrained in the filter, then dry at 100 °C ± 20 until two successive readings yield the same value within ±1%. Record the final weight.

7.2.10 Determine the percent solids as follows:

$$\% \text{ solids} = \frac{(W - F) \times 100}{T}$$

W = Weight of sample remaining on filter
F = Weight of filter
T = Initial weight of sample used

7.2.11 If the sample contains <0.5% solids, the filtrate is defined as the sample leachate. **NOTE:** Additional aliquots may need to be filtered to generate sufficient volume for all of the analysis. Proceed to Section 7.5.10.

7.3 Determination of Particle Size

7.3.1 Evaluate the solid portion of the sample for particle size. If the solid portion of sample has a surface area equal to or greater than 3.1 cm^2/gram, then the sample does not require particle size reduction. This would apply to samples such as paper, filter material or rags.

7.3.2 Alternatively, if the solid portion of the sample is smaller than 1 cm in its narrowest dimension (i.e. is capable of passing through a 9.5 mm sieve), then the sample does not require particle size reduction.

7.3.3 If the sample does not meet the particle size criteria listed above, then prepare the solid portion of the sample for extraction by crushing, cutting, or

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grinding the sample. Care should be taken to minimize any potential metals contamination.

7.3.4 Document the sample description and any particle size reduction in the TCLP_SPLP Description Log.

7.4 Determination of Extraction Fluid

7.4.1 Transfer a 5.0 gram aliquot of the solid phase of the sample to a 250 ml beaker.

7.4.2 Add 96.5 ml of DI water to the beaker. Place a magnetic stir bar in the beaker and cover with a watch glass. Stir vigorously for 5 minutes. Measure and record the pH in the TCLP Fluid Determination Log. If the pH is <5.0, use extraction fluid #1, and proceed to step 7.5.

7.4.3 If the pH is >5.0, add 3.5 ml of 1.0N HCl and swirl gently. Cover the beaker with a watch glass and heat to 50°C for 10 minutes.

7.4.4 Allow the solution to cool. Stir vigorously for 5 minutes. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

NOTE: All pH measurements must be recorded to two places after the decimal point.

7.5 Extraction of Semivolatile Organics and Metals

7.5.1 If the sample contains 100% solids, and the sample does not need particle size reduction transfer a minimum of 100g aliquot of the sample to the extraction vessel. Record the weight to ± 0.1 gram on the TCLP_SPLP Sample Prep Sheet. Add 20 times the sample weight of the appropriate extraction fluid to the leaching vessel. Swirl gently and watch for the evolution of carbon dioxide. If no gasses are evolved, cap the container and mount on the rotary agitator. Proceed to section 7.5.4.

7.5.2 If the sample contains <0.5% solids, the filtrate obtained in step 7.2 is defined as the sample leachate. Proceed to section 7.5.10.

7.5.3 If the sample is mixed phase and/or contains >0.5% solids, transfer a 100g aliquot of the sample or more, based on % solids to the filtration device. Assemble the filtration apparatus, and gradually apply pressure to remove any free liquids. Expelled liquid is stored in a glass container, and is to be recombined with sample leachate. Transfer the solid portion of the sample to the appropriate extraction vessel. Add a volume of the appropriate TCLP fluid, calculated as follows:

$$\frac{20 \times \text{sample wt (g)} \times \text{\% solids}}{100} = \text{ml extraction fluid}$$

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Swirl gently and watch for evolution of carbon dioxide. Cap the extraction bottle and attach to rotary agitator. Allow the extraction to proceed 18 ± 2 hrs.

Note: If the sample contains <25% solids, more sample can be filtered to obtain sufficient solids for leaching such that all analyses may be performed. Consult the Department Supervisor.

7.5.4 Rotate at 30 ±2 rpm. Make sure to measure and record the rotation rate on the TCLP_SPLP Sample Prep Sheet. Allow the extraction to proceed for 18 ± 2 hrs.

The vessels should be vented periodically during the first hour to prevent pressure build up.

NOTE: The temperature of the extraction room must be 23 ± 2 °C during the extraction period. Record the temperature on TCLP_SPLP Sample Prep Sheet. Use a Hi/Lo thermometer to monitor the room temperature through out the extraction period.

7.5.5 After the leaching period has elapsed, remove the extraction vessels from the rotary agitator and allow them to settle.

7.5.6 Assemble the filtration device. Filter each sample into an appropriately labeled container. Multiple filters may be used. Be sure to thoroughly clean the filtration apparatus between samples.

7.5.7 If a compatible liquid was obtained in Section 7.5.3, combine the liquids at this time.

7.5.8 If the liquids are not compatible, submit both fractions for analysis. Notify the Department Supervisor.

7.5.9 Measure and record the pH of each leachate in the TCLP Fluid Determination Log.

NOTE: All pH measurements must be recorded to two places after the decimal point.

7.5.10 Aliquot the leachate for the necessary analyses. Listed below are the minimum quantities required for analysis. Additional aliquots will be needed for QC samples.

7.5.10.1	Metals (including Mercury)	100ml
7.5.10.2	Semivolatiles by 8270	100ml
7.5.10.3	Pesticides by 8081	100ml

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7.5.10.4	Herbicides by 8151	100ml
7.5.10.5	TPH by 8015	100ml

7.5.11 Leachates for metals analysis are stored in labeled plastic bottles and transferred to the metals department.

7.5.12 All other aliquots (semi-volatile organics) are transferred to amber glass bottles and stored at 4°C until the subsequent extractions can be performed.

8.0 Documentation

8.1 Documentation for this analysis is quite extensive. At minimum the following logbooks or prep sheets must be filled out completely.

8.1.1 TCLP_SPLP Description Log

8.1.2 TCLP Fluid Determination Log

8.1.3 TCLP_SPLP Sample Prep Sheet

8.1.4 Extraction Bottle Tracking Log

8.1.5 Organics Reagent Log

8.2 For every 20 extractions performed in an extraction vessel, a leachate blank must be performed using that vessel. Document this in the Extraction Bottle Tracking Log.

8.3 For each original matrix type extracted, (soil, water, sludge, etc.) a leachate spike must be performed. Various unique matrices may require their own leachate spikes.

9.0 Quality Assurance and Quality Control

9.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 6 hours; however, samples should not be added after the QC set has been completed. **NOTE:** All samples and QC samples must leached for the required amount of time.

9.2 A leachate blank (LB) and sample duplicate (DUP) must be leached with each batch of samples.

9.3 All spiking for the matrix spike (MS) and matrix spike duplicate (MSD), leachate spike (LS), and leachate blank spike (LBS) occurs after filtration.

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10.0 Safety and Waste Disposal

10.1 Safety

10.1.1 Safety glasses, gloves and lab coats should be worn when handling acids, samples, standards or solvents.

10.1.2 Perform all filtration in a fume hood.

10.1.3 Material Safety Data Sheets (MSDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS prior to using any new reagents or solvents.

10.2 Waste Disposal

10.2.1 The TCLP filter and remaining sample is placed in a waste container.

10.2.2 Extra leachate is rinsed down the drain with large amounts of water.

10.2.3 Waste soil from the homogenizing process should be place in the “soil waste” container. **NOTE:** Waste soil from foreign soils must follow “foreign soil” disposal requirements.

11.0 References

11.1 SW-846 Method 1311, Rev. 0, 07/92

11.2 SW-846 Method 1312, Rev. 0, 09/94

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STANDARD OPERATING PROCEDURE FOR THE TOXICITY CHARACTERISTIC LEACHING OF VOLATILE ORGANICS (TCLP)

Prepared by: Norm Farmer Date: 02/20/06

Reviewed by: Stephanie Weber Date: 02/20/06

Annual Review

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Effective 7 days after "*" date

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TEST NAME: STANDARD OPERATING PROCEDURE FOR THE TOXICITY CHARACTERISTIC LEACHING OF VOLATILE ORGANICS (TCLP)

Method: SW846 1311

Dept: OP

1.0 Summary, Scope and Application

1.1 Summary

For liquid and aqueous samples containing less than 5.0% solids, the sample is filtered via ZHE through TCLP filter paper and the filtrate is defined as the TCLP leachate. The leachate can then be analyzed for volatile organics.

For solid samples, the solid portion of the sample is extracted via ZHE by adding extraction fluid equal to 20 times the weight of the sample and rotating the sample for 18 hours at 30 rpm. After leaching, the sample is filtered through TCLP filter paper. The leachate can then be analyzed for volatile organics.

1.2 Scope and Application

This procedure is applicable to samples submitted for TCLP volatile analysis.

1.2.1 Volatiles by 8260

1.2.2 Volatiles by 8021

1.2.3 Volatile TPH by 8015

2.0 Discussion and Comments

This procedure is adapted from SW-846 method 1311. The method utilizes a zero headspace extraction (ZHE) vessel and rotary agitation device to evaluate the presence and mobility of volatile analytes. It is not applicable for evaluating the mobility of semivolatile organics or metals analytes.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples. Samples should be collected with minimal headspace.

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3.1.2 The samples must be protected from light and refrigerated at 4°C from the time of collection until leaching.

3.1.3 Samples for TCLP analysis should not be chemically preserved prior to leaching. After filtration, the TCLP leachate for volatile organics should be preserved to a pH <2 with hydrochloric acid.

3.1.4 TCLP Leachates for volatile organics must be protected from light and stored at 4°C from the time of filtration until analysis.

3.2 Holding Time

3.2.1 Samples submitted for the analysis of volatile organics must be leached within 14 days of collection.

3.2.2 Leachates for volatile organics must be analyzed by the appropriate procedure within 14 days of filtration.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

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4.7 Leachate Blank Spike (LBS): An aliquot of TCLP fluid spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Leachate blank spike recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.8 Leachate Blank (LB): An aliquot of TCLP fluid to which all reagents are added in the same volumes or proportions as used in sample processing. The leachate blank is processed simultaneously with the samples through all the steps of the analytical procedure. The Leachate blank is used to document contamination resulting from the analytical process.

4.9 Leachate Spike (LS): A sample leachate aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The leachate spike recoveries are used to document the bias of a method in a given sample matrix.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Reagent water – distilled or deionized - free of interferences

5.2 Sodium Hydroxide, 10.0N Fisher brand or equivalent

5.3 Glacial Acetic Acid, Reagent Grade

5.4 Extraction Fluid #1: Add 57 ml of glacial acetic acid and 64.3 ml of 10.0N sodium hydroxide to a 1000ml graduated cylinder and dilute to 1 liter with reagent water. Transfer to a PTFE lined carboy. Using a 1000ml graduated cylinder, transfer an additional 9 liters of reagent water to the carboy. Mix thoroughly. This will prepare 10 liters of Fluid #1. The preparation of all TCLP fluids must be logged in the Organics Reagent Logbook and the pH must be verified prior to use. The pH of this solution should be 4.93 $\pm$ 0.05.

NOTE: If the pH of the fluid is out of range, remake the fluid.

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6.0 Glassware and Apparatus

6.1 Agitation apparatus – Environmental Express, Millipore Corp., or equivalent. Must be capable of rotating the extraction vessels in an end-over-end fashion at 30 ± 2 rpm.

6.2 Zero Headspace Extraction (ZHE) Vessels – Millipore Corp., or equivalent. The vessel should have an internal volume of 500 ml and be able to accommodate a 90 mm filter. The vessel must be gas tight and free of organic contaminants.

ZHE vessels must be leak tested prior to being placed into service. Additional leak testing should be performed whenever a loss of pressure during an extraction is observed. To leak test an extractor, pressurize the ZHE to 50 psi and allow it to stand unattended for one hour, and recheck the pressure.

6.2.1 If pressure is lost, check all fittings and o-rings. Replace any worn o-rings and repeat the leak test procedure.

6.2.2 If the pressure still does not hold at 50 psi, then pressurize the extractor to 10 psi and allow it to stand unattended for one hour and recheck the pressure. If it does not hold at 10 psi, the ZHE must be removed from all service until the problem is resolved.

6.2.3 If the ZHE will hold pressure at 10 psi and can maintain a filtering pressure of 50 psi when gas pressure is applied, then it can be used for extractions. Notify the Department Supervisor that further maintenance is required on this extractor.

6.3 Filtration device – Millipore Corp.142 mm, or equivalent, capable of exerting pressures of up to 50 psi (for %solids evaluation).

6.4 Fluid metering pump – Environmental Express Model TP1200, or equivalent

6.5 Filters – Environmental Express or equivalent, 0.7um glass fiber, 90 and 142 mm diameter.

6.6 VOA Vials – 40ml preserved with HCl

6.7 Tedlar Bags

6.8 Luer Tip Syringe – 50 to 60 ml capacity.

6.9 pH meter - capable of reading ± 0.05 pH units

6.10 pH paper – narrow range (0.0-3.0)

6.11 Balance - capable of weighing ± 0.01 g

6.12 Graduated cylinders – 100ml, 250ml, and 1000ml

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6.13 PTFE lined 20 liter carboy

6.14 Thermometer, calibrated against an NIST traceable thermometer

7.0 Procedure

7.1 The preparation of all samples must be documented. See Section 8.1 for the various logbooks and prep sheets that are required for this method. The prep sheet will include such items as: sample ID, bottle number, initial volume, final volume, pHs, lot numbers, batch numbers, and dates. The extraction technician is responsible for filling out all the required information. A copy of the prep sheet will be submitted to the analyst with the leachates.

7.2 Determination of Percent Solids

7.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration, proceed to Section 7.5.

7.2.2 If the sample is liquid or mixed-phase (solid and liquid), proceed as follows. **Note:** If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as “totals” instead of TCLP.

7.2.3 Pre-weigh the filter and container that will receive the filtrate. Document all weights in the TCLP_SPLP Description Log.

7.2.4 Assemble the filtering apparatus as per the manufacturer's instructions.

7.2.5 Transfer a 100 gram aliquot of the sample to a beaker and record the weight. If a 100 gram aliquot is not available, inform the Department Supervisor.

7.2.6 Quantitatively transfer the sample aliquot to the filter apparatus. Slurries may be allowed to settle and the liquid portion filtered prior to transferring the solid portion of the sample. **NOTE**: If sample material has adhered to the sample container, obtain the weight of this residue and subtract from the total weight of the sample.

7.2.7 Complete the assembly of the filtration device, and gradually apply pressure until fluid is expelled or 10 psi is obtained. If no fluid is expelled, gradually increase the pressure in 10 psi increments to a maximum of 50 psi. If no fluid is expelled in any 2 minute period, stop the filtration. Shut off the pressurizing gas and vent the filtration system using the top vent.

CAUTION: Do not remove flange clamps while system is pressurized! Serious injury may result.

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NOTE: Instantaneous application of high pressure can cause the filter to clog prematurely.

7.2.8 The material in the filtration apparatus is defined as the solid phase.

NOTE: Some high viscosity liquids (oils, paints) will not filter under these circumstances. The material remaining within the filtration device is defined as the solid phase.

7.2.9 Remove the solid phase of the sample and the filter from the filtration apparatus. If there is a noticeable amount of filtrate entrained in the filter, then dry at 100 °C ± 20 until two successive readings yield the same value within ±1%. Record the final weight.

7.2.10 Determine the percent solids as follows:

$$\% \text{ solids} = \frac{(W - F) \times 100}{T}$$

W = Weight of sample remaining on filter
F = Weight of filter
T = Initial weight of sample used

7.2.11 If the sample contains <5.0% solids, the filtrate is defined as the sample leachate.

NOTE: This aliquot can NOT be used for the analysis of Volatile Organics. Proceed to Section 7.5.

7.3 Determination of Particle Size

7.3.1 Evaluate the solid portion of the sample for particle size. If the solid portion of sample has a surface area equal to or greater than 3.1 cm^2/gram, then the sample does not require particle size reduction. This would apply to samples such as paper or rags.

7.3.2 Alternatively, if the solid portion of the sample is smaller than 1 cm in its narrowest dimension (i.e. is capable of passing through a 9.5 mm sieve), then the sample does not require particle size reduction.

7.3.3 If the sample does not meet the particle size criteria listed above, then prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the sample. This should be performed as quickly as possible in order to minimize the amount of volatile analytes that may be lost.

7.3.4 Document the sample description and any particle size reduction in the TCLP_SPLP Description Log.

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7.4 Determination of Extraction Fluid

Samples submitted for the analysis of volatile organics by TCLP are always extracted with Fluid #1.

7.5 Zero Headspace Extraction

7.5.1 Percent solids is 100%.

7.5.1.1 If the sample is 100% solid, weigh out a 10 to 25 gram aliquot and quickly transfer to the ZHE. Record the weight to ± 0.1 gram on the TCLP_SPLP ZHE Sample Prep Sheet.

7.5.1.2 Insert the filter and screens. Seal the vessel. Connect the gas supply to the lower fitting on the ZHE. Close the lower vent.

7.5.1.3 Apply 10 to 15 psi of pressure to the ZHE. Slowly open the top valve to expel any air from the ZHE. Slowly increase the pressure to keep the piston moving. DO NOT EXCEED 50 PSI.

7.5.1.4 Once the piston will no longer move, close the tope valve.

7.5.1.5 Add 20 times the sample weight of extraction fluid to a graduated cylinder. Attach the pump and the transfer line to the ZHE.

7.5.1.6 Disconnect the gas supply from the lower ZHE fitting and open the lower vent.

7.5.1.7 Open the top valve to the transfer line and then turn on the pump. Transfer the fluid to the ZHE. After the fluid has been transferred, close the top valve and remove the transfer line.

7.5.1.8 Close the lower vent and reattach the gas supply line. Pressurize the ZHE to 25-30 psi. Disconnect the gas supply line.

7.5.1.9 Rotate the ZHE 2-3 times. Open the top valve and expel any residual headspace. Stop at the first sign of liquid. Disconnect the gas supply line.

7.5.1.10 Check the ZHE carefully for leaks and make sure that the pressure is holding to within 2 psi of the initial pressure. If not, discard the sample and set up a new aliquot in a different ZHE. Record the initial pressure on the TCLP_SPLP ZHE Prep Sheet. Proceed to section 7.5.4.

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7.5.2 Percent solids is <5.0%.

7.5.2.1 If the waste contains <5.0% solids, transfer at least 200ml of sample to the zero headspace extractor (ZHE).

7.5.2.2 Insert the filter and screens. Seal the vessel. Connect the gas supply to the lower fitting on the ZHE. Close the lower vent.

7.5.2.3 Apply 10 to 15 psi of pressure to the ZHE. Slowly open the top valve to expel any air from the ZHE. At the first appearance of any liquid, close the valve.

7.5.2.4 Attach a 50 ml Luer tip syringe to the ZHE. Open the valve to expel approximately 50 ml of filtrate. If the flow stops, gradually increase the gas pressure. Once you have collected 50 ml of filtrate, close the top valve.

7.5.2.5 Transfer the filtrate to a 40 ml VOA vial and cap the vial. Make sure that there is no headspace in the vial. Repeat the process to obtain three vials of filtrate. Store at 4°C until analysis. Proceed to section 7.5.11.

7.5.3 Mixed phase samples

7.5.3.1 If the sample is mixed-phase (solid and liquid), proceed as follows. **NOTE:** If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as “totals” instead of TCLP.

7.5.3.2 Charge the ZHE with enough sample to obtain 10-25 grams of solid using the formula shown below:

$$\text{Grams of Solid} = \frac{(\text{Grams of Total Sample}) \times (\text{\% solids})}{100}$$

7.5.3.3 Insert the filter and screens. Seal the vessel. Connect the gas supply to the lower fitting on the ZHE. Close the lower vent.

7.5.3.4 Apply 10 to 15 psi of pressure to the ZHE. Slowly open the top valve to expel any air from the ZHE. Slowly increase the pressure to keep the piston moving. At the first appearance of any liquid, close the valve.

7.5.3.5 Attach a 50 ml Luer tip syringe to the ZHE. Open the valve to expel the liquid phase. If the flow stops, gradually increase the gas pressure to a maximum of 50 psi. Once you have collected the liquid phase, close the top valve.

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7.5.3.6 Transfer the liquid phase to a Tedlar bag or a 40 ml VOA vial. Make sure that there is no headspace in the vial.

7.5.3.7 Add 20 times the sample weight of extraction fluid into a graduated cylinder. Attach the pump and the transfer line to the ZHE.

7.5.3.8 Disconnect the gas supply from the lower ZHE fitting and open the lower vent.

7.5.3.9 Open the top valve to the transfer line and then turn on the pump. Transfer the fluid to the ZHE. After the fluid has been transferred, close the top valve and remove the transfer line.

7.5.3.10 Close the lower vent and reattach the gas supply line. Pressurize the ZHE to 25-30 psi. Disconnect the gas supply line.

7.5.3.11 Rotate the ZHE 2-3 times. Open the top valve and expel any residual headspace. Stop at the first sign of liquid. Disconnect the gas supply line.

7.5.3.12 Check the ZHE carefully for leaks and make sure that the pressure is holding to within 2 psi of the initial pressure. If not, discard the sample and set up a new aliquot in a different ZHE. Record the initial pressure on the TCLP_SPLP ZHE Prep Sheet. Proceed to section 7.5.4.

7.5.4 Rotate at 30 ±2 rpm. Make sure to measure and record the rotation rate on the TCLP_SPLP ZHE Sample Prep Sheet. Allow the extraction to proceed for 18 ± 2 hrs.

NOTE: The temperature of the extraction room must be 23 ± 2 °C during the extraction period. Record the temperature on TCLP_SPLP ZHE Sample Prep Sheet. Use a Hi/Lo thermometer to monitor the room temperature through out the extraction period.

7.5.5 After the leaching period has elapsed, remove the extraction vessels from the rotary agitator and allow them to settle.

7.5.6 Check and record the pressure of each ZHE on the TCLP_SPLP ZHE Prep Sheet. If the pressure has dropped by more than 10 psi, the samples should be discarded and reprepped.

7.5.7 Attach a 50 ml Luer tip syringe to the ZHE. Connect the gas supply to the lower fitting on the ZHE. Open the top valve to expel approximately 50 ml of filtrate. If the flow stops, gradually increase the gas pressure (DO NOT EXCEED 50 PSI). Once you have collected 50 ml of filtrate, close the top valve.

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7.5.8 Transfer the filtrate to a 40 ml VOA vial and cap the vial. Make sure that there is no headspace in the vial. Repeat the process to obtain three vials of filtrate. Using narrow range pH paper, check the pH. If the pH is > 2 adjust the pH with HCl until it is < 2. Store at 4°C until analysis.

7.5.9 If a compatible liquid was obtained in Section 7.5.3.6, combine the liquids at this time. It may be easier to transfer the initial filtrate and the final leachate to a Tedlar bag for mixing.

7.5.10 If the liquids are not compatible, submit both fractions for analysis. Notify the Department Supervisor.

7.5.11 Aliquot the leachate for the necessary analyses. Listed below are the minimum quantities required for analysis. Additional aliquots will be needed for QC samples.

7.5.11.1	Volatiles by 8260	3 x 40ml
7.5.11.2	Volatiles by 8021	3 x 40ml
7.5.11.3	Volatile TPH by 8015	3 x 40ml

7.5.12 All aliquots for volatile organics are transferred to 40 ml VOA vials and stored at 4°C until analysis.

8.0 Documentation

8.1 Documentation for this analysis is quite extensive. At minimum the following logbooks or prep sheets must be filled out completely.

8.1.1 TCLP_SPLP Description Log

8.1.2 TCLP_SPLP ZHE Sample Prep Sheet

8.1.3 ZHE Tracking Log

8.1.4 Organics Reagent Log

8.2 For every 20 extractions performed in an extraction vessel, a leachate blank must be performed using that vessel. Document this in the ZHE Tracking Log.

8.3 For each original matrix type extracted, (soil, water, sludge, etc.) a leachate spike must be performed. Various unique matrices may require their own leachate spikes.

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9.0 Quality Assurance and Quality Control

9.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 6 hours; however, samples should not be added after the QC set has been completed. **NOTE:** All samples and QC samples must leached for the required amount of time.

9.2 A leachate blank (LB) and sample duplicate (DUP) must be leached with each batch of samples.

9.3 All spiking for the matrix spike (MS) and matrix spike duplicate (MSD), leachate spike (LS), and leachate blank spike (LBS) occurs after filtration.

10.0 Safety and Waste Disposal

10.1 Safety

10.1.1 Safety glasses, gloves and lab coats should be worn when handling acids, samples, standards or solvents.

10.1.2 Perform all filtration in a fume hood.

10.1.3 Material Safety Data Sheets (MSDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS prior to using any new reagents or solvents.

10.2 Waste Disposal

10.2.1 The TCLP filter and remaining sample is placed in a waste container.

10.2.2 Extra leachate is rinsed down the drain with large amounts of water.

10.2.3 Waste soil from the homogenizing process should be place in the “soil waste” container. **NOTE:** Waste soil from foreign soils must follow “foreign soil” disposal requirements.

11.0 References

11.1 SW-846 Method 1311, Rev. 0, 07/92

11.2 SW-846 Method 1312, Rev. 0, 09/94

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TITLE: CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION BY SW846 METHOD 8151A

REFERENCES: SW846 8151A (Revision 1, December 1996)
SOP: EGC8151-04

REVISED SECTIONS: 3, 7-19

1.0 Scope & Application

1.1 Method 8151 is a capillary gas chromatographic (GC) method for determining certain chlorinated acid herbicides and related compounds in aqueous, soil and waste matrices. Specifically, Method 8151 may be used to determine the following compounds:

Compound	CAS Number
2,4-D	94-75-7
2,4-DB	94-82-6
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dalapon	75-99-0
Dicamba	1918-00-9
Dichloroprop	120-36-5
Dinoseb	88-85-7
4-Nitrophenol	100-02-1
Pentachlorophenol	87-86-5
Picloram	02/01/1918

[a] Chemical Abstract Service Registry Number

1.2 Because these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), Method 8151 describes a hydrolysis step that can be used to convert herbicide esters into the acid form prior to analysis.

2.0 Summary

2.1 Method 8151 is a procedure for extracting, derivatizing, and analyzing sample extracts by gas chromatography for chlorinated acid herbicides in water, soil, and waste samples. It is also includes procedures for the hydrolysis and analysis of herbicide esters.

2.4 Water samples are extracted with diethyl ether and then esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.4 Soil and waste samples are extracted and esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.4 If herbicide esters are to be determined using this method, hydrolysis conditions for the esters in water and soil extracts are described.

3.0 Reporting Limit & Method Detection Limit

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3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve. This concentration is defined as the lower limit for quantitative accuracy. The reporting limits for the herbicides that can be determined by this method range from 5 ug/l to 500 ug/l (compound dependent). The reporting limits for specific waste samples may vary depending upon the sample size, moisture content, nature of interferences present, and the sample matrix.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs are determined annually for aqueous and solid matrices.

3.2.2 Process all raw data for the replicate analysis in each MDL study. Forward the processes data to the QA group for archiving.

4.0 Definitions

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank; Instrument Blank, Storage Blank, and Sulfur Blank**.**

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

CONTINUING CALIBRATION - analytical standard run after every 10 injections and at the end of a run to verify the initial calibration of the system.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest.

RESOLUTION - also termed separation or percent resolution, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RETENTION TIME (RT) - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SURROGATES (Surrogate Standard) - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

5.0 Health & Safety

5.1 The analyst must follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses. Specifically, concentrated sulfuric acid and the 10 N sodium hydroxide solution are moderately toxic and extremely irritating to skin and mucous membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with these reagents.

5.3 Several analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens. Primary standards of target analytes should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5.4 Only experienced analysts, due to the potential hazards associated with its use must use Diazomethane.

The following precautions should be taken:

Use a safety screen.
Use mechanical pipetting aides.
Do not heat above 90°C - EXPLOSION may result.
Avoid grinding surfaces, ground-glass joints, sleeve bearings, and glass stirrers - Explosion may result.
Store away from alkali metals - Explosion may result.

6.0 Collection, Preservation, & Holding Times

6.1 Water samples may be collected in 1 L (or 1 quart) amber glass containers, fitted with screw-caps lined with Teflon. If amber containers are not available, the samples should be protected from light. Soil samples may be collected in glass containers or closed end tubes (e.g., brass sleeves) in sufficient quantity to perform the analysis.

6.2 All samples must be iced or refrigerated at 4°C (± 2°C) from the time of collection until

extraction.

6.3 Procedure for Sample Storage

6.3.1 The samples must be protected from light and refrigerated at 4°C (±2°C).

6.3.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants.

6.4 Procedure for Sample Extract Storage

6.4.1 Sample extracts must be protected from light and stored at 4°C (± 2°C). Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants. Samples, sample extracts, and standards must be stored separately.

6.5 Holding Times

6.5.1 Extraction of water samples must be completed within seven (7) days of sample collection. Extraction of soil/sediment samples by sonication must be completed within fourteen (14) days of sample collection.

6.5.2 Analysis of sample extracts must be completed within 40 days following the start of extraction.

7.0 Apparatus and Materials

7.1 Gas Chromatographic System

7.1.1 Gas Chromatograph. HP-5890 Model which includes an analytical system completes with a temperature programmable gas chromatograph. Accessories required include syringes, capillary chromatographic columns, and gases. The capillary column is directly coupled to the source. The chromatograph has a Grob type injector.

7.1.2 GC columns. Narrow-bore columns- two required:

7.1.2.1 Primary column 1 - 30 m x 0.25 mm, 5% phenyl/95% methyl silicone (DB-5, J&W Scientific, or equivalent), 0.25 um film thickness.

7.1.2.2 Confirmation column – 30 m x 0.25 mm, 14% cyanopropyl phenyl silicone, (DB-1701, J&W Scientific, or equivalent), 0.25 um film thickness.

7.1.3 Electron capture detector (ECD).

7.2 Data System

7.2.1 HP Chemstation, interfaced to the gas chromatograph which allows the continuous acquisition and storage on machine readable media (disc) of all chromatographic data obtained throughout the duration of the analysis.

7.2.2 The ENVIROQUANT data system is capable of quantitation using multi-point calibration.

7.2.3 Lagato Networker with lookup database on 4mm DAT tape for long term, off line magnetic storage of data.

7.3 Autosampler

7.3.1 Hewlett Packard Model 7673A, capable of holding 100 of 2-ml crimp vials.

7.4 Kuderna-Danish (K-D) apparatus.

7.4.1 Concentrator tube - 10-mL graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.

7.4.2 Evaporation flask - 500-mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

7.4.3 Snyder column - Three-ball macro (Kontes K-503000-0121 or equivalent).

7.5 Diazomethane generator.

7.5.1 Assemble from two 20 mm x 150 mm test tubes, two Neoprene rubber stoppers, and a source of nitrogen. Use Neoprene rubber stoppers with holes drilled in them to accommodate glass delivery tubes. The exit tube must be drawn to a point to bubble diazomethane through the sample extract. The generator assembly is shown in Figure 1. The procedure for use of this type of generator is given in Sec. 10.7

7.6 Beaker - 400-mL, thick-walled.

7.7 Funnel - 75mm diameter.

7.8 Separatory funnel - 500-mL, with polytetrafluoroethylene (PTFE) stopcock.

7.9 Centrifuge bottle - 250-mL, Pyrex® 1260 or equivalent.

7.10 Erlenmeyer flasks - 250-mL and 500-mL, with a ground-glass joint at the neck.

7.11 Pipet - Pasteur, glass, disposable (140 mm x 5 mm ID).

7.12 Vials - 10-mL, glass, with PTFE-lined screw-caps.

7.13 Volumetric flasks, Class A - 10-mL to 1000-mL.

7.14 Filter paper - 15cm diameter (Whatman No. 1 or equivalent).

7.15 Glass wool - Pyrex®, acid washed.

7.16 Boiling chips - Solvent-extracted with methylene chloride, approximately 10/40 mesh (silicon carbide or equivalent).

7.17 Water bath - Heated, with concentric ring cover, capable of temperature control (± 2 C). The bath should be used in a hood.

7.18 Balance - Analytical, capable of accurately weighing to 0.0001 g.

7.19 Centrifuge.

7.20 Ultrasonic extraction system - A horn-type device equipped with a titanium tip, or a device that will give equivalent performance, should be used. The disrupter must have a minimum power wattage of 300 watts, with pulsing capability. A device designed to

reduce the cavitation sound is recommended. Follow the manufacturer's instructions for preparing the disrupter for extraction of samples. Use a 3/4" horn for most samples.

7.21 Sonabox - Recommended with above disrupters for decreasing cavitation sound (Heat Systems - Ultrasonics, Inc., Model 432B or equivalent).

7.22 pH paper - wide range

7.23 Silica gel cleanup column (Bond Elut - Analytichem, Harbor City, CA or equivalent).

7.24 Drying column - 400 mm x 20 mm ID Pyrex® chromatographic column with Pyrex® glass wool at bottom and a PTFE stopcock.

7.25 Nitrogen blowdown apparatus.

8.0 Standards & Reagents

8.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 Organic-free reagent water. All references to water in this method refer to organic-free water, as defined in Chapter One.

8.3 Sodium hydroxide solution (0.1 N), NaOH - Dissolve 4 g of NaOH in organic-free reagent water and dilute to 1.0 L.

8.4 Potassium hydroxide solution (37% aqueous solution (w/v)), KOH - Dissolve 37 g of potassium hydroxide pellets in organic-free reagent water and dilute to 100 mL.

8.5 N-methyl-N-nitroso-p-toluenesulfonamide (Diazald) - High purity (Aldrich Chemical Co., or equivalent).

8.6 Silicic acid, H_2SiO_5 - 100-mesh powder, store at 130°C.

8.7 Sodium sulfate (granular, acidified, anhydrous), Na_2SO_4 - Purify by heating at 400°C for 4 hours in a shallow tray. Acidify by slurrying 100g sodium sulfate with enough diethyl ether to just cover the solid; then add 0.1 mL of concentrated sulfuric acid (or equivalent ratio) and mix thoroughly. Remove the ether by placing in a hot water bath. Mix 1 g of the resulting solid with 5 mL of organic-free reagent water and measure the pH of the mixture. It must be below a pH of 4. Store the remaining solid in the 50±5°C drying oven.

8.8 Solvents - All solvents should be pesticide quality or equivalent.

8.8.1 Methylene chloride, CH_2Cl_2.

8.8.2 Acetone, CH_3COCH_3.

8.8.3 Methanol, CH_3OH.

8.8.4 Toluene, $C_6H_5CH_3$.

8.8.5 Diethyl Ether, $C_2H_50C_2H_5$. Must be free of peroxides as indicated by test strips (EM

Quant, or equivalent). Procedures for removal of peroxides are provided with the test strips.

NOTE: Diethyl ether used for this procedure should be stabilized with BHT, not with ethanol, as when ethanol-stabilized ether is used, the methylation reaction may not proceed efficiently, leading to low recoveries of target analytes.

8.8.6 Isooctane, $(CH_3)_3CH_2CH(CH_3)_2$.

8.8.7 Hexane, C_6H_{14}.

8.8.8 Carbitol (diethylene glycol monoethyl ether), $C_2H_5OCH_2CH_2OCH_2CH_2O$ - optional, for producing alcohol-free diazomethane.

8.9 Stock standard solutions.

8.9.1 Calibration – Herbicide methyl esters -Varied concentration (11 compound mix) certified derivatized solutions #92510 commercially purchased from Absolute (or equivalent).

8.9.2 Continuing Calibration Verification second source- Herbicide methyl esters -Varied concentration (11 compound mix) certified derivatized solutions #92510, different lot #, commercially purchased from Absolute (or equivalent).

8.9.3 Store the stock standard solutions at 4°C, protected from light. These solutions should be checked frequently for signs of degradation or evaporation, especially immediately prior to preparing calibration standards from them.

8.10 Intermediate Calibration Standard Solution.

8.10.1 The intermediate solution is prepared by diluting 20ul of stock standard and 100ul of surrogate compound into a 10 ml volumetric flask. Dilute the solution to the volume with hexane and mix thoroughly. The final concentrations range from 100 µg/l –1,000 mg/l depending upon the compounds. Refer to Table 3 for details.

8.10.2 Store this solution in a refrigerator at 4°C for six months or before the manufacturer's expiration date, whichever is sooner.

8.11 Calibration Standards.

8.11.1 Initial Calibration Standards

8.11.1.1 Calibration standards should be made by quantitative dilutions of the above intermediate solution. Suggested levels and preparations are shown in Table3. All standards should be brought to the final volume with hexane.

8.11.1.2 A calibration range with standards made with 100ul, 200ul, 300ul, 400ul, 500ul, 600ul and 1,000ul of stock standard solution mix must be constructed for each surrogate compound. Accordingly, add 100ul of 100ug/ml Intermediate surrogate solution to the calibration solution to define a range equivalent to the target compounds (Table5).

8.11.2 Continuing Calibration Verification (Second Source Check Standard).

8.11.2.1 Two standards near the mid-point of the calibration range are prepared. The

ICV is prepared with 300ul of intermediate and the CCV is prepared with 400ul of intermediate standard. During the analysis, these two solutions are run alternatively to check the initial calibration.

8.11.2.2 Herbicide methyl esters -Varied concentration (11 compound mix) certified derivatized solutions #92510, different lot #, commercially purchased from Absolute (or equivalent).

8.11.3 The calibration solution must be replaced after six months, or sooner before manufacturer's expiration date or if comparison with check standards indicates a problem.

8.11.4 Prepare a second source calibration check standard following Table 3 by using the above intermediate solution.

8.12 Blank Spike (LCS) Standard.

8.12.1 Prepare an intermediate solution of the herbicide mixture- Herbicide Free Acid Standard- varied concentration (11 compound mix) purchased from Absolute (or equivalent).

8.12.2 Prepare following Table 4 by using the stock standard from 8.12.1 and diluting 40ul of stock into 50ml of Methanol. This is a varied concentration intermediate solution.

8.13 Surrogate standard.

8.13.1 Spike each sample, standard, and blank with the surrogate solution, 100 ug/ml- 2,4-Dichlorophenylacetic acid (DCAA),# M-8150B-SS, purchased from Accustandard (or equivalent) to encompass the range of the temperature program used in this method.

8.13.2 100ul of the 100ug/ml surrogate solution from 8.13.1 is diluted to 10mls with hexane to give a intermediate solution of 1000ppb. It is used for samples and QC samples.

8.13.3 See table 5 for the amount of intermediate surrogate spike used for calibration.

8.13.4 1ml of surrogate spiking solution is added to samples and QC, prior to extraction, this results in a varied final concentration in the extract of 1 mg/L or 1 mg/kg based on 30 g dry weight.

8.14 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Standard.

8.14.1 Prepare an intermediate solution of the herbicide mixture from 8.12.

8.14.2 1ml of spiking solution is added to randomly selected MS/MSD samples, prior to extraction, this results in a varied final concentration in the extract of 1 mg/L or 1 mg/kg based on 30 g dry weight.

8.15 pH Adjustment Solutions

8.15.1 Sodium hydroxide, NaOH, 6 N.

8.15.2 Sulfuric acid, H_2SO_4, 12 N.

8.16 Sodium Chloride (granular, anhydrous) NaCl. Purify by heating at 400°C for 4 hours in a shallow tray. Store the solid at 130°C.

9.0 Interferences

9.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

9.1.1 Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used in it. Detergent washing with hot water and rinse with tap water and then with organic-free reagent water is the next step. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used.

9.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

9.2 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from waste to waste, depending upon the nature and diversity of the waste being sampled.

9.3 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure. The determination using pentafluorobenzylation is more sensitive and more prone to interferences from the presence of organic acids or phenols than by methylation.

9.4 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids.

9.5 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

9.6 Sample extracts should be dry prior to methylation or else poor recoveries will be obtained.

10.0 Procedure

10.1 Extraction and hydrolysis of high concentration waste samples

10.1.1 Follow Method 3580, Waste Dilution, with the following exceptions:

10.1.1.1 Use diethyl ether as the dilution solvent.

10.1.1.2 Use acidified anhydrous sodium sulfate and acidified glass wool.

10.1.1.3 Spike the sample with surrogate(s) according to Sec. 8.13.

10.1.2 If the sample is to be analyzed for both herbicide esters and acids, then the sample extract must be hydrolyzed. In this case, transfer 1.0 mL (a smaller

volume or a dilution may be required if herbicide concentrations are large) to a 250-mL Erlenmeyer flask with a ground-glass joint at the neck. Proceed to Sec. 10.2. If the analysis is for acid herbicides only, proceed to Sec. 10.7 for derivatization by diazomethane.

10.2 Extraction and hydrolysis of soil, sediment, and other solid samples by Ultrasonic extraction.

10.2.1 Add 30 g (dry weight) of the well-mixed solid sample to a 400-mL thick-wall beaker. Adjust the pH to 2 or less with concentrated hydrochloric acid using 1 ml at a time using a dispenser, thoroughly mix the contents with a glass-stirring rod. Check the pH using a disposable glass pipette and taking a small aliqout of sample and placing it on the pH strip. Add more HCl if necessary and record the pH in the logbook.

10.2.2 Weigh out 30 g of anhydrous sodium sulfate into each of two, clean beakers to be used as the method blank and spike blank. A randomly selected sample should be weighed in triplicate for the batch matrix spike and matrix spike duplicate. Spike the spike blank, matrix spike and matrix spike duplicate with herbicide spiking solution. Add 1.0 ml of the surrogate solution (Sec. 8.13) to all samples, spiked samples, QC samples, and blanks.

10.2.3 Each day, prior to use, the sonicator must be tuned per manufacturer's instructions. In order for the ultrasonic extractor to efficiently extract solid samples, the sample must be free flowing when the solvent is added. Acidified anhydrous sodium sulfate should be added to soils (normally 1:1), until a free flowing mixture is obtained.

10.2.4 Add 100 mL of methylene chloride/acetone (1:1 v/v) to the beaker. Filter the samples through filter paper (Whatman #1 or equivalent) containing 7-10cm of acidified Na_2SO_4 into a K-D or 500 Erlenmeyer flask.

10.2.5 Ultrasonically extract the sample twice more using 100 mL of methylene chloride and the same ultrasonic conditions.

10.2.6 Combine the three organic extracts from the sample. Filter the combined extract through filter paper (Whatman #1, or equivalent) containing 7-10 cm of acidified sodium sulfate into a 500-mL Erlenmeyer flask. Add 10 g of acidified anhydrous sodium sulfate to the to the flask. Periodically, vigorously shake the extract and drying agent and allow the drying agent to remain in contact with the extract for a minimum of 2 hours.

10.2.7 Quantitatively transfer the contents of the flask to a 500-mL K-D flask with a 10-mL concentrator tube attached. Add 1-2 boiling chips and attach the macro Snyder column. Evaporate the extract on the water bath at a temperature of 60-65°C to a volume of approximately 5mL. Remove the flasks from the water bath and allow them to cool to room temperature.

10.2.4 If hydrolysis or additional cleanup is not required and the sample is dry, proceed to Section 10.6.3. Otherwise, proceed to section 10.3 for hydrolysis or Sec. 10.4 for cleanup.

10.3 Hydrolysis of soil, sediment, or other solid sample extracts.

<u>Use this step only if herbicide esters in addition to herbicide acids are to be determined.</u>

10.3.1 Add 5 mL of 37% KOH, and then add 30 mL of DI water to the extract. Reflux the mixture on a water bath at 60 - 65°C until the hydrolysis step is completed (usually 1 - 2 hours). Remove the flasks from the water bath and cool to room temperature.

CAUTION: The presence of residual acetone will result in the formation of aldol condensation products, which will cause GC interference.

10.3.2 Transfer the 35mls of hydrolyzed aqueous solution to a 250-mL separatory funnel and extract the solution two times with 100-mL portions of methylene chloride. Discard the methylene chloride phase into the chlorinated waste container. At this point, the basic (aqueous) solution contains the herbicide salts.

10.3.3 Adjust the pH of the solution to <2 with cold (4°C) sulfuric acid (1:3) and extract once with 40 mL of diethyl ether and twice with 20-mL portions of ether. Combine the extracts and pour them through a pre-rinsed drying column containing 7 to 10 cm of acidified anhydrous sodium sulfate. Collect the dried extracts in a 500-mL Erlenmeyer flask (with a 24/40 joint) containing 10 gm of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract and drying agent and allow the drying agent to remain in contact with the extract for a minimum of 2 hours.

10.3.4 Carefully transfer the contents of the flask to a 500-mL Kuderna-Danish flask with a 10-mL concentrator tube attached.

10.3.5 Proceed to Section 10.6 for the extract concentration. If additional cleanup is required, proceed to Sec. 10.4.

10.4 Cleanup of non-hydrolyzed herbicides.

Use this step if additional cleanup of the non-hydrolyzed herbicides is required.

10.4.1 Partition the herbicides by extracting the methylene chloride from 10.2.3 (or diethyl ether) three times with 15-mL portions of aqueous base prepared by carefully mixing a 2:1 ratio of reagent water to 37% aqueous potassium hydroxide. Discard the methylene chloride or ether phase. At this point the basic (aqueous) solution contains the herbicide salts.

10.4.2 Adjust the pH of the solution to <2 with cold (4°C) sulfuric acid (1:3) and extract once with 40 mL of diethyl ether and repeat twice with 40-mL portions of ether. Combine the extracts and pour them through a pre-rinsed drying column containing 7-10 cm of acidified anhydrous sodium sulfate. Collect the dried extracts in a 500-mL Erlenmeyer flask (with a 24/40 joint) containing 10 g of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract and drying agent and allow the drying agent to remain in contact with the extract for a minimum of 2 hours.

10.4.3 Carefully transfer the contents of the flask to a 500-mL Kuderna-Danish flask with a 10-mL concentrator tube attached.

10.4.4 For TCLP soils: Use 100ml of the leachate for the sample volume. Add surrogates and spikes to the required samples, blank and QC samples as noted in Section 8.

10.4.5 Proceed to Sec. 10.6 for extract concentration.

10.5 Preparation of aqueous samples.

10.5.1 Draw line at meniscus of water sample in the 1-L sample container for future measure. Transfer a 1-L sample aliquot to a 2-L separatory funnel.

10.5.2 Measure two 1-L aliquots of deionized water for a method blank and spike blank into separate 2-L separatory funnels. A randomly selected sample should be selected for the batch matrix spike and matrix spike duplicate. Measure three 1-L aliquots of this sample into separate 2-L separatory funnels. Spike the spike blank, matrix spike and matrix spike duplicate with herbicide spiking solution. Add 1.0 ml of the surrogate solution (Sec. 8.13) to all samples, spiked samples, QC samples, and blanks.

10.5.3 Add 250 g of NaCl to the sample, seal, and shake to dissolve the salt.

10.5.4 <u>Use this step only if herbicide esters, in addition to herbicide acids, are to be determined.</u>

10.5.4.1 Add 17 mL of 6 N NaOH to the sample, seal, and shake. Check the pH of the sample with pH paper. If the sample does not have a pH greater than or equal to 12, adjust the pH by adding more 6 N NaOH. Let the sample sit at room temperature until the hydrolysis step is completed (usually 1 - 2 hours), shaking the separatory funnel and contents periodically.

10.5.4.2 Add 60 mL of methylene chloride to the sample bottle and rinse both the bottle and the graduated cylinder. Transfer the methylene chloride to the separatory funnel and extract the sample by vigorously shaking the funnel for 2 minutes, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for 5 - 10 minutes. Discard the methylene chloride phase.

Note: If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods.

10.5.4.3 Add a second 60-mL volume of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, discarding the methylene chloride layer into the chlorinated waste bottle. Perform a third extraction in the same manner.

10.5.5 Add 17 mL of cold (4°C) 12 N sulfuric acid to the sample (or hydrolyzed sample), seal, and shake to mix. Check the pH of the sample by using a glass pipette to obtain a small aliquot of sample and place it on the pH paper. If the sample does not have a pH $\leq$ 2, adjust the pH by adding more acid.

10.5.6 Add 120mL diethyl ether to the sample, seal, and extract the sample by vigorously shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. Remove the aqueous phase from separatory funnel into original sample bottle and collect the ether phase in a 500-mL Erlenmeyer flask containing approximately 10 g of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract and drying agent.

10.5.7 Return the aqueous phase to the separatory funnel, add 60 mL of diethyl ether to the sample, and repeat the extraction procedure a second time, combining the extracts in the 500-mL Erlenmeyer flask. Perform a third extraction with 60mL diethyl ether in

the same manner. Allow the extract to remain in contact with the sodium sulfate for approximately 2 hours.

NOTE: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

10.5.8 Filter the dried extract through a funnel plugged with acid washed glass wool, and collect the extract in the K-D concentrator. Proceed to Sec. 10.6 for extract concentration.

10.5.9 For liquid TCLP samples, use 100ml for sample volume. Add surrogates and spikes to the required samples, blank and QC samples as noted in Section 8. Proceed to Section 10.5.2 to 10.5.8.

10.6 Extract concentration

10.6.1 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of diethyl ether to the top of the column. Attach the solvent vapor recovery glassware (condenser and collection device) to the Snyder column of the K-D apparatus following manufacturer's instructions. Place the K-D apparatus on a hot water bath at 50-55°C so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10 - 20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

10.6.2 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of diethyl ether. If more than 2.5ml of extract remains, the extract may be further concentrated by using nitrogen blowdown technique (Sec. 10.6.3).

10.6.3 Nitrogen blowdown

10.6.3.1 Place the concentrator tube in a warm water bath (approximately 35°C) and evaporate the solvent volume to 2.5ml using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon). Remove the boiling chips from the concentrator tube.

CAUTION: Do not use plasticized tubing between the carbon trap and the sample.

10.6.3.2 The internal wall of the tube must be rinsed down several times with diethyl ether during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). Under normal operating conditions, the extract should not be allowed to become dry.

10.6.4 Dilute the extract with 1 mL of isooctane and 0.5 mL of methanol. Dilute to a final volume of 4 mL with diethyl ether. The sample is now ready for methylation with diazomethane.

10.7 Esterification - Diazomethane Derivatization.

10.7.1 Bubbler method - Assemble the diazomethane bubbler (see Figure 1).

CAUTION: Diazomethane is a carcinogen and can explode under certain conditions.

10.7.2 The diazomethane derivatization procedures described below will react efficiently with all of the chlorinated herbicides described in this method and should be used only by experienced analysts, due to the potential hazards associated with its use.

The following precautions should be taken:

* Use a safety screen.
* Use mechanical pipetting aides.
* Do not heat above 90 C - EXPLOSION may result.
* Avoid grinding surfaces, ground-glass joints, sleeve bearings, and glass stirrers EXPLOSION may result.
* Store away from alkali metals - EXPLOSION may result.
* Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

10.7.3 Add 5 mL of diethyl ether to the first test tube. Add 1 mL of diethyl ether, 1 mL of carbitol, 1.5 mL of 37% KOH, and 0.1 - 0.2 g of Diazald to the second test tube. Immediately place the exit tube into the concentrator tube containing the sample extract. Apply nitrogen flow (10-12 mL/min), do not exceed 12mL/min, to bubble diazomethane through the extract for 5 minutes, 10 minutes or more for TCLP, or until the yellow color of diazomethane persists. There will not be a color change for TCLP soil extracts, but make sure the extract is bubbled for 5 minutes. The amount of Diazald used is sufficient for esterification of approximately three sample extracts. An additional 0.1 - 0.2 g of Diazald may be added (after the initial Diazald is consumed) to extend the generation of the diazomethane. There is sufficient KOH present in the original solution to perform a maximum of approximately 20 minutes of total esterification.

10.7.4 Remove the concentrator tube and seal it with a Neoprene or PTFE stopper. Store at room temperature in a hood for 20 minutes.

10.7.5 Destroy any unreacted diazomethane by adding 0.1 - 0.2 g of silicic acid to the concentrator tube. Allow to stand until the evolution of nitrogen gas has stopped. Transfer the sample into a concentration tube or 25ml vial and evaporate the solvent to 1ml. Adjust the sample volume to 10.0 mL with hexane and store refrigerated if further processing will not be performed immediately. Analyze by gas chromatography.

11.0 Calibration

11.1 Initial Calibration.

11.1.1 The calibration range covered for this analysis employs standards in Table 3. The method reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Lower concentration standard may be needed to meet the reporting limit

requirements of state specific regulatory program. The linear range covered by this calibration is the highest concentration standard.

11.1.1.1 The total number of calibration standards prepared must meet the minimum of five for linear, 6 for second order, and 7 for third order functions.

11.1.1.2 The range of calibration may be narrowed and the calibration points at the lowest and/or highest levels are not included in the least squares regression.

11.1.1.3 If the lowest standard is eliminated, then the RL is increased.

11.1.1.4 If the highest standard is eliminated, then the sample concentration must fall within the calibration range.

11.1.2 A calibration range must be constructed for the surrogate compound. Accordingly, add appropriate amounts of 2,4-Dichlorophenylacetic acid (DCAA) to the calibration solution to define a range similar to the target compounds. See Table 5.

11.1.3 Program the GC system and autosampler to the GC conditions detailed in Table 1. Load the calibration standards into the auto sampler for analysis and initiate the calibration run.

11.1.4 Each analyte is quantitatively determined using the external standard technique. The calibration factor (CF) is defined in Section 15.1.

11.1.5 For the initial calibration to be valid, the RSD of the mean calibration factors must be less than or equal to 20% over the calibration range. If any analyte exceeds the 20% acceptance limit for a given calibration, corrective action must be taken.

11.1.5.1 If the problem is associated with a single standard, reanalyze that standard and recalculate the RSD.

11.1.5.2 Alternatively, narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range.

11.1.5.2.1 The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

11.2 Second Source Calibration Check Standard.

11.2.1 The calibration is verified with a second source calibration check standard at mid-level concentration from an independent source (Section 8.12). It must be performed right after the initial calibration.

11.2.2 The percent difference (% D) (Section 15.3) for all the target compounds must meet the %D criteria of 15% used for calibration verification.

11.2.2.1 If % D is greater than 15%, reanalyze the second source check. If the criteria cannot be met upon re-injection, re-prepare the second source solution using a fresh ampoule and repeat the process.

11.2.2.2 If the %D criteria cannot be achieved after re-preparation of the second source, Make fresh calibration standards and re-calibrate.

11.3 Continuing Calibration Verifications.

11.3.1 Continuing calibration verification standards (Section 8.11.2) must be acquired every 12-hour or sooner and at the end of the analysis sequence. Two different level standards are alternated to serve as a further check on the initial calibration.

11.3.2 This check standard must be within 15% of the calibration response factor from the initial calibration. Calculate the percent difference for calibration verification of linear calibrations by evaluating the instrument response difference between the initial calibration and each subsequent analysis of the verification standard. Calculate the percent difference (%D) using the following formula in Section 15.3.

11.3.3 Each sample analysis must be bracketed with acceptable calibration verification standards (each 12-hour analytical shift). More frequent analyses of standards will minimize the number of sample extracts that would have to be reinjected if the QC limits are violated. If %D criteria fails during a mid sequence calibration check or at the end of the analysis sequence, a continuing calibration check is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed. In situations where the first check fails to meet the criteria, the instrument logbook should have clear documented notations as to what the problem was and what corrective action was implemented to enable the second check to pass.

11.3.4 When a calibration verification standard fails to meet the QC criteria at the end of the analysis sequence, all samples injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations, and re-injection of the sample extracts may be required.

11.3.4.1 The analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed when the calibration standard response is above the initial calibration response.

11.3.4.2 The analyte was detected in the specific samples analyzed during the analytical shift, or the calibration standard response is below the initial calibration response, then the extracts for those samples need to be reanalyzed.

11.3.5 Each subsequent injection of a continuing calibration standard during the 12-hour analytical shift must be checked against the retention time windows established in Section 12.0. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

12.0 Retention Time Windows

12.1 The absolute retention time windows must be calculated for each analyte and surrogate on each GC column and instrument. New retention time windows must be established whenever a new chromatographic column is installed, or when there are significant changes in the operating conditions. The retention time windows must be reported with the analysis results in support of the

identifications made. See Table 6 for Retention Times (Minutes) of Methyl Derivatives of Chlorinated Herbicides.

12.2 Employ the following approach to establish retention time windows.

12.2.1 Make three injections of all standard mixtures over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

12.2.2 Record the retention time for each analyte and surrogate to three decimal places (e.g., 0.007). Calculate the mean and standard deviation of the three absolute retention times for each analyte and surrogate.

12.2.3 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

12.2.4 The width of the retention time window for each analyte and surrogate is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation is employed, the width of the window will be 0.03 minutes.

12.2.5 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

12.3 The surrogates retention times in the calibration standards are useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window, the analyst must determine the cause and correct the problem before continuing analyses.

13.0 Procedure

13.0 Gas Chromatograph Conditions.

13.1.1 Establish instrument conditions following the specifications detailed in Table 1. Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification should be approved by team leader/manager.

13.2 Initial Calibration.

13.2.1 Refer to Section 11.1.

13.3 Second Source Calibration Check.

13.3.1 Refer to Section 11.2.

13.4 Continuing Calibration Verifications.

13.4.1 Refer to Section 11.3.

13.5 Sample Extracts Analysis (Primary).

13.5.1 All samples and quality control samples are injected into the Gas Chromatograph using the autosampler. Program the sampler for an appropriate number of syringe rinses and a 2µl injection size. A splitless injection technology is used.

13.5.2 Sample concentrations are calculated by comparing sample responses with the initial calibration of the system (Section 15.4). If sample response exceeds the limits of the initial calibration range, dilute the extract and reanalyze. Extracts should be diluted so that all peaks are on scale, as overlapping peaks are not always evident when peaks are off scale.

13.5.3 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the sample meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

13.5.4 If chromatographic peaks are masked by the presence of interferences, further sample cleanup is necessary. Refer to the sample preparation steps for extract cleanup alternatives.

13.5.4.1 If extract cleanup is required, all QC samples must also be processed through the cleanup method.

13.6 Confirmation Analysis.

13.6.1 Confirmation analysis is to confirm the presence of all compounds tentatively identified in the primary analysis. The primary and confirmation (secondary) analysis is conducted simultaneously.

13.6.1.1 All instrument performance quality control criteria for calibration and retention times must be satisfied on the confirmation column.

13.6.2 Each tentative identification must be confirmed using either a second GC column of dissimilar stationary phase or using anther technique such as GC/MS.

13.6.2.1 GC/MS confirmation may be used in conjunction with dual-column analysis if the concentration is sufficient for detection in GC/MS. Refer to Standard Operating Procedure for Method SW846 8270 for the appropriate GC/MS operating conditions and analysis procedures.

13.6.2.1.1 GC/MS will normally require a concentration of approximately 10 ng/µl in the final extract for each compound.

13.7 Sample Dilution.

13.7.1 Establish dilution of sample in order to fall within calibration range or to minimize the matrix interference.

- Utilize screen data (specific project only).
- Utilize acquired sample data.
- Utilize the history program or approval from client/project.
- Sample characteristics (appearance, odor).

13.7.2 If no more lower dilution been reported, the dilution factor chosen should keep the response of the largest peak for a target compound in the upper half of the initial calibration range of the instrument.

13.7.3 Preparing Dilutions.

13.7.3.1 Prepare sample dilutions quantitatively. Dilute the sample extract with hexane using logical volume to volume ratios, i.e., 1:5, 1:10, 1:50, etc.

13.7.3.2 Transfer an appropriate amount of extract to a volumetric flask. Fill the flask to the volume mark with hexane. Gently shake to disperse the extract throughout the solvent.

13.8 Data interpretation.

13.8.1 Qualitative identification.

13.8.1.1 The compounds shall be identified by analysts with competent knowledge interpreting retention time and/or chromatographic pattern by comparison of the sample to the standard of the suspected compound. The criteria required for a positive identification are:

13.8.1.1.1 The sample component must elute at the daily absolute retention time window for both primary and confirmation column.

13.8.1.1.2 Be aware of matrix interfering effects on peak shape and relative peak ratios which could distort the pattern. Interpretation of this phenomenon may require a highly experienced chromatographer or at least a second opinion.

13.8.2 Quantitative analysis.

13.8.2.1 When a target compound has been identified, concentration (see section 15.4) will be using the integrated area of the sample peak or, as an alternative, the sample peak height and calculated by external standard technique.

13.8.2.2 When sample results are confirmed using two dissimilar columns or with two dissimilar detectors, the agreement between the quantitative results must be evaluated after the identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula in Section 15.6.

13.8.2.2.1 A program to perform the RPD calculation had been developed and incorporated into ENVIROQUANT software. Whenever an analyte has a RPD exceeds 40% between the results on the primary and secondary columns, a “P” qualifier will be shown on the quantitation report next to its results.

13.8.2.2.2 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration.

13.8.2.2.3 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the result from the primary column with the footnote (remark) indicating “More than 40% RPD for detected concentrations between two GC

columns".

14.0 Quality Assurance

14.1 Control parameters have been established for critical steps within this method that enable the analyst to assess method performance and conduct corrective action for defective processes before, during and after the analysis of samples. The analyst must exercise his/her responsibilities to take corrective action if any of the following controls are not within established specifications.

Initial Calibration	Whenever needed
Second Source Calibration Check Standard	Following initial calibration
Continuing Calibration Verifications	Every 12 hours
Method Blank	One per batch*
Blank Spike	One per batch*
Matrix Spike	One per batch*
Matrix Spike Duplicate	One per batch*
Surrogate	Every sample and standard

*The maximum number of samples per extraction batch is twenty or per project specification.

14.2 Initial Calibration.

14.2.1 Refer to Section 11.1.

14.3 Second Source Calibration Check Standard.

14.3.1 Refer to Section 11.2.

14.4 Continuing Calibration Verifications.

14.4.1 Refer to Section 11.3.

14.5 Method Blank.

14.5.1 The method blank is either DI water or sodium sulfate (depending upon the sample matrix) which must be extracted and analyzed with each set of 20 or less samples. The blanks are processed through all steps required for sample preparation and measurement.

14.5.2 Corrective action for non-compliant blanks must be taken if contamination is detected that interferes with detection of targeted analytes. If the method blank contains a target analyte above its MDL, the entire batch must be re-extracted and re-analyzed. Exceptions can be evaluated based on client specifications. These will be documented in the case narrative. Corrective action is unnecessary unless detected contamination has a negative effect on sample data.

14.5.3 Surrogate compounds are added to all method blanks prior to extraction and analysis. If the surrogate accuracy in the blank does not meet criteria, the batch must be re-extracted and re-analyzed. If the surrogate recovery is biased high and the associated samples are non-detect, then the data is acceptable. If the surrogate recovery is biased low, the batch must be re-extracted and re-analyzed.

14.6 Blank Spike.

14.6.1 A blank spike must be extracted and analyzed with each set of 20 or less samples. The

blank spike consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. It is spiked with the target analytes at a detectable concentration, usually the mid-point concentration of the calibration range.

14.6.2 The blank spike recoveries are assessed using in house limits which are established using the procedure documented in SOP TQA043. If a blank spike is out of control, the following corrective actions must be taken and all the associated samples must be re-extracted and reanalyzed. The exception is if the blank spike recovery is high and no hits reported in associated samples and QC batch. In that case, the sample results can be reported with footnote (remark) and no further action is required.

14.6.2.1 Check to be sure that there are no errors in the calculations, or spike solutions. If errors are found, recalculate the data accordingly.

14.6.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample batch.

14.6.2.3 If no problem is found, re-extract and reanalyze the sample batch.

14.6.3 When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the BS results are used to verify that the laboratory can perform the analysis in a clean matrix.

14.7 Matrix Spike/Matrix Spike Duplicate.

14.7.1 For every batch of 20 or less samples, a pair of matrix spike and matrix spike duplicate must be prepared and analyzed. Matrix spike and matrix spike duplicate are randomly selected replicate sample aliquots to which a surrogate compound and a known amount of matrix spike compounds are added. The MS and MSD are then extracted and run through any clean-up procedures along with the other samples in that batch to monitor the precision and accuracy of the extraction.

14.7.2 Assess the matrix spike recoveries and relative percent deviation (RPD) against the in-house control limits established using the procedure in SOP TQA043.

14.7.3 If the matrix spike accuracy of any individual compound is out of control, the accuracy for the compound in the blank spike must be within control. Matrix interference is assumed and the data is reportable. No further corrective action is required.

14.8 Surrogate.

14.8.1 2,4-Dichlorophenylacetic acid (DCAA) is used as a surrogate standard to monitor the efficiency of the extraction and clean-up procedures. 1.0ml of surrogate standard is added to each sample including the QCs prior to extraction. The surrogate recovery data from individual samples must be evaluated versus the surrogate control limits developed by the laboratory using procedures found in SOP TQA043.

14.8.2 If surrogate recovery is not within established control limits, corrective action must be performed if surrogate recoveries indicate that a procedural error may have occurred during the analysis of the sample. If recoveries are biased high, and samples are non-detect, the samples may be reported. If recoveries are biased low, the batch is re-extracted and re-analyzed.

14.8.2.1 Check the surrogate calculations for calculation or integration errors and perform corrections if detected.

14.8.2.2 Re-analyze the extract if calculation errors are not detected or if recoveries are biased low. If the surrogate recoveries for the re-analyzed extract are in control, report data from the re-analysis only.

14.8.2.3 If data from the reanalysis is also out of control, re-extract and reanalyze the sample.

14.8.2.4 If, upon reanalysis, the surrogate recoveries are acceptable, report the reanalysis data. If the recovery is again not within limits, the problem is considered to be matrix interference. Submit both data sets with the original analysis being reported.

14.8.3 The retention time shift for surrogate must be evaluated after the analysis of each sample. The sample must be reanalyzed when the retention time of any surrogate compound is outside the retention window.

14.8.3.1 Reanalysis may not be required for samples having visible matrix interference, defined as excessive signal levels from target or non-target interfering peaks. Check with the team leader or supervisor before making any decisions.

15.0 Calculations

15.1 Calculation Factor (CF).

$$CF = \frac{A_s}{C_s}$$

where:
A_s = Area of the peak for the compound being measured.
C_s = Concentration of the compound being measured (µg/l).

15.2 Percent Relative Standard Deviation (% RSD).

$$\%RSD = \frac{SD}{CF_{av}} \times 100$$

where:
SD = Standard Deviation.
CF_{av} = Average calibration factor from initial calibration.

15.3 Percent Difference (% D).

$$\% D = \frac{|CF_{av} - CF_c|}{CF_{av}} \times 100$$

where:
CF_c = CF from continuing calibration (CBCHK).

15.4 Concentration (Conc.).

15.4.1 <u>For water:</u>

$$\text{Conc. } (\mu g/l) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{V_i}$$

15.4.2 For soil/sediment (on a dry weight basis):

$$\text{Conc. } (\mu g/kg) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{W_s \times S}$$

where:
A_c = Area of peak for compound being measured.
V_f = Final Volume of total extract (ml).
D = Secondary dilution factor.
V_i = Initial volume of water extracted (ml).
W_s = Weight of sample extracted (g).
S = (100 - % moisture in sample) / 100 or % solid/100.
M = Multiplier

15.5 Percent Recovery (% R).

$$\% R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

15.6 Relative Percent Difference (RPD).

$$RPD = \frac{|C_1 - C_2|}{(1/2)(C_1 + C_2)} \times 100$$

where:
C_1 = Matrix Spike Concentration or the result on column 1.
C_2 = Matrix Spike Duplicate Concentration or the result on column 2.

16.0 Documentation

16.1 Record the analytical sequence in the instrument logbook prior to initiating the analysis.

16.1.1 Document reasons for reanalysis in the comments section of the log book.

16.2 Record all standards preparation data in the standards logbook. All columns must be completed and countersigned by a supervisor. Cross-reference the Accutest lot number on the standard vial.

16.3 The instrument maintenance logbook must be completed whenever maintenance is performed on the instrument. There is a separate logbook for each instrument.

16.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

16.5 Unused blocks of any form must be Z'ed by the analyst before submitting the data for review.

16.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance

of the logbooks and the accuracy of the recorded information should also be verified during this review.

17.0 Data Review & Reporting

17.1 Initial and continuing calibration check. Verify that all calibration and continuing calibration criteria have been achieved. If the criteria had not been achieved, corrective action must be performed to bring the system in control before analyzing any samples.

17.1.1 If samples had been analyzed under non-compliant calibration criteria, all sample extracts must be re-analyzed once the system is brought into control.

17.2 Quality Control Data Review. Review all QC data. If QC criteria were not achieved, perform corrective action according to Section 14.0 before proceeding with analysis.

17.2.1 In some situation, corrective action may demand that the entire sample batch be re-extracted and re-analyzed before processing data.

17.3 Chromatogram Review. The chromatogram of each sample is evaluated for target compounds.

17.3.1 Check specific retention time windows for each target compound for the presence of the target compound in each chromatogram.

17.3.1.1Each sample may require the reporting of different target compounds. Review the login to assure that the correct target compounds are identified.

17.3.2 The compound must be identified on the primary and confirmatory column before assigning a qualitative identification.

17.3.3 Manual integration of chromatographic peaks must be identified by the analysts by initialing and dating the changes made to the report.

17.4 Transfer to LIMS. Following the initial screen review, transfer the processed data to the LIMS.

17.4.1 Print the processed data and compare the printed values to the original values to verify transfer accuracy.

17.4.2 If transfer errors occurred, the error s must be corrected before the data is re-submitted.

17.5 Hardcopy Print & Data Package Assembly.

17.5.1 After successful transfer is verified, approve the data and print a hard copy.

17.5.2 Assemble the data package combining the LIMS output and instrumental data.

17.5.3 Pass the entire package forward to the supervisor for final review and release approval.

18.0 Pollution Prevention & Waste Management

18.1 Pollution Prevention. Users of this method must perform all procedural steps in a manner that

controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 18.2.

18.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, TSM003. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

18.2.1 Non hazardous aqueous wastes.

18.2.2 Hazardous aqueous wastes.

18.2.3 Chlorinated organic solvents.

18.2.4 Non-chlorinated organic solvents.

18.2.5 Hazardous solid wastes.

18.2.6 Non-hazardous solid wastes.

19.0 References

19.1 Test Methods for Evaluating Solid Wastes, Physical Chemical Methods (SW846) Third Edition All Updates, January 1998.

19.2 Code of Federal Regulations (CFR) 40, Parts 136 - Revised as of July 1, 1995, Appendix B-Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11.

Table 1. Operating Conditions.

Temperature program	60°C to 280°C, at 4°C/min
Helium carrier flow	30 cm/sec
Injection volume	2 uL, splitless, 45 sec delay
Injector temperature	200°C
Detector temperature	300°C

Table 2 Stock Standard Concentration.
Commercially purchase prepared Herbicide Methyl Esters custom mixture from Absolute Standards (or equivalent).

Compound Name	Stock ug/ml
2,4-D	500
2,4-DB	1000
Dalapon	500
Dicamba	100
Dichloroprop	500
Dinoseb	100
4-Nitrophenol	500
Picloram	100
2,4,5-TP (Silvex)	100

2,4,5-T	100
Pentachlorophenol	25

Table 3 Preparation of Intermediate Calibration Solution. Dilute 20ul of Stock standard into 10mls of hexane in a 10-ml volumetric flask:

Standards are prepared using the following Table.

Compound Name	Stock ug/ml	Concentration ug/ml						
		Std 1	Std 2	Std 3	Std 4	Std 5	Std 6	Std 7
2,4-D	500	100	200	300	400	500	600	1000
2,4-DB	1000	200	400	600	800	1000	1200	2000
Dalapon	500	100	200	300	400	500	600	1000
Dicamba	100	20	40	60	80	100	120	200
Dichloroprop	500	100	200	300	400	500	600	1000
Dinoseb	100	20	40	60	80	100	120	200
4-Nitrophenol	500	100	200	300	400	500	600	1000
Picloram	100	20	40	60	80	100	120	200
2,4,5-TP (Silvex)	100	20	40	60	80	100	120	200
2,4,5-T	100	20	40	60	80	100	120	200
Pentachlorophenol	25	5.0	10	15	20	25	30	50

Table 4 Preparation of Blank Spike (LCS) Solution. Dilute 40ul of Stock standard into 50mls of methanol in a 50-ml volumetric flask:

Compound Name	Stock ug/ml	Conc. ug/ml
Dalapon	2500	2000
4-Nitrophenol	2500	2000
Dicamba	500	400
Dichloroprop	2500	2000
2,4-D	2500	2000
Pentachlorophenol	125	100
2,4,5-TP (Silvex)	500	400
2,4,5-T	500	400
2,4-DB	5000	4000
Dinoseb	500	400
Picloram	500	400

Table 5 Preparation of Surrogate – 100ug/ml- 2,4-Dichlorophenylacetic Acid Calibration Solution.

Dilute 100ul of Stock surrogate standard into 10mls of hexane in a 10 ml volumetric flask:

Calibration Standard ug/ml	Volume Intermediate ul	Final Volume ml	Final Conc. ug/ml
STD 1- 100	100	1	100
STD 2- 200	200	1	200
STD 3- 300	300	1	300
STD 4- 400	400	1	400
STD 5- 500	500	1	500
STD 6- 600	600	1	600

STD 7- 1000	1000	1	1000

Table 6 - Retention Times (Minutes) Of Methyl Derivatives Of Chlorinated Herbicides		
	Narrow Bore Columns	
Analyte	**Primary[a] Column**	**Confirmation[a] Column**
Dalapon	3.4	4.7
4-Nitrophenol	18.6	20.5
DCAA (surrogate)	22	14.9
Dicamba	22.1	22.6
Dichloroprop	25	25.6
2,4-D	25.5	27
Pentachlorophenol	28.3	27
2,4,5-TP	29.7	29.5
2,4,5-T	30.5	30.9
2,4-DB	32.2	32.2
Dinoseb	32.4	34.1
Picloram	34.4	37.5
Primary Column: DB-5 Narrow Bore	*Confirmation Column: DB-1701 Narrow Bore*	

Figure 1 – Diazomethane Generator

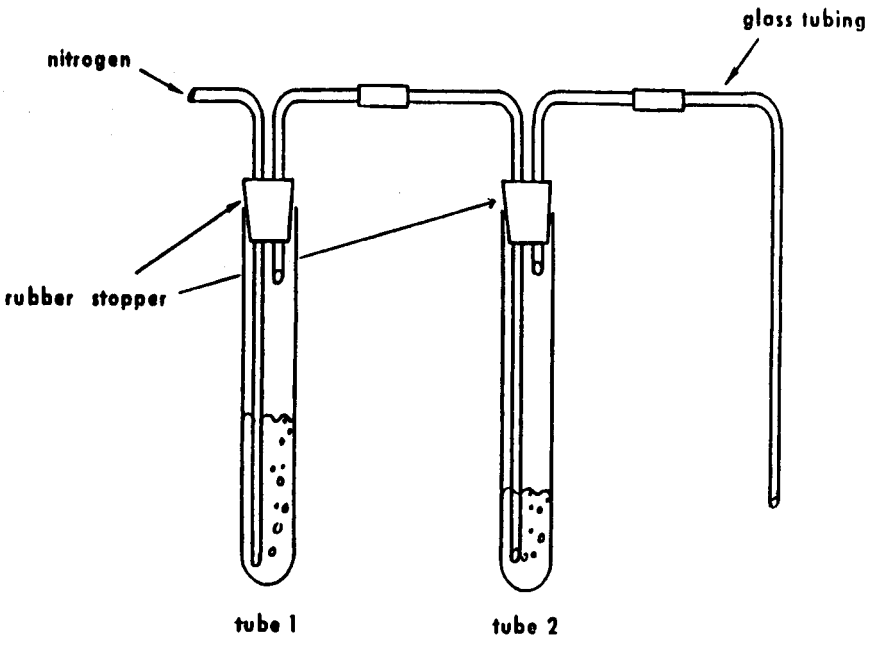
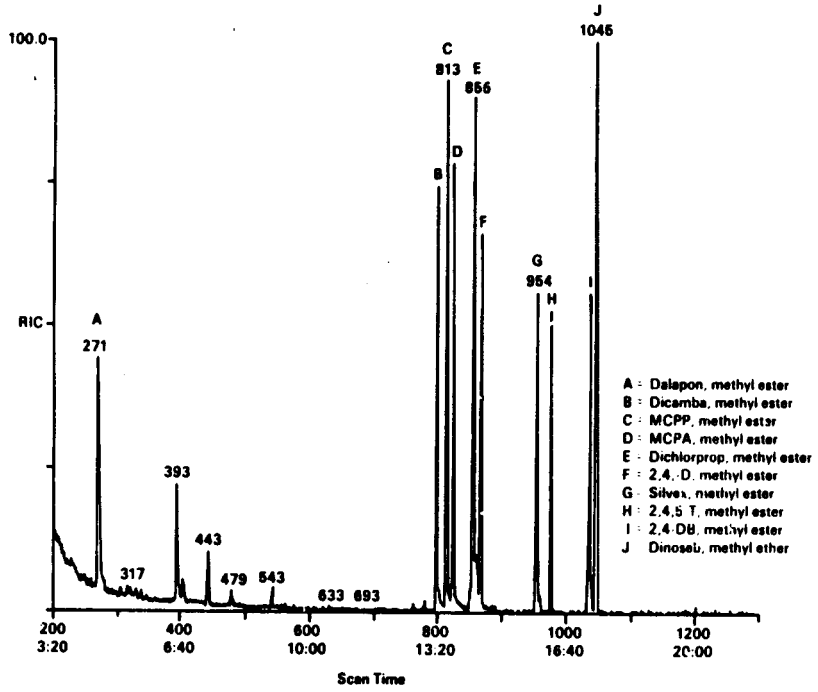


Figure 2 – Chromatagram of Methyl Esters of Chlorophenoxyacids



Micro Distillation of CYANIDE in Aqueous and Solid Matrices

Prepared by: Steve Lee Date: 06/21/06

Reviewed by: Kelliam Garcia Date: 06/21/06

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

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TITLE: Micro Distillation of CYANIDE in Aqueous and Solid Matrices

REFERENCES: EPA Method 335.2 M/ EPA 335.4/ SW846 9010, SW 846 9014

Revised Sections*: *new procedure

1.0 SCOPE AND APPLICATION

1.1 This method is applicable for the distillation of aqueous or liquid waste samples for cyanide. All samples must be taken through the distillation process before they can be analyzed for cyanide.

2.0 SUMMARY

2.1 Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid by reaction in a reflux system of a mineral acid in the presence of magnesium ion. The sample is distilled in a micro distillation unit. The sample is treated to remove interference from oxidizing agents, sulfides, and nitrites.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. See determinative method.

3.2 Method Detection Limit. MDLs should be established using a solution spiked at approximately 3 to 5 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year for frequently analyzed parameters.

4.0 DEFINITIONS

BATCH. A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using control limits of 90 to 110%.

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SPIKE BLANK SAMPLE. Digest and analyze a spiked blank with each batch of samples. This spiked blank must have a recovery of 85 to 115%. If the spike blank is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the spike blank recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag.

MATRIX. The component or substrate (e.g., air, water, or soil) which contains the analyte of interest.

MATRIX SPIKE. The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} \times 100 = \text{Matrix Spike Recovery}$$

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLs). MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT GRADE. Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents, which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER. Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

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REFERENCE MATERIAL. A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 Cyanide is very toxic. Samples should be kept basic until time of distillation to prevent the formation of HCN. Make sure that the distillation flasks are tightly closed and leak free during the distillation process.

5.3 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses.

6.0 PRESERVATION AND HOLDING TIME

6.1 All samples should be preserved with NaOH to a pH of $\geq$12 and stored in a refrigerator at 4°C. Ascorbic acid should be added to prevent decomposition of the cyanides by oxidizing agents such as chlorine. Ascorbic acid is normally added during the field with the initial sample preservation. The pH for each sample should be checked before the distillation process is started.

6.2 All samples should be analyzed within 14 days of the date of collection.

7.0 APPARATUS

7.1 Distillation apparatus. Micro Distillation block, Lachat part number A17102/202 or equivalent.

7.2 MicroDistillation tubes and caps, Lachat part number A17117

7.3 Autopipetters, calibrated as per QA006.04.

7.4 Volumetric Pipets, class A.

7.5 Volumetric flasks, class A.

7.6 50 ml graduated cylinders.

7.7 Lead Acetate paper.

7.8 Wide range pH paper capable of measuring pH values from 0 to 14.

7.9 Analytical balance capable of accurately weighing to the nearest 0.0001g.

8.0 REAGENTS

All reagents should be made from ACS grade reagents unless otherwise noted. Deionized water should be used whenever water is needed as in all preparation of reagents, record the amounts used, the lot numbers of the reagents, the date made, the expiration date, storage requirements, and your initials in the reagent log book.

8.1 7.11 M sulfuric acid/ 0.79 M magnesium chloride: In the hood place a 500 ml beaker on a top loading balance with a 400g capacity or greater. Tare the balance; place 110.8g of DI water. Then add and dissolve 32.2g of magnesium chloride hexahydrate (MgCl2. 6H2O). Slowly add 139g of sulfuric acid (in small increments). The solution will become hot.

8.2 Sulfuric acid, concentrated.

8.3 Sulfamic acid (NH_2SO_3H).

8.4 1000 ppm CN Stock: Dissolve 2.51 g of KCN and 2 g KOH in 900 ml of DI water and then dilute to a final volume of 1 liter with DI water. Standardize with 0.0192 N $AgNO_3$ following the procedure outlined in the standardization section below. The cyanide stock should be standardized a minimum of once per week. Alternatively this standard can be purchased commercially.

8.4.1 If an external check solution is to be prepared use the same procedure as outlined in 8.5, but use a different lot of KCN.

8.5 0.100 ppm CN spiking solution. Add 10.0 uL of 1000 ppm stock solution to a 100 ml volumetric flask. Dilute to volume with DI water.

8.6 Intermediate Cyanide Solution, 10.0 mg/L. Pipet 1.0 ml of 1000 mg/l stock solution of CN into a 100 ml volumetric flask containing approximately 50 ml of .25 N NaOH. Dilute to a final volume with .25 N NaOH and mix well.

8.7 Cyanide Standard Solutions. Suggested standard concentrations are shown below. All standards are made up using the above intermediate solution (10.0 mg/L) as outlined in the table below. All the standards must be diluted to a final volume with .25 N NaOH. Alternate standard concentrations may be

used as long as all method requirements are met. **Standards should have a final concentration of 0.25 N NaOH.**

ml of 10.0 mg/L CN	Final volume	Concentration (mg/L)
5	100	0.5
3	100	0.3
2	100	0.2
1	100	0.1
0.5	100	0.05
0.1	100	0.01

8.8 Standard Silver Nitrate Solution, 0.0192 N. Crush approximately 5 g of silver nitrate crystals and dry to constant weight at 40°C. Weigh out 3.2647 g of dried silver nitrate into a 1liter flask containing approximately 800 ml of DI water. Dilute to a final volume of 1 liter with DI water and mix well.

8.9 Benzalrhodanine indicator. Dissolve 20 mg (0.02 gms) p-dimethylaminobenzalrhodanine in 100 ml acetone.

8.10 Sodium Hydroxide, 0.25 N. Add 10.0 g of sodium hydroxide to approximately 800 mL of DI water. Mix well and then dilute to a final volume of 1000 mL.

8.11 Sodium Hydroxide trapping solution, 0.95 N. Add 19.0 g of sodium hydroxide to approximately 400 ml of DI water. Mix well and then dilute to a final volume of 500 ml.

8.12 Cadmium Carbonate, powdered.

8.13 pH Adjusted Blank Solution. For method blanks and spike blanks, add 1.25 N to DI water to adjust the pH to $\geq$12.

9.0 INTERFERENCES

9.1 Most interferences are eliminated by the distillation procedure. Oxidizing agents such as chlorine are removed using the ascorbic acid preservative. Sulfides will distill over, but can be removed by adding cadmium carbonate to the sample. Nitrites can be an interference, but can be removed by adding sulfamic acid to the sample.

9.2 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10 mg/l were not found to interfere.

9.3 Fatty acids, detergents, surfactants, and other compounds may cause foaming during the distillation when they are present in high concentrations. This interference can be minimized by using a smaller sample size or by following a special extraction procedure. Check with the lab manager or

supervisor for additional information.

10.0 CYANIDE STANDARDIZATION PROCEDURE

10.1 If the cyanide stock solution has not been standardized within a week, then the stock must be standardized before proceeding further with the analysis. The standardization can be done following the procedure outlined below.

10.2 Volumetrically measure out 25.0 ml of the 1000 mg/l cyanide stock solution into a graduated plastic beaker. Add 10 to 12 drops of benzalrhodanine indicator to the solution. Place a stir bar in the beaker.

10.3 Fill a buret with 0.0192N silver nitrate solution. Titrate the sample with continuous stirring until the color changes from yellow to a brownish-pink. Approximately 25 ml of silver nitrate solution should be needed to reach this endpoint. Also analyze a method blank that has been brought to pH > 12 with KOH following this procedure.

10.4 Calculate the concentration of the stock cyanide solution using the equation shown below. If multiple standardizations of the stock are done at a given time, then the average of the standardizations should be used as the final stock concentration.

$$\text{CN, mg/l} = \frac{(A - B) \times 1000}{25 \text{ ml}}$$

Where: A = the volume of $AgNO_3$ used to titrate the sample.
B = the volume of $AgNO_3$ used to titrate the blank.

11.0 TEST FOR SULFIDES

Every sample to be distilled for cyanide should first be tested for sulfides as outlined below.

11.1 Put a drop of sample on a piece of Lead Acetate paper.

11.2 Check to see if there is darkening of the paper. The darkening indicates that sulfide is present.

11.3 If sulfide is present, take a 25-ml aliquot of the preserved sample and add powdered cadmium carbonate. Continue to add the cadmium carbonate until no more yellow precipitate is formed. Avoid a large excess of cadmium and a long contact time, which may cause the sample to lose cyanide.

11.4 Filter the sample through dry filter paper into a dry beaker to remove the yellow precipitate. Check the filtered sample with lead acetate paper to verify that no additional sulfide is present.

11.5 The filtered sample can now be distilled. Note that cadmium carbonate was added to the sample in the comments section of the distillation log. Document

the sulfide results on the distillation log.

12.0 CYANIDE DISTILLATION PROCEDURE - MICRO DISTILLATION

12.1 Set the controller temperature to 120 ºC. Allow the heater block to warm up. This will take about 40 minutes.

12.2 Each sample tube should be labeled with the sample ID, the product, and the distillation date. Each QC tube should be labeled with the sample ID, the product, the distillation date, and the batch ID.

12.3 Check the pH of each sample with pH paper and record the pH on the distillation log. If the pH is less than 12, adjust the pH to $\geq$ 12 with NaOH and notify the supervisor and document the pH adjustment on the distillation log. Make sure to also document the sample numbers and the initial volumes in the distillation log.

12.3.1 Check the sample first with the 12.5 to 14 pH paper. If the paper changes color, then record the pH. If it does not, use the 1 to 12 pH paper to verify that the sample is at pH 12.

12.3.2 For the method blanks and spike blanks, add 50 ml of DI water which has had the pH adjusted to $\geq$12 as outlined in section 8.13.

12.4 Add 6.0 ml of sample or standard to the sample tube and 0.02g of sulfamic acid solution. For blank spike add 6 ml of 0.100 mg/L CN spike solution and for matrix spike follow the same procedure used for making the 0.100 mg/L CN spiking solution but use the sample instead DI water and transfer 6 ml to the sample tube.

12.4.1 For soil samples :
Mix the sample well and weigh out from 0.5 to 1.0g of sample. Record the weight. Place the sample in the sample tube, then add 5.5 ml of DI water.
Make the Matrix Spike as follows:
Place 0.5g of well-mixed sample in the sample tube and add 5.5 ml of 0.10 mg/L CN spike solution.

12.5 Add 1.51 ml of 1 N NaOH (trapping solution) to **M** and place the membrane and cap.

12.6 Add 0.750 ml of MgCl2/H2SO4 to the sample tube and immediately push D end to the sample tube and press.

12.7 Place the samples and quality control into the preheated distillation block (120 ^{o}C) and set the timer for a minimum of 30 minutes. Make sure that the samples are fully pushed down into the block to ensure complete distillation.

12.8 After 30 minutes, take out the samples one at a time following the procedure outlined below.

12.8.1 Put on heat and chemical resistant gloves. Take one sample tube from the distillation unit and quickly (within 4 seconds), pull off the bottom of the tube and discard the sample liquid into waste. Let the distillate portion cool for 10 minutes.

12.8.2 Take the distillate portion of the tube and carefully swirl all of the sample from the sides into the collection end. Tap the tube gently to bring all of the drops to the bottom. Snap off the top end (D end) and throw it away.

12.8.3 Bring the final sample volume to 6 ml with DI water and mix well. Cap and parafilm both ends of the sample and store at 4 ºC if not analyzed immediately.

12.9 Document the final volume for each sample on the distillation log. Initial and date each sample. Make sure to any comments or observations to the distillation log.

13.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

14.0 QUALITY CONTROL

Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

14.1 Method Detection Limits (MDLs). MDLs should be established using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation

of the replicate analyses by 3.14, which is the student's ***t*** value for a 99% confidence level. MDLs should be determined approximately once per year or whenever there is a significant change in the background or instrument response.

14.2 Calibration curve. A calibration curve, consisting of a minimum of 6 points and a blank with the lowest point at or below the reporting limit, must be distilled before analyses. A distilled curve must be used for all sample analysis. Refer to the analytical method for calibration curve acceptance criteria.

14.3 Quality Control Sample (also referred to as Initial Calibration Verification Standard, (ICV)). For the EPA methods, a standard from a different source than the calibration standard must be analyzed after Initial Calibration Curve, before actual sample analysis is to proceed. For SW846 9010/9014, an external should be analyzed with each calibration. Normally this is analyzed at the beginning of the run after the CCV and CCB checks. For both EPA methods, the ICV should be within 10% of the true value. For the SW846 methods, the ICV should be within 15% of the true value.

14.4 Method Blank. The laboratory must distill and analyze a method blank with each set of samples. A minimum of one method blank is required for every batch. The method blank must contain the analyte at less that the reporting limit. If the method blank contains over that limit, the samples must be redigested or reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

14.5 Spike Blanks. For samples prepared on the micro distillation unit where all standards are distilled, only one blank spike is required per batch on each day that a standard curve is distilled. This standard must have a recovery of 85 to 115%. It is recommended that this standard be prepared from the same source as the calibration standards. If the blank spike does not meet the recovery limits, then the associated samples must be redistilled and reanalyzed. The exception to this rule is if the blank spike recovery is high and the results of the samples to be reported are less than the reporting limit, then the sample results can be reported with no flag.

14.5.1 Note: for the midi distillation unit, when the calibration curve is not distilled, then a high and a low standard must be distilled with each batch of 20 samples.

14.6 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 per batch. For drinking waters, a minimum of 1 in 10 samples must be spiked. (For Florida, a matrix spike must be added to 1 in 10 water samples).

14.6.1 For non-potable samples, the spike recovery must be assessed using in house limits. Until these limits can be generated, then default limits of 75 to 125 percent recovery must be applied. If a matrix spike is out of control, then the results must be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the spike

recovery should not be assessed against the control limits and should be footnoted to that effect.

14.6.2 The matrix spike recovery must be calculated as shown below.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} * 100 = \text{MS Recovery}$$

14.6.3 The duplicate RPD must be assessed using in house limits. Until these limits can be generated, then default limits of 20 percent RPD should be applied. If a duplicate is out of control, then the results must be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

14.6.4 The duplicate RPD must be calculated as shown below.

$$\frac{|\text{Sample Result - Duplicate Result}| * 100}{\text{(Sample Result + Duplicate Result)/2}} = \%\ \text{RPD}$$

14.7 Summary of major QC requirements. The table below shows a summary of the major QC requirements for cyanide.

QC Description	Frequency	Method and limits
ICV (external)	With every ICAL	SW846 9010/9014: 85-115% EPA 335.2: 90-110%
CCV	Every 10 samples	All methods 90-110%
CCB	Every 10 samples	All methods <1/2 RL
Distilled Std (spike blank)	One per distillation batch	All methods 85-115%
Method Blank	One per distillation batch	All methods <1/2 RL
Matrix Spike	Every distillation batch	SW846 9010/9014 75-125% EPA 335.2 M 75-125%
Duplicate	Every distillation batch	%RPD all methods ≤20%

15.0 DOCUMENTATION REQUIREMENTS

15.1 A worksheet must be used to record all distillation data. Make sure that all sample ID's, initial weights or volumes, and final volumes are clearly labeled on the worksheet. Any unusual characteristics of the samples must be noted in the comment section. All reagent information such as lot numbers, vendor, date made, etc. must be recorded in the reagent log book. As each batch is completed, the distillation log must be given to the lab supervisor for review. Place a copy of the completed analysis form in the cyanide distillation book.

16.0 DATA REVIEW AND REPORTING

16.1 All samples must be updated to QC batches in the LIMS system. The analyst is responsible for reviewing all data for compliance with the QC outlined in this SOP. They are responsible for making sure that the raw data is fully documented and it is updated and entered into the LIMS system.

16.2 After the analyst review is completed, the supervisor or a designated reviewer shall review the distillation data for technical compliance to the SOP.

17.0 POLLUTION PREVENTION & WASTE MANAGEMENT

17.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 17.1.1.

17.1.1 Waste Management. Individuals performing this method must follow established waste management procedures as described in SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

18.0 ADDITIONAL REFERENCES

18.1 Lachat Micro Distillation unit reference manual

STANDARD OPERATING PROCEDURE FOR CYANIDE ANALYSIS USING LACHAT™ AUTOANALYZER

Prepared by: Kelliam Garcia Date: 06/21/2006

Reviewed by: Steve Lee Date: 06/21/2006

Annual Review

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TITLE: STANDARD OPERATING PROCEDURE FOR CYANIDE ANALYSIS USING LACHAT™ AUTOANALYZER

METHOD REFERENCE: EPA 335.3 for waste waters (with EPA 335.2 modified for distillation.
SW846 9010/9014 modified for soils or aqueous wastes

***Revised Sections*: new procedure**

1.0 SCOPE AND APPLICATION

1.1 The purpose of this method is to determine the amount of total cyanide in any given sample. The sample is distilled prior to analysis to remove interferences. The distilled cyanide is reacted with chloramine-T at a pH of less than 8 to form cyanogen chloride. The cyanogen chloride is then reacted with pyridine-barbituric acid reagent to form a red-colored complex. The intensity of the dye is measured by recording the absorbance at 570 nm.

2.0 SUMMARY OF METHOD

2.1 The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by automated UV colorimetry.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. For waters, the reporting limit for this method is established at the lowest concentration standard in the calibration curve which is normally 0.010 mg/l. For soils, the reporting limit is established at .060 mg/kg. Detected concentrations below this concentration cannot be reported without qualification.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method.

3.2.2 Process all raw data for the replicate analysis in each MDL study.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For

QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of approximately 10% (For some methods this is mandatory and for some it is a recommendation only. Refer to individual method SOPs). For most methods, the mid-level calibration check standard criteria is ±10% of the true value.

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. For cyanide, method specific limits are defined. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK SAMPLE. Digest and analyze a high and a low standard with each batch of samples. These standards must have a recovery of 85 to 115 %. If the spike blank is outside of the control limits for a parameter, all samples must be redistilled and reanalyzed for that parameter. The exception is if the spike blank recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent should be used.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} \times 100 = \text{Matrix Spike Recovery}$$

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or

redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of three replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank). Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents, which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method, which would achieve the performance specifications for ASTM Type II water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards, which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 Cyanide is very toxic. Samples should be kept basic until the time of distillation to prevent the formation of HCN. Make sure that the distillation flasks are tightly closed and leak free during the distillation process.

5.3 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses.

6.0 PRESERVATION & HOLDING TIME

6.1 Water samples should be preserved with sodium hydroxide to a pH of $\geq$ 12 and cooled to 4 °C at the time of collection. Ascorbic acid should also be added to samples that are suspected to contain oxidizing agents such as chlorine. Soil samples should be cooled to 4°C at the time of collection.

6.2 All cyanides should be analyzed within 14 days of the date of collection.

7.0 INTERFERENCES

7.1 Many of the interferences in this method can be reduced or eliminated by the distillation step. All samples should be distilled before analysis. Sulfides can cause interferences but these can be removed by treatment of the distillate with cadmium carbonate solution. High results may also be obtained due to interferences from nitrate or nitrite. These interferences can be eliminated by pretreatment of the sample with sulfamic acid during the distillation step. Oxidizing agents interfere by decomposing most of the cyanide. Ascorbic acid, added to the sample at the time of collection, will minimize the effect of these oxidizing agents.

7.2 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10 mg/kg were not found to interfere.

7.3 Fatty acids, detergents, surfactants, and other compounds may cause foaming during the distillation when they are present in high concentrations.

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This interference can be minimized by using a smaller sample size or by following a special extraction procedure. Check with the lab manager or supervisor for additional information.

8.0 APPARATUS

8.1 Automated continuous flow analyzer designed to deliver and react sample and reagents in the required order and ratios. Lachat QuickChem 8500 is being used.

8.1.1 Autosampler

8.1.2 Multichannel pump

8.1.3 Reaction manifold.

8.1.4 Colorimetric detector

8.1.5 In-line heating unit

8.1.6 Real time data acquisition device (either electronic or hard copies.)

8.2 Balance. Analytical balance capable of accurately weighing to the nearest 0.0001 g.

8.3 Volumetric glassware. Class A volumetric pipettes and flasks as required. Note: All glassware should be washed with soap and tap water and then well rinsed with distilled, deionized water

9.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. Deionized water should be used whenever water is required. The expiration date for standards and reagents is the date supplied by the manufacturer. If the manufacturer gives no expiration date, then default of 6 months is used.

9.1 Carrier solution, 0.25 M Sodium Hydroxide. Dissolve 10.0g of sodium hydroxide in a 1liter volumetric flask containing approximately 800 ml of DI water. Dilute to a final volume of 1 liter and mix well.

9.2 Phosphate Buffer Solution, 1.0 M. Dissolve 138 g of sodium phosphate monobasic (NAH_2PO_4 H_2O) in approximately 800 ml of DI water, dilute to a final volume of 1 liter and mix well.

9.3 Chloramine-T Solution. Add 2.0 g of chloramine-T to a 500ml volumetric flask containing approximately 250 ml of DI water. Dilute to a final volume of

500 ml with DI water and mix well. Note: This reagent should be prepared daily.

9.4 Pyridine-Barbituric Acid Reagent. Note: Prepare this reagent in a hood. Weigh 15.0 g of barbituric acid in 1-liter volumetric flask. Add 100.0 ml of water, rinsing down the sides of the flask to wet the barbituric acid. Add 75 ml of pyridine with stirring and mix until the barbituric acid dissolves. Then add 15 ml of concentrated hydrochloric acid and mix. Dilute to a final volume of 1 liter with DI water and mix well. Prepare fresh weekly.

9.5 Cyanide Stock Solution, 1000 mg/l. Dissolve 2.51 g of KCN and 2.0 g of KOH in approximately 900 ml of DI water in a 1-liter volumetric flask. Dilute to a final volume of 1 liter. Standardize weekly with silver nitrate solution. This standard can be purchased commercially.

9.6 Standard Silver Nitrate Solution, 0.0192 N. Crush approximately 5 g of silver nitrate crystals and dry to constant weight at 40 °C. Weigh out 3.2647 g of dried silver nitrate into a 1 liter flask containing approximately 800 ml of DI water. Dilute to a final volume of 1 liter with DI water and mix well.

9.7 Intermediate Cyanide Solution, 10.0 mg/l. Pipet 1.0 ml of 1000 mg/l stock solution of CN into a 100 ml volumetric flask containing approximately 50 ml of DI water. Dilute to a final volume of 100 ml and mix well. Note: These volumes may need to be adjusted to obtain the final concentration of 20.0 mg/l based on the standardized value of the cyanide stock. This intermediate may be used for a maximum of one week or until the expiration date of the weekly stock standardization, whichever is sooner.

9.8 Cyanide Standard Solutions. Suggested standard concentrations are shown below. All standards are made up using the above intermediate solution as outlined in the table below. Alternate standard concentrations may be used as long as all method requirements are met. **Standards should be diluted with 0.25 N NaOH**. These standards should be made fresh daily before distillation or analysis.

ml of 10.0 mg/l CN	Final Volume	Concentration (mg/l)
5.0 ml	100 ml	0.50 mg/l
3.0 ml	100 ml	0.30 mg/l
2.0 ml	100 ml	0.20 mg/l
1.0 ml	100 ml	0.10 mg/l
0.50 ml	100 ml	0.050 mg/l
0.10 ml	100 ml	0.010 mg/l

9.9 Benzalrhodanine indicator. Dissolve 20 mg p – dimethylaminobenzalrhodanine in 100 ml acetone.

10.0 PROCEDURE

10.1 Below is a step-by-step procedure for the analysis of samples for the determination of cyanide. At the end of this SOP is a short summary outlining the overall procedure.

10.2 If the cyanide stock solution (made in the laboratory) has not been standardized within a week, then the stock must be standardized before proceeding further with the analysis. The standardization can be done following the procedure outlined below.

10.2.1 Volumetrically measure out 25.0 ml of the 1000 mg/l cyanide stock solution into a graduated plastic beaker. Add 10 to 12 drops of benzalrhodanine indicator to the solution. Place a stir bar in the beaker.

10.2.2 Fill a buret with 0.0192 N silver nitrate solution. Titrate the sample with continuous stirring until the color changes from yellow to a brownish-pink. Approximately 25 ml of silver nitrate solution should be needed to reach this endpoint. Also analyze a method blank that has been brought to pH > 12 with KOH following this procedure.

10.2.3 Calculate the concentration of the stock cyanide solution using the equation shown below.

$$\text{CN, mg/l} = \frac{(A - B) \times 1000}{25 \text{ ml}}$$

Where A = the volume of $AgNO_3$ used to titrate the sample.
B = the volume of $AgNO_3$ used to titrate the blank.

Make sure to modify the volumes used in the preparation of the intermediate cyanide stocks based on the result of this standardization.

10.3 Install the cyanide reaction manifold. Check all tubing and change any tubing that is flat, dirty, etc. Install the appropriate sample loop and the appropriate filter. Place the tubing in the bottles for the pyridine/barbituric acid, the chloramine-T, the phosphate buffer, and the 0.25 M NaOH carrier. Also make sure that the waste container is in place. Refer to the manufacturer's instructions for additional information.

10.4 Turn on the pump, turn on the QuickChem 8500 system and turn on the autosampler.

10.5 Check reagent flow through the system.

10.6 Use the following commands for running QuickChem software.

10.6.1 From the desktop select Omnion 3.0 by double clicking on the icon.

10.6.2 On the left upper corner select “open”.

10.6.3 Select CN file and then select the template.

10.6.4 Template should display Cal Blk, Cal 1, Cal 2, Cal 3, Cal 4, and Cal 5. Cal Std’s will be highlighted in blue.

10.6.5 Click “preview” (left upper corner) to baseline. Let system stabilize for 2 to 3 minutes. At this time load the autosampler tray.

10.6.6 When you are ready to start running the curve, click “stop” (left upper corner) to end the preview then click “start” (upper left corner) to begin the run.

10.6.6.1 While the samples are running, you will see a stop sign on the tool bar. To stop the autosampler, click on the “stop” icon and select the appropriate option.

10.6.7 After the calibration standards have finished running, click on the last icon in the channel 1 window to view the curve. If the calibration does not meet the criteria, then the instrument must be recalibrated. If the calibration meets criteria, then proceed with the section 10.6.8

10.6.8 Using the same template, delete the calibration standards. Then begin to type in the run following the sample log.

10.6.9 To insert sample lines, click on the gray box to the left of the sample line. Right click on the box and choose “append many” and enter the corresponding number of sample lines. Then type in the sample identification numbers. Next change the cup numbers to correspond with the correct position on the autosampler tray.

10.6.10 When the run is complete, the stop sign goes away. Prepare a customized report. Go to “report” (left upper corner) and choose “open format” and then choose “CN template” and click “OK”.

10.7 At the end of the run, rinse out the system with DI water.

10.8 Calculations may be either done manually, using the Lachat software, or using the Accutest LIMS system. Check with the lab supervisor or manager for more instructions.

10.8.1 <u>For water samples</u>, the following calculations should be used:

$$\text{Sample conc. (mg/l)} = \frac{\text{Calculated value (mg/l)} * \text{DF} * \text{final distillation volume (ml)}}{\text{Initial distillation volume (ml)}}$$

10.8.2 For soil samples, the following calculations should be used:

$$\text{Sample conc. (mg/kg/)} = \frac{\text{Calculated value (mg/l)} * \text{DF} * \text{final distillation volume (ml)}}{\text{Initial distillation weight (g)} * (\%\text{sol}/100)}$$

10.9 Review data and assemble a data package including the following items:

- Inorganic run narrative
- Results report, showing dilution and weight correction factors.
- Printout of peaks.
- Printout of the curve
- Distillation prep sheet
- Reagent sheets
- Copy of the logbook

11.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

12.0 QUALITY CONTROL

12.1 Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

12.2 Method Detection Limits (MDLs). MDLs should be established using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's ***t*** value for a 99% confidence level. MDLs should be determined approximately once per year or whenever there is a significant change in the background or instrument response.

12.3 Linear Dynamic Range (LDR). For each instrument, the upper limit of the linear dynamic range must be established. A linear calibration should be prepared from 3 standards, one of that is close to the upper limit of the linear range. The LDR is determined by analyzing exceedingly higher standard concentrations of an analyte until the observed analyte concentration is no more than 10 percent below the true value of the

standard. Note: The normal cyanide calibration curve can be used to verify the linear dynamic range.

12.4 Calibration Curve. Each day a calibration curve consisting of a minimum of 6 points and a blank must be run. The calibration curve should have a correlation coefficient of at least 0.995 and the concentration intercept must be less than the reporting limit for the method. If the calibration curve fails then the problem must be resolved and the curve reanalyzed before any samples can be analyzed.

The calculation to be used for the calibration curve is shown below.

Correlation Coefficient

$$r = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\sqrt{\Sigma(x - \bar{x})^2 \Sigma(y - \bar{y})^2}}$$

Where: r = correlation coefficient
x = amount of analyte
y = response of instrument

$\bar{x}$ = average of x values

$\bar{y}$ = average of y values

12.5 Quality Control Sample (also referred to as Initial Calibration Verification Standard, (ICV)). For the EPA methods, a standard from a different source than the calibration standard must be analyzed and pass after each Initial Calibration before analysis of actual samples may proceed. For both EPA methods, the ICV should be within 10% of the true value. For the SW846 methods, the ICV should be within 15% of the true value.

12.6 Method Blank. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every batch. The method blank must contain the analyte <1/2RL. If the method blank contains over that limit, the samples must be redigested or reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

12.7 Spike Blank. For samples prepared on the micro distillation unit, one blank spike is required per batch. This standard must have a recovery of 85 to 115%. It is recommended that this standard be prepared from the same source as the calibration standards. If the blank spike does not

meet the recovery limits, then the associated samples must be redistilled and reanalyzed. The exception to this rule is if the blank spike recovery is high and the results of the samples to be reported are less than the reporting limit, then the sample results can be reported with no flag.

Blank Spike recovery calculated as shown below:

$$\frac{\text{(Spike Result)}}{\text{(Amount Spiked)}} * 100 = \text{BS Recovery}$$

12.8 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples for non-potable and soil samples.

12.8.1. For non-potable and solid samples, the spike recovery should be assessed using in house limits. Until these limits can be generated, then default limits of 75 to 125 percent recovery should be applied. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the spike recovery should not be assessed against the control limits and should be footnoted to that effect.

The matrix spike recovery should be calculated as shown below.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} * 100 = \text{MS Recovery}$$

12.8.2. The duplicate %RPD should be assessed using in house limits. Until these limits can be generated, then default limits of 20 percent RPD should be applied. If a duplicate is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of $\pm$ the reporting limit, then the duplicate is considered to be in control.

The duplicate RPD should be calculated as shown below.

$$\frac{|\text{Sample Result - Duplicate Result}| * 100}{\text{(Sample Result + Duplicate Result)/2}} = \%\ \text{RPD}$$

12.9 Continuing Calibration Verification (a.k.a. the instrument performance check solution). Analyze the continuing calibration verification solution and the continuing calibration blank after the initial calibration, after every tenth sample, and at the end of the sample run. If the CCV solution is not within 10 percent of the true value, then no samples can be reported in the area bracketed by that CCV. (Note: the exception is if the CCV is biased high and the samples are less than the detection limit. In that case, the samples can be reported with no flag). The CCV concentration should be at or near the mid-range of the calibration curve.

12.10 Continuing Calibration Blank. Analyze the continuing calibration verification solution and the continuing calibration blank after the initial calibration, after every tenth sample, and at the end of the sample run. If the CCB is not less than the reporting limit, then no samples can be reported in the area bracketed by the failing CCB.

12.11 Summary of major QC requirements. The table below shows a summary of the major QC requirements for cyanide.

QC Description	Frequency	Method and limits
ICV (external)	With every ICAL	SW846 90109014: 85-115% EPA 335.2: 90-110%
CCV	Every 10 samples	All methods 90-110%
CCB	Every 10 samples	All methods <1/2 RL
Distilled Std (spike blank)	One per distillation batch	All methods 85-115%
Method Blank	One per distillation batch	All methods <1/2 RL
Matrix Spike	Every distillation batch	SW846 9010/9014 75-125% EPA 335.2 M 75-125%
Duplicate	Every distillation batch	%RPD all methods ≤20%

13.0 DOCUMENTATION REQUIREMENTS

13.1 Each analyst should review all data and assemble a data package consisting of the following information.

- Inorganic run narrative
- Results report, showing dilution and weight correction factors.
- Printout of peaks.
- Printout of the curve
- Distillation prep sheet
- Reagent sheets
- Copy of the logbook

13.2 In addition, all reagent information such as lot numbers should also be recorded in the reagent logbook. Any unusual characteristics of the samples should be noted in the final result section of the final data report. Make sure that all sample ID's and dilutions are labeled on the data.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to

the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.0 PROCEDURE SUMMARY

15.1 The procedure outlined below is a summary for quick reference purposes only. Make sure to read and understand the entire SOP before starting an analysis.

a. Prepare reagents. Standardize CN stock.
b. Insert the reaction module, connect all reagents, and start the instrument.
c. Pour the calibration standards and start calibrating.
d. Enter sample information into autosampler table. (Make sure to save this and print this.)
e. Approve calibration.
f. Submit autosampler tray for analysis.
g. Print out peaks and results.
h. Review results and copy to the network.
i. Turn in reviewed data to general chemistry supervisor.

16.0 ADDITIONAL REFERENCES

16.1 Lachat QuickChem 8500 Autoanalyzer manuals.

16.2 Lachat QuickChem Method 10-204-00-1-X

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SULFIDE

Prepared by: _____Ellen Pampel_____ Date: _03/16/04_

Reviewed by: _____Svetlana Izosimova_____ Date: _04/05/04_

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Document Control

Issued to: _____QA Department_____ Date: _04/05/04_

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TEST NAME: SULFIDE

METHOD REFERENCE: EPA 376.1/ SW846 9030

DEPARTMENT: GN

DETECTION LIMIT: 1.0 mg/l for waters and 20 mg/kg for soils.

Added Doc Control page, revised sec 2, 3, 12, 14

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is used as a measure of sulfides and is applicable to all water and wastewater. A modification of this method is used to determine water-soluble sulfides in soil samples. The iodometric titration is based on EPA Method 376.1.

1.2 Excess iodine is added to the sample, which may have been preserved with zinc acetate and sodium hydroxide to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur when the sample is acidified with HCl. The excess iodine is back titrated with sodium thiosulfate.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

Water samples should be preserved with zinc acetate and sodium hydroxide to pH ≥ 9 and collected in three 250-ml bottles. Water and soil samples should be kept under refrigeration at 4°C until they are analyzed.

2.2 Holding Time

All samples should be analyzed as soon as possible, but definitely within 7 days of the date of collection.

3.0 REPORTING LIMITS AND METHOD DETECTION LIMITS

3.1 The reporting limit for sulfide in water has been established at 1.0 mg/l and 20 mg/kg for soils

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria. Experimental MDLs must be determined annually for this method.

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3.3 MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level.

4.0 INTERFERENCES

Reduced sulfur compounds, which decompose in acid, such as sulfite and thiosulfate, may yield erratic results. Volatile iodine consuming substances will give high results. Samples should be taken with a minimum of aeration as oxidation may affect results.

5.0 APPPARATUS

The following items are needed for the analysis of samples following the method outlined below:

5.1 25 ml buret

5.2 Graduated beakers or flasks

5.3 Stir bars

5.4 Stir plates

5.5 Volumetric pipets

5.6 Filtering apparatus with 934 AH Whatman glass fiber filters or equivalent

6.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. De-ionized water should be used whenever water is required.

6.1 Hydrochloric acid, HCl, 6N. Add 50 ml of concentrated HCl to 50 ml of DI water and mix.

6.2 Standard iodine solution, 0.0250 N: Dissolve 20 to 25 g of KI in a little water in a 1 liter volumetric and add 3.2 g of iodine. Allow it to dissolve. Dilute to 1 liter and standardize against 0.025 N sodium thiosulfate.

6.3 Sodium Thiosulfate solution, 0.025 N: This solution is commercially available.

6.4 Starch indicator solution: This solution is commercially available.

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6.5 Zinc acetate solution: Dissolve 220 g of zinc acetate in 870 ml of DI water.

6.6 Sodium hydroxide, 6N: Weigh out 24 g of sodium hydroxide and dilute to a final volume of 100 ml with DI water.

6.7 Sodium hydroxide, 1N: Dissolve 40 g of NaOH in 500 ml of DI water. Dilute to 1 liter with DI.

6.8 Sulfide Stock Solution, 537 mg/l: Dissolve 4.02 g of $Na_2S \bullet 9H_2O$ in approximately 980 ml DI water. Adjust the pH to between 9 and 11 with 1N NaOH (6.7). Dilute to 1 liter with DI in a volumetric flask. **NOTE:** Sodium sulfite nonahydrate is extremely hydroscopic. This solution must be made weekly.

7.0 TITRATION PROCEDURE

7.1 Before starting on the samples, standardize the iodine solution using the following procedure.

7.1.1 Volumetrically pipette 5.0 ml of the iodine solution into the beaker. Then measure out 200 ml of DI water into the beaker. Place a stir bar in the solution and place the beaker on a stir plate. Add several drops of HCl, to bring the pH to less than 2 and then add several drops starch solution.
Note: The amount of HCl solution needed may vary with the strength of the solution. If you do not get a blue color with several drops of starch, first verify that the pH is less than 2. If the pH is correct, then add addition starch solution until a blue color is obtained.

7.1.2 Fill a 25-ml buret with 0.025 N sodium thiosulfate solution. Titrate the iodine solution with the sodium thiosulfate until the last blue color disappears. Record the volume of sodium thiosulfate used. At a minimum, perform this titration in duplicate.

7.1.3 Calculate the normality of the iodine as shown below. The iodine normality used should be the average of the normality for the two titrations of the DI blanks.

$$\text{N of iodine solution} = \frac{(\text{ml of Na thiosulfate}) * (0.025\ \text{N})}{(5.00\ \text{ml of iodine})}$$

7.2 For soil samples, weigh out 25.0 g and dilute to 250 ml with DI water. Mix well. Let settle and filter through Whatman 934 AH filters until clear. Measure out 200 ml to use for analysis. Prepare a duplicate sample, a matrix spike, a spike blank, and a method blank. For the method blank, use 200 ml of DI

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water. **NOTE:** The soil sample should be prepared immediately before titration.

7.3 For water samples that are **not preserved** with Zn acetate/ NaOH, mix the sample well and measure out 200 ml to use for analysis. Continue with step 7.5

7.4 For water samples that are **preserved** with Zn acetate/ NaOH, mark the side of the sample bottle at the level of the sample meniscus.

7.5 Volumetrically pipette 3.0 ml of the iodine solution (6.2) into a flask and add 2 ml of 6N HCl and swirl to mix. Gently transfer the contents of the sample bottle to the flask (or the 200 ml from step 7.3) with a minimum of aeration under the surface of the iodine. Rinse the preserved sample bottle with sufficient DI to ensure a quantitative transfer to the flask. Add a stir bar and stir slowly. The solution should be yellow. If not, add additional iodine. Add a small amount of starch solution and the solution should turn blue.
Note: The amount of HCl solution needed may vary with the strength of the solution. If you do not get a blue color with several drops of starch, first verify that the pH is less than 2. If the pH is correct, then add addition starch solution until a blue color is obtained.

7.6 Titrate the sample slowly with the 0.025N sodium thiosulfate solution until the last bluish tint disappears. The solution is normally clear at the end point. Note: The sample should be gently, but continuously stirred during the titration.

7.7 Each batch of samples, set up quality control samples, consisting of a method blank, a spike blank, a matrix spike and a duplicate. The spike blank and the matrix spike are spiked with 5.0 ml of sulfide Stock solution (6.8) when the sample is transferred to the flask. **NOTE:** For MS and SB, use 10.0 ml of iodine, instead of 3.0.

7.8 For water samples that are preserved with Zn acetate/ NaOH, measure the volume used by filling the original bottle with water to the line marked on the side of the bottle then pour the water into a graduated cylinder. Record the volume.

7.9 If interference is suspected, the samples may be taken through a pre-treatment step as described below.

7.9.1 If the sample was not preserved, measure out 200 ml of sample. Add 0.30 ml of zinc acetate solution and 4 to 5 drops of 6N NaOH solution to bring the pH above 9. Mix gently. Let the precipitate settle for approximately 30 minutes. Continue with step 7.9.3

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7.9.2 If the sample was preserved, verify the pH > 9 and mark the level of the meniscus on the side of the bottle. Additional Zn acetate may be added to ensure complete precipitation. Continue with step 7.9.3.

7.9.3 Filter the sample through glass fiber filter paper and then place the filter paper in a beaker with approximately 200-ml of DI water. Proceed with the titration starting at step 7.5.

8.0 CALCULATIONS

The calculations for both soils and waters are outlined below.

For water samples, the following calculations should be used.

$$\text{Sulfide in mg/l} = \frac{((V_iN_i) - (V_tN_t)) \times 16000}{\text{sample volume in ml}}$$

Where:
V_i = volume of iodine solution in ml
N_i = normality of iodine solution in ml
V_t = volume of sodium thiosulfate solution in ml
N_t = normality of sodium thiosulfate solution

For soil sample, the following calculation should be used.

$$\text{Sulfide in mg/kg} = \frac{((V_iN_i) - (V_tN_t)) \times 16000 \times V_f}{V_s \times (\%\text{solids}/100) \times W_i}$$

Where:
V_i = volume of iodine solution in ml
N_i = normality of iodine solution in ml
V_t = volume of sodium thiosulfate solution in ml
N_t = normality of sodium thiosulfate solution
V_s = volume of sample titrated
V_f = final volume of sample after preparation
W_i = initial weight of sample prepared

9.0 QUALITY CONTROL

9.1 A method blank is required at a minimum of one per batch of 20 samples or one for each analysis day. The concentration of sulfide in the MB must be less than ½ the reporting limit.

9.2 A spike blank is required at a minimum of one per batch of 20 samples or one for each analysis day. Until in-house limits can be generated, use 80 to 120% recovery as the limits. See Table 1.

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9.3 A matrix spike is analyzed for every batch of 20 samples or one for each analytical day. In-house recovery limits should be used to assess the data. Use 75 to 125% recovery until the limits can be generated. If there is insufficient sample for a matrix spike, a second spike blank must be analyzed.

9.4 A duplicate is analyzed for every batch of 20 samples or one for each analytical day. Until in-house limits can be generated, 20% RPD should be used as the limit. A spike blank duplicate may be used, if there is insufficient sample available for the duplicate. If the RPD is greater than 20 and the sample and the duplicate are less than 5 times the RL, then the duplicate is considered in control and appropriately footnoted.

10.0 GLASSWARE CLEANING

All glassware should be washed with soap and tap water and then well rinsed with de-ionized water.

11.0 DOCUMENTATION REQUIREMENTS

All data should be recorded in a sulfide analysis log. Any unusual characteristics of the samples should be noted in the comment section. In addition, all reagent information such as lot numbers, vendors, date made, etc. should be recorded in the reagent logbook. All sample ID's and dilutions should be clearly labeled on the data.

12.0 SAFETY

The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual. Safety glasses and lab coats should be worn at all times in the lab. Gloves should be worn when handling samples.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

13.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in Section 13.2.

13.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples.

14.0 DEFINITIONS

14.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours which ever comes first.

14.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

14.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run.

14.4 holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

14.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

14.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

14.7 Continuing Calibration Verification 2 (CCV2): A check standard used to verify instrument calibration. CCV2 is analyzed at the time of instrument calibration and is usually the same concentration as the lowest standard or the reporting limit.

14.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

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14.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and bias of a method in a given sample matrix.

14.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

14.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

14.12 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

Table 1 QC Criteria

Quality Control	Acceptance Criteria	Corrective Action
Initial Calibration: r^2 = coefficient of determination, or: r = coefficient of correlation	≥0.990 ≥0.995	Rerun calibration standards, and/or prepare new calibration standards and recalibrate the instrument, or document why the data are acceptable.
Initial Calibration Verification standard (ICV)	± 10% of the standard's true value	Rerun standard, and/or prepare new standard, and/or recalibrate instrument, or document why the data are acceptable.
Continuing Calibration Verification standard (CCV), and Low-Level Standard (where applicable)	± 10% of the standard's true value ± 10% of the standard's true value	Rerun standard, and/or recalibrate instrument and reanalyze all samples run since the last acceptable CCV, or document why the data are acceptable.
Method blank (MB)	< 1/2RL	Reanalyze, and/or stop the run and determine the source of contamination, or document why the data are acceptable.
Blank Spike (BS or LSC)	± 10 to 20%, or as per method	Determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable.

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Quality Control	Acceptance Criteria	Corrective Action
MS/MSD	± 15 to 25%, or as per method.	Determine and correct the problem, reanalyze samples and MS/MSD, or document why data are acceptable
Sample Duplicate	± 15 to 20%, or as per method.	Determine and correct cause of the poor reproducibility

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IGNITABILITY

Prepared by: _____Ellen Pampel_____ Date: _12/29/04__

Reviewed by: _____Svetlana Izosimova_____ Date: _03/21/05_

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

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TEST NAME: IGNITABILITY

REFERENCES: SW846 1010, SW846 Chapter 7

DEPARTMENT: GN

DETECTION LIMIT: Not Applicable

Added Document Control pg,

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is used to determine the flash point of various solid and liquid samples.

1.2 The sample is loaded in the cup of the tester. While the sample is being stirred and slowly heated at a constant rate, a flame is directed into the cup. The flash point is defined as the lowest temperature at which the vapor, generated by the sample, will ignite.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation: The preservation for this analysis is not specified.

2.2 Holding Time: The holding time for this analysis is not specified.

3.0 REPORTING and METHOD DETECTION LIMIT

Not Applicable

4.0 INTERFERENCES

Ambient pressure, sample homogeneity, drafts, and operator bias can affect flash point values.

5.0 APPARATUS

5.1 Pensky-Martens Closed Flash Tester

5.2 Thermometer, minimum range of 60 to 212°F.

6.0 REAGENTS

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6.1 p-Xylene reference solution.

7.0 PROCEDURE

7.1 Thoroughly clean and dry all parts of the cup and its accessories before starting the test.

7.2 Fill the cup with the sample to be tested to the level indicated by the fill mark.

7.3 Place the lid on the cup and set the latter in the stove. Be sure to properly engage the locking device.

7.4 Insert the thermometer.

7.5 Light the test flame and adjust it to a diameter of 4 mm. Most samples should be heated at a rate of approximately 9 to 11°F per minute.

7.6 For liquid samples, turn on the stirrer. The stirrer should turn at approximately 90–120 rpm for most liquid samples. For more viscous samples, a higher stirrer speed may be required.

7.7 Apply the test flame and test for ignitability as described below.

NOTE: All samples that show a flash point under 212°F must have the results verified by a second analyst. Both the original analyst and the verifying analyst must sign the data. Both results must be recorded on the data sheet.

7.7.1 Apply the test flame at each temperature that is a multiple of 2°F up to a temperature of 212°F.

7.7.2 Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is lowered into the vapor space of the cup in 0.5 sec, left in its lowered position for 1 sec., and quickly raised to its high position.

NOTE: Do not stir the sample while applying the test flame.

7.8 Record the starting temperature for each sample.

7.9 Record as the flash point, the temperature at the time the test flame application causes a distinct flash in the interior of the cup. If the flash point is greater than 212°F, then record the results as greater than the final temperature tested (212°F).

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NOTE: Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame. The actual flash will have occurred when a large flame propagates itself over the surface of the sample.

8.0 CALCULATIONS

8.1 Record the temperature read in degrees Fahrenheit in the ignitability logbook. The calculation of degrees C to degrees F is as follows:

9/5 (C) + 32 = F

8.2 Correct for barometric pressure using the following equation:

Corrected Results = F + (0.06*(760-(P*760mmHg/1013mbarHg)))

Where: F = observed temperature
P = barometric pressure in mb Hg

8.3 When pressure is in mmHg, the equation simplifies to:

Corrected Results = F + (0.06*(760-P))

9.0 QC REQUIREMENTS

9.1 Thermometer calibration. The thermometers used for this test must be calibrated against an NBS certified thermometer in the temperature range of use at least once per year. The calibration must be documented and any correction factor applied when used. If a correction factor is used, the raw data must show the uncorrected and the corrected temperatures, the correction factor, and the date the thermometer was calibrated.

9.2 p-xylene check standard. An aliquot of p-xylene must be analyzed daily or with each batch of samples. The flash point of p-xylene is 77 ± 4.3°F

9.3 Duplicate. A duplicate must be analyzed for every batch of 10 samples, or on each analysis day, whichever is more frequent. The results obtained must be within 5 percent of the original result.

9.4 All samples with an ignitability of less than or equal to 212°F must have results verified by a second analyst. Both the original and the verification result must be recorded and both analysts should sign the worksheet. In addition, a blank must be analyzed between the original result and the second result to confirm that there was no carryover from an earlier sample or check.

10.0 SAFETY

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The analyst should follow normal safety procedures as outlined in the Laboratory Safety Manual. All work should be done in a hood and the analyst should wear a lab coat and safety glasses for all analytical work. Gloves should be worn when handling samples.

11.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

11.0 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in Section 11.2.

11.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples.

12.0 DEFINITIONS

12.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours which ever comes first.

12.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

12.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run.

12.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

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12.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

12.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

12.7 Continuing Calibration Verification 2 (CCV2): A check standard used to verify instrument calibration. CCV2 is analyzed at the time of instrument calibration and is usually the same concentration as the lowest standard or the reporting limit.

12.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

12.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and bias of a method in a given sample matrix.

12.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

12.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

12.12 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

Table 1 QC Criteria

Quality Control	Acceptance Criteria	Corrective Action
Initial Calibration: r^2 = coefficient of determination, or: r = coefficient of correlation	$\geq$0.990 $\geq$0.995	Rerun calibration standards, and/or prepare new calibration standards and recalibrate the instrument, or document why the data are acceptable.

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Quality Control	Acceptance Criteria	Corrective Action
Initial Calibration Verification standard (ICV)	± 10% of the standard's true value	Rerun standard, and/or prepare new standard, and/or recalibrate instrument, or document why the data are acceptable.
Continuing Calibration Verification standard (CCV), and Low-Level Standard (where applicable)	± 10% of the standard's true value ± 10% of the standard's true value	Rerun standard, and/or recalibrate instrument and reanalyze all samples run since the last acceptable CCV, or document why the data are acceptable.
Method blank (MB)	< 1/2RL	Reanalyze, and/or stop the run and determine the source of contamination, or document why the data are acceptable.
Blank Spike (BS or LSC)	± 10 to 20%, or as per method	Determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable.
MS/MSD	± 15 to 25%, or as per method.	Determine and correct the problem, reanalyze samples and MS/MSD, or document why data are acceptable
Sample Duplicate	± 15 to 20%, or as per method.	Determine and correct cause of the poor reproducibility

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CORROSIVITY (SOIL pH BY ELECTRODE)

Prepared by: _____Ellen Pampel_____ Date: _03/05/04_

Reviewed by: _____Steve Lee_____ Date: _07/25/04_

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

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TEST NAME: CORROSIVITY (SOIL pH BY ELECTRODE)

METHOD REFERENCE: SW846 9045C, Chap 7.2.1 M

DEPARTMENT: GN

DETECTION LIMIT: NOT APPLICABLE

Added Doc Control page, MDL sec, Definitions, revised sec 1, 6, 7,

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Per SW846, corrosivity has two properties; pH and corrosivity toward steel.

1.2 This method measures the pH of soil by mixing equal amounts of the soil and DI water and then measuring the pH while the sample is being continuously stirred. This method is based on a modification of SW846 method 9045C.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

The sample containers should be filled completely and sealed tightly and stored at 4°C.

2.2 Holding Time

Samples should be analyzed as soon as possible, preferably in the field or as soon as they are received by the laboratory.

3.0 REPORTING and METHOD DETECTION LIMITS

Not applicable

4.0 INTERFERENCES

4.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of greater than 10, the measured pH may be incorrectly low. For samples with a true pH of less than 1, the measured pH may be incorrectly high.

4.2 In addition, temperature fluctuations and dirty electrodes can cause measurement errors. (See electrode-cleaning procedure below.)

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5.0 APPARATUS

The following items are needed for the analysis of pH:

5.1 pH meter with means for temperature compensation

5.2 pH electrode

5.3 Graduated plastic beakers

5.4 Stir plate

5.5 Magnetic stir bars

5.6 Thermometer or temperature sensor for automatic compensation.

6.0 REAGENTS

All reagents listed below must be made from reagent grade chemicals.

6.1 De-ionized water. This water must be monitored daily before use.

6.2 Buffer solution at pH 4, pH 7 and pH 10. Commercially available solutions that have been validated by comparison to NIST standards are recommended for routine use. All buffers must be labeled on receipt and after opening. Fresh buffer must be poured each time a new analysis is started.

6.3 Buffers with pH < 4 and > 10 may be used, to insure accuracy at very low or very high pH's.

7.0 PROCEDURE

Below is detailed description of the procedure that must be followed for the determination of pH. Make sure to properly document each step of the analysis.

7.1 Make sure that the pH electrode is clean. If the electrode is coated with oil or grease, then it must be washed with detergent and then rinse it well with DI water. Place the electrode in a beaker containing 1:10 HCL so that the lower third of the electrode is submerged and let the electrode soak for approximately 10 to 30 minutes. Then thoroughly rinse the electrode with DI water and place in a pH 7 buffer to store.

7.2 Weigh out approximately 20 g or each sample to be analyzed into a clearly labeled graduated plastic beaker. Add 20 ml of DI water to each sample.

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Record the weights and the volumes used in the pH logbook. Stir the suspension for 15 minutes.

Note: If the waste is hygroscopic and absorbs all the reagent water, begin the experiment again using 20 g of waste and 40 ml of reagent water

Note: If the supernatant is multiphase, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned if it becomes coated with oily material.

7.3 Connect the pH electrode to the pH meter. Enter the proper temperature and then calibrate the meter using two points, either pH 4 and pH 7 or pH 7 and pH 10 depending on where the pH of the samples are expected to be.

Note: For specific calibration procedure, see the instruction manual for the meter being used.

7.4 After the calibration is complete, analyze the 3 buffer solutions to ensure that an accurate calibration was obtained. Record the results on the pH worksheet. Record the temperature of the sample.

7.5 Place the sample in a clean glass beaker using sufficient volume to cover the sensing elements of the electrodes and to give adequate clearance for the magnetic stirring bar.

7.6 Place the sample on the stir plate and stir. Lower the pH electrode into the solution and record the pH.

7.7 After every 10 samples have been analyzed, reanalyze the buffer solutions and record the results on the pH worksheet.

8.0 QUALITY CONTROL

Below is the quality control requirement that should be followed for pH analyses on soil samples. Refer to the Accutest quality control manual for additional information on quality control calculations and requirements.

8.1 A new calibration must be performed and documented each day that analysis are done. A pH 4, pH 7 and pH 10 solution should be analyzed immediately after the calibration is completed. The buffer checks must be within 0.05 pH units of the true values.

8.2 A calibration check standard should be analyzed after every 10 samples and after the last sample analyzed in the batch. The check standard should be within 0.05 pH units of the true value of the check standard solution.

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8.3 A duplicate analytical sample should be analyzed for every 20 samples. The RPD for the sample and the duplicate should not exceed 10 percent.

9.0 DOCUMENTATION REQUIREMENTS

All data must be recorded in the appropriate logbooks, which show the results for all quality control and samples. In addition, the pH meter that was used and the buffer solutions used should be documented. All work must be dated and signed by the analyst. Any changes should be crossed out with a single line and should be initialed and dated.

10.0 SAFETY

The analyst should follow normal safety procedures as outlined in the Laboratory Safety Manual. A lab coat and safety glasses should be worn for all lab work.

11.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

11.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in Section 11.2.

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12.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of

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the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

12.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run.

12.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

12.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

12.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

12.7 Continuing Calibration Verification 2 (CCV2): A check standard used to verify instrument calibration. CCV2 is analyzed at the time of instrument calibration and is usually the same concentration as the lowest standard or the reporting limit.

12.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

12.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and bias of a method in a given sample matrix.

12.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

12.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

12.12 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

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Table 1 QC Criteria

Quality Control	Acceptance Criteria	Corrective Action
Initial Calibration: r^2 = coefficient of determination, or: r = coefficient of correlation	≥0.990 ≥0.995	Rerun calibration standards, and/or prepare new calibration standards and recalibrate the instrument, or document why the data are acceptable.
Initial Calibration Verification standard (ICV)	± 10% of the standard's true value	Rerun standard, and/or prepare new standard, and/or recalibrate instrument, or document why the data are acceptable.
Continuing Calibration Verification standard (CCV), and Low-Level Standard (where applicable)	± 10% of the standard's true value ± 10% of the standard's true value	Rerun standard, and/or recalibrate instrument and reanalyze all samples run since the last acceptable CCV, or document why the data are acceptable.
Method blank (MB)	< 1/2RL	Reanalyze, and/or stop the run and determine the source of contamination, or document why the data are acceptable.
Blank Spike (BS or LSC)	± 10 to 20%, or as per method	Determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable.
MS/MSD	± 15 to 25%, or as per method.	Determine and correct the problem, reanalyze samples and MS/MSD, or document why data are acceptable
Sample Duplicate	± 15 to 20%, or as per method.	Determine and correct cause of the poor reproducibility

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Quality Systems Manual

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Laboratory Director	Technical Director	Quality Assurance Officer

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The Accutest Laboratories Southeast, Inc. (Accutest SE) Quality Assurance Program, detailed in this plan, has been designed to meet the quality program requirements of the National Environmental Laboratories Accreditation Conference (NELAC) and ISO Guide 25. The plan establishes the framework for documenting the requirements of the quality processes regularly practiced by the Laboratory. The Quality Assurance Officer is responsible for changes to the Quality Assurance Program, which are appended to the LQSM as they occur. The plan is reviewed annually for compliance purposes by the Laboratory Director and Technical Director and edited if necessary. Changes that are incorporated into the plan are summarized in the plan introduction. Changes to the plan are communicated to the general staff in a meeting conducted by the Quality Assurance Officer following the plan's approval.

The Accutest SE plan is supported by standard operating procedures (SOPs), which provide specific operational instructions on the execution of each quality element and assure that compliance with the requirements of the plan are achieved. Accutest SE employees are responsible for knowing the requirements of the SOPs and applying them in the daily execution of their duties. These documents are updated as changes occur and the staff is trained to apply the changes.

At Accutest, we believe that satisfying client requirements and providing a product that meets or exceeds the standards of the industry is the key to a good business relationship. However, client satisfaction cannot be guaranteed unless there is a system that assures the product consistently meets its design requirements and is adequately documented to assure that all procedural steps are executed and are traceable.

This plan has been designed to assure that this goal is consistently achieved and the Accutest product withstands the rigors of scrutiny that are routinely applied to analytical data and the processes that support its generation.

Summary of Changes
Accutest SE Quality System Manual – February and March 2005

Section	Description
1	Responsibilities for knowledge of QSM content added to policy
2	Modified Accutest Laboratories organizational chart
4	Job descriptions added for non-managerial staff, minimum qualifications statement added, Inorganics manager removed, replaced with WetChem ad Metals managers, Tech Support supervisor and Assistant Manager are joined together,
4	Minimum qualifications added, Training database added.
5	Title Clarification
6	Reference to paper forms is removed
7	Reference Standard traceability modification for thermometers and weights
8	Uncertainty added, Detection limit modified, Sources and References, Method validation added.
8	Amended Capabilities Table – Added Petroleum Methods for MO ORO, MO DRO
8	Amended Capabilities Table -removed 413.1, 418.1, Sulfite, Tetraethyl Lead,
9.1	Grammar and wording
9.3	Tracking through status change
9.4	Sample acceptance policy clarified
9.6	Subcontract procedure updated
9.7	Sample storage revised
9.8	Sample Log-in
9.9	Sample retrieval revised
9.10	Sample disposal
10	Added instrumentation
10.5	Linear Range clarified
12	Batch definition expanded, Blank contamination, Marginal Exceedances added, Post-digestion spike clarified, Method of standard additions removed, Data qualifiers added, Data package review revised.
14	Client services addition assistance appended
13	Corr. Action Database – Process changes and procedural modification revised
14	Resource evaluation revised.
15	Complaint documentation revised.
19	Health and Safety section added
Ap1	Workcell definition added

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1.0 QUALITY POLICY

1.1 *Accutest Mission*:

Accutest Laboratories provides analytical services to commercial and government clients in support of environmental monitoring and remedial activities as requested. The Laboratory's mission is dedicated to providing reliable data that satisfies clients requirements as explained in the following: "Provide easy access, high quality, analytical support to commercial and government clients which meet or exceeds data quality objectives and provides them with the data needed to satisfy regulatory requirements and/or make confident decisions on the effectiveness of remedial activities."

These services are provided impartially and are not influenced by undue commercial or financial pressures, which might impact the staff's technical judgment. Coincidentally, Accutest does not engage in activities that endanger the trust in our independent judgment and integrity in relation to the testing activities performed.

1.2 *Policy Statement*:

The management and staff of Accutest Laboratories share the responsibility for product quality. Accordingly, Accutest's quality assurance program is designed to assure that all processes and procedures, which are components of environmental data production, meet established industry requirements, are adequately documented from a procedural and data traceability perspective, and are consistently executed by the staff. It also assures that analytical data of known quality, meeting the quality objectives of the analytical method in use and the data user's requirements, is consistently produced in the laboratory. This assurance enables the data user to make rational, confident, cost-effective decisions on the assessment and resolution of environmental issues.

The laboratory Quality System also provides the management staff with data quality and operational feedback information. This enables them to determine if the laboratory is achieving the established quality and operational standards, which are dictated by the client or established by regulation. The information provided to management, through the QA program, is used to assess operational performance from a quality perspective and to perform corrective action as necessary.

All employees of Accutest Laboratories participating in environmental testing receive quality system training and are responsible for knowing and complying with the system requirements. The entire staff shares Accutest's commitment to good professional practice.

Harry Behzadi, Ph.D.
Laboratory Director

2.0 ORGANIZATION

2.1 ***Organizational Entity***. Accutest Laboratories is a privately held, independent testing laboratory founded in 1956 and registered as a New Jersey Corporation. The laboratory is located in Dayton, New Jersey where it has conducted business since 1987. Satellite laboratories are maintained in Marlborough, Massachusetts; Orlando, Florida and Houston, Texas.

2.2 ***Management Responsibilities***

Requirement: Each laboratory facility will have an established chain of command. The duties and responsibilities of the management staff are linked to the President/CEO of Accutest Laboratories who establishes the agenda for all company activities.

President/CEO*.* Primarily responsible for all operations and business activities. Delegates authority to laboratory directors, general managers, and quality assurance director to conduct day-to-day operations and execute quality assurance duties. Each of the three operational entities (New Jersey, Florida and Massachusetts) reports to the President/CEO.

Corporate Quality Assurance Director*.* Responsible for design, oversight, and facilitation of all quality assurance activities established by the Quality Program. Directly reports to the President/CEO.

Vice President Operations/Laboratory Director*.* There is Laboratory Director assigned to each of the operational entities: New Jersey, Massachusetts and Florida. Executes day-to-day responsibility for laboratory operations including technical aspects of production activities and associated logistical procedures. Directly reports to the President/CEO.

Quality Assurance Officer *(on location).* Responsible for oversight, implementation and facilitation of all quality assurance activities established by the Quality Program. Directly reports to the Laboratory Director. Also exchanges information with and submits laboratory performance data (PE scores, audit reports, accreditation changes, etc.) to Corporate QA Director.

Technical Director. Responsible for oversight and implementation of technical aspects of production activities in the environmental testing laboratory. In the event of prolonged absence Quality Assurance Officer is designated a deputy Technical Director, and Technical Director is designated a deputy Quality Assurance Officer.

Department Managers*.* Executes day-to-day responsibility for specific laboratory areas including technical aspects of production activities and associated logistical procedures. Directly report to the Laboratory Director.

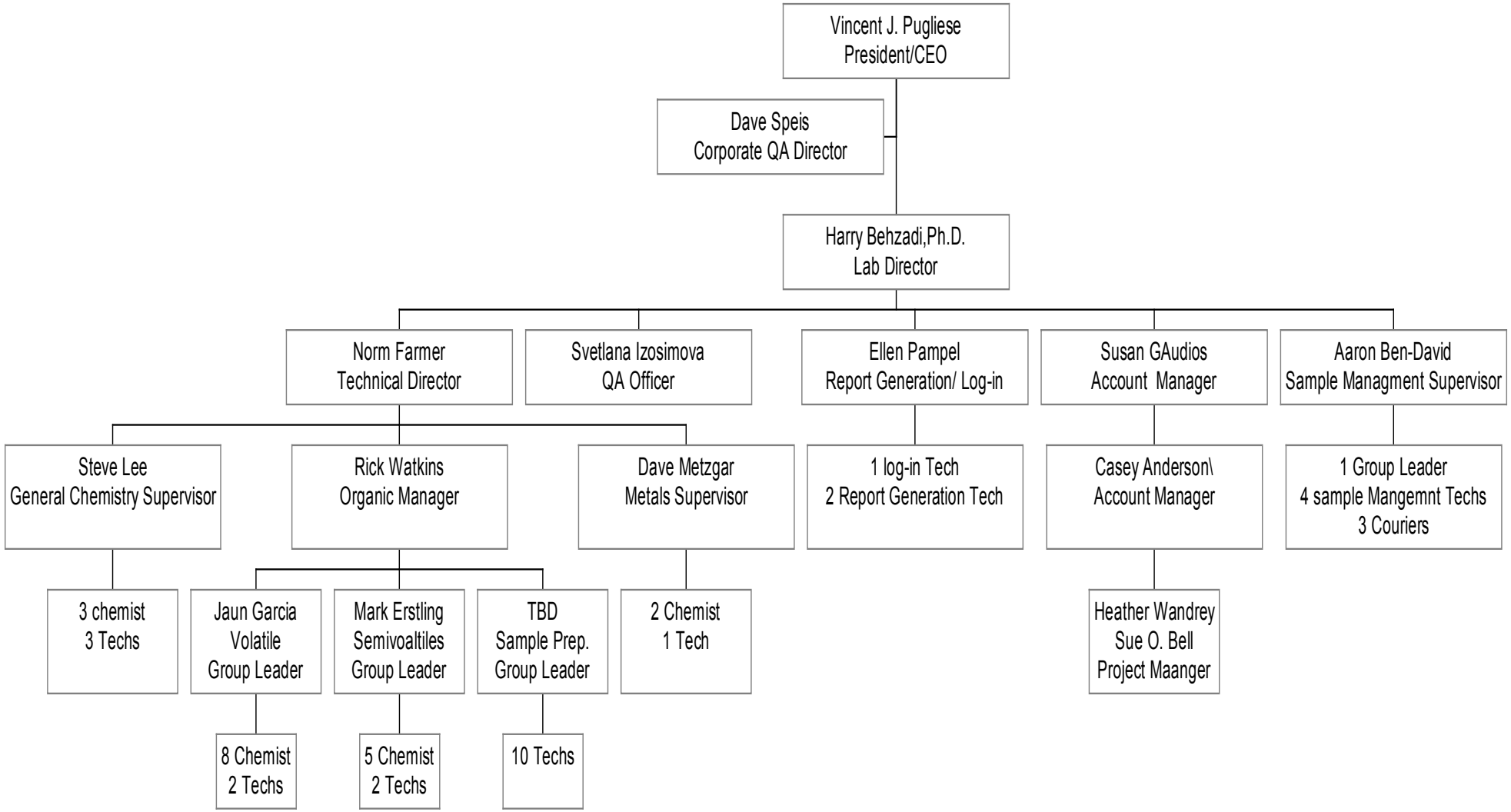
Section Supervisors. Executes day-to-day responsibility for specific laboratory units including technical aspects of production activities and associated logistical procedures. Directly report to the Department Manager.

2.3 ***Chain of Command***

The responsibility for managing all aspects of the Company's operation is delegated to specific individuals, who have been assigned the authority to act in the absence of the senior staff. These individuals are identified in the following Chain of Command:

Harry Behzadi, Ph.D., Laboratory Director
Norm Farmer, Technical Director
Rick Watkins, Organics Supervisor (Operations and IT) ***and*** Heather Wandrey, Project Manager (Client Services)

Accutest Laboratories Southeast Organizational Chart



3.0 QUALITY RESPONSIBILITIES OF THE MANAGEMENT TEAM

3.1 ***Requirement*:** Each member of the management team has a defined responsibility for the Quality Program. Program implementation and operation is designated as an operational management responsibility. Program design and implementation is designated as a Quality Assurance Responsibility.

President/CEO*:* Primary responsibility for all quality activities. Delegates program responsibility to the Quality Assurance Director. Serves as the primary alternate in the absence of the Quality Assurance Director. Has the ultimate responsibility for implementation of the Quality Program.

Vice President Operations/Laboratory Director*.* Responsible for implementing and operating the Quality Program in all laboratory areas. Responsible for the design and implementation of corrective action for defective processes. Has the authority to delegate Quality Program implementation responsibilities.

Corporate Quality Assurance Director*.* Responsible for design, implementation support, training, and monitoring of the quality system. Identifies product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

Quality Assurance Officer *(on location).* Responsible for design support, implementation support, and monitoring support of the quality system. Training personnel in various aspects of quality system. Conducts audits and product reviews to identify product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

Technical Director. Responsible for oversight and implementation of technical aspects of Quality System as they are integrated into method applications and employed to assess analytical controls on daily basis. The Technical Director reviews and acknowledges the technical feasibility of proposed quality system involving technical applications.

Department Managers*.* Responsible for applying the requirements of the Quality Program in their section and assuring subordinate supervisors and staff apply all program requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Group Leaders*.* Responsible for applying the requirements of the Quality Program to their operation and assuring the staff applies all program requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Bench Analysts. Responsible for applying the requirements of the Quality Program to the analyses they perform, evaluating QC data and initiating corrective action for quality control deficiencies within their control. Implements global corrective action as directed by superiors.

3.2 ***Program Authority***:

Authority for program implementation on corporate level originates with the President/CEO who bears ultimate responsibility for program design, implementation, and enforcement of requirements. This authority and responsibility is delegated to the Director of Quality Assurance who performs quality functions independently without the encumbrances or biases created by operational or production responsibilities to ensure an honest, independent assessment of quality issues.

Laboratory Director and Quality Assurance Officer mirror this authority on location.

3.3 ***Data Integrity Policy***:

The Accutest Data Integrity Policy reflects a comprehensive, systematic approach for assuring that data produced by the laboratory accurately reflects the outcome of the tests performed on field samples and has been produced in a bias free environment by ethical professionals. The policy includes a commitment to technical ethics, staff training in ethics and data integrity, an individual attestation to data integrity and procedures for evaluating data integrity. Senior management assumes the responsibility for assuring compliance with all technical ethics elements and operation of all data integrity procedures. The staff is responsible for compliance with the ethical code of conduct and for practicing data integrity procedures.

The Accutest Data Integrity Policy is as follows:

"Accutest Laboratories is committed to producing data that meets the data integrity requirements of the environmental regulatory community. This commitment is demonstrated through the application of a comprehensive data integrity program that includes ethics and data integrity training, data integrity evaluation procedures, staff participation and management oversight. Adherence to the specifications of the program assures that data provided to our clients is of the highest possible integrity and can be used for decision making processes with high confidence."

Data Integrity Responsibilities

***Management*.** Senior management retains oversight responsibility for the data integrity program and retains ultimate responsibility for execution of the data integrity program elements. Senior management is responsible for providing the resources required to conduct ethics training and operate data integrity evaluation procedures. They also include responsibility for creating an environment of trust among the staff and being the lead advocate for promoting the data integrity policy and the importance of technical ethics.

***Staff*.** The staff is responsible for adhering to the company ethics policy as they perform their duties and responsibilities associated with sample analysis and reporting. By executing this responsibility, data produced by Accutest Laboratories retains its high integrity characteristics and withstands the rigors of all data integrity checks.

The staff is also responsible for adhering to all laboratory requirements pertaining to manual data edits, data transcription and data traceability. These include the application of approved manual peak integration and documentation procedures. It also includes establishing traceability for all manual results calculations and data edits.

***Ethics Statement*.** The Accutest ethics statement reflects the standards that are expected for businesses that provide environmental services to regulated entities and regulatory agencies on a commercial basis. The Ethics Policy is comprised of key elements that are essential to organizations that perform chemical analysis for a fee. As such, it focuses on elements related to personal, technical and business activities.

Accutest Laboratories provides analytical chemistry services on environmental matters to the regulated community. The data the company produces provides the foundation for determining the risk presented by a chemical pollutant to human health and the environment. The environmental industry is dependent upon the accurate portrayal of environmental chemistry data. This process is reliant upon a high level of scientific and personal ethics.

It is essential to the Company that each employee understands the ethical and quality standards required to work in this industry. Accordingly, Accutest has adopted a code of ethics, which each employee is expected to adhere to as follows:

- Perform chemical and microbiological analysis using accepted scientific practices and principles.
- Perform tasks in an honest, principled and incorruptible manner inspiring peers & subordinates.
- Maintain professional integrity as an individual.
- Provide services in a confidential, honest, and forthright manner.

- Produce results that are accurate and defensible.
- Report data without any considerations of self-interest.
- Comply with all pertinent laws and regulations associated with assigned tasks and responsibilities.

Data Integrity Procedures.

Four key elements comprise the Accutest data integrity system. Procedures have been implemented for conducting data integrity training and for documenting that employees conform to the Accutest Data Integrity and Ethics policy.

The data integrity program consists of routine data integrity evaluation and documentation procedures to periodically monitor and document data integrity. These procedures are documented as SOPs. SOPs are approved and reviewed annually following the procedures employed for all Accutest SOPs. Documentation associated with data integrity evaluations is maintained on file and is available for review.

Data Integrity Training, .Accutest employees receive technical ethics training during new employee orientation. Employees are also required to attend annual ethics refreshment training and sign an ethical conduct agreement annually, which verifies their understanding of Accutest's ethics policy and their ethical responsibilities. The agreement is refreshed annually and appended to each individual's training file.

The training focuses on the reasons for technical ethic training, explains the impact of data fraud on human health and the environment, and illustrates the consequences of criminal fraud on businesses and individual careers. Accutest's ethics policy and code of ethics are reviewed and explained for each new employee.

Training on data integrity procedures are conducted by individual departments for groups involved in data operations. These include procedures for manual chromatographic peak integration, traceability for manual calculations and data transcription.

Data Integrity Training Documentation. Records of all data integrity training are maintained in individual training folders. Attendance at all training sessions is documented and maintained in the training archive.

Accutest Data Integrity and Ethical Conduct Agreement. All employees are required to sign a Data Integrity and Ethical Conduct Agreement annually. This document is archived in individual training files, which are retained for duration of employment.

The Data Integrity and Ethical Conduct Agreement is as follows**:**

I. *I understand the high ethical standards required of me with regard to the duties I perform and the data I report in connection with my employment at Accutest Laboratories.*

II. *I have received formal instruction on the code of ethics that has been adapted by Accutest Laboratories and agree to comply with these requirements.*

III. *I have received formal instruction on the elements of Accutest Laboratories' Data Integrity Policy and have been informed of the following specific procedures:*

   a. *Routine data integrity monitoring is conducted on sample data, which may include an evaluation of the data I produce,*

   b. *Formal procedures for the confidential reporting of data integrity issues are available, which can be used by any employee,*

   c. *A data integrity investigation is conducted when data issues are identified that may negatively impact data integrity.*

IV. *I am aware that data fraud is a punishable crime that may include fines and/or imprisonment upon conviction.*

V. *I also agree to the following:*

   a. *I shall not intentionally report data values, which are not the actual values observed or measured.*

   b. *I shall not intentionally modify data values unless the modification can be technically justified through a measurable analytical process.*

   c. *I shall not intentionally report dates and times of data analysis that are not the true and actual times the data analysis was conducted.*

   d. *I shall not condone any accidental or intentional reporting of inauthentic data by other employees and immediately report it's occurrence to my superiors.*

   e. *I shall immediately report any accidental reporting of inauthentic data by myself to my superiors.*

Data Integrity Monitoring. Several documented procedures are employed for performing data integrity monitoring. These include regular data review procedures by supervisory and management staff (Section 12.7), supervisory review and approval of manual integrations and periodic reviews of GALP audit trails from the LIMS and all computer controlled analysis.

Data Review. All data produced by the laboratory undergoes several levels of review, which includes two levels of management review. Detected data anomalies that appear to be related to data integrity issues are isolated for further investigation. The investigation is conducted following the procedures described in this section.

Manual Peak Integration Review and Approval. Routine data review procedures for all chromatographic processes includes a review of all manual chromatographic peak integrations. This review is performed by the management staff and consists of a review of the machine integration compared to the manual integration. Manual integrations, which have been performed in accordance with Accutest's manual peak integration procedures are approved for further processing and release. Manual integrations which are not performed to Accutest's specifications are set aside for corrective action, which may include analyst retraining or further investigation as necessary.

GALP Audit Trail Review. Good Automated Laboratory Practice (GALP) audits are comprehensive data package audits that include a review of raw data, process logbooks, processed data reports and GALP audit trails from individual instruments and LIMS. GALP audit trails, which record all electronic data activities, are available for the majority of computerized methodology and the laboratory information management system (LIMS). These audit trails are periodically reviewed to determine if interventions performed by technical staff constitute an appropriate action. The review is performed on a recently completed job and includes interviews with the staff who performed the analysis. Findings indicative of inappropriate interventions or data integrity issues are investigated to determine the cause and the extent of the anomaly.

Confidential Reporting Of Data Integrity Issues. Data integrity concerns may be raised by any individual to their supervisor. Employees with data integrity concerns should always discuss those concerns with their immediate supervisors as a first step unless the employee is concerned with the confidentiality of disclosing data integrity issues or is uncomfortable discussing the issue with their immediate supervisors. The supervisor makes an initial assessment of the situation to determine if the concern is related to a data integrity violation. Those issues that appear to be violations are documented by the supervisor and referred to the Director of Quality Assurance for investigation.

Documented procedures for the confidential reporting of data integrity issues in the laboratory are part of the data integrity policy. These procedures assure that laboratory staff can privately discuss ethical issues or report items of ethical concern without fears of repercussions with senior staff.

Employees with data integrity concerns that they consider to be confidential are directed to the Corporate Human Resources Manager in Dayton, New Jersey. The HR Manager acts as a conduit to arrange a private discussion between the employee and the Corporate QA Director or a local QA Officer.

During the employee - QA discussion, the QA representative evaluates the situation presented by the employee to determine if the issue is a data integrity concern or a legitimate practice. If the practice is legitimate, the QA representative clarifies the process for the employee to assure understanding. If the situation appears to be a data integrity concern, the QA representative initiates a Data Integrity Investigation following the procedures specified in SOP EQA059.

Data Integrity Investigations. Follow-up investigations are conducted for all reported instances of ethical concern related to data integrity. Investigations are performed in a confidential manner by senior management according to a documented procedure. The outcome of the investigation is documented and reported to the company president who has the ultimate responsibility for determining the final course of action in the matter. Investigation documentation includes corrective action records, client notification information and disciplinary action outcomes, which is archived for a period of five years.

The investigations are conducted by the senior staff and supervisory personnel from the affected area. The investigation team includes the Laboratory Director and the Quality Assurance Director. Investigations are conducted in a confidential manner until it is completed and resolved.

The investigation includes a review of the primary information in question by the investigations team. The team performs a review of associated data and similar historical data to determine if patterns exist. Interviews are conducted with key staff to determine the reasons for the observed practices.

Following data compilation, the investigations team reviews all information to formulate a consensus conclusion. The investigation results are documented along with the recommended course of action.

Corrective Action, Client Notification & Discipline. Investigations that reveal systematic data integrity issues will go through corrective action for resolution and disposition (Section 13). If the investigation indicates that an impact to data has occurred and the defective data has been released to clients, client notification procedures will be initiated following the steps in Section 17.6.

In all cases of data integrity violations, some level of disciplinary action will be conducted on the responsible individual. The level of discipline will be consistent with the violation and may range from retraining and/or verbal reprimand to termination.

4.0 JOB DESCRIPTIONS OF KEY STAFF

4.1 ***Requirement*:** Descriptions of key positions within the organization must defined to ensure that clients and staff understand duties and the responsibilities of the management staff and the reporting relationships between positions.

President/Chief Executive Officer. Responsible for all laboratory operations and business activities. Establishes the company mission and objectives in response to business needs. Direct supervision of the Vice President of Operations, each laboratory director, client services, management information systems, and quality assurance.

Vice President, Operations/Laboratory Director. Reports to the company president. Establishes laboratory operations strategy. Direct supervision of organic chemistry, inorganic chemistry, field services, and sample management..

Director, Quality Assurance. Reports to the company president. Establishes the company quality agenda, develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities monitors the quality system and provides quality system feedback to management to be used for process improvement.

Chief Information Officer (CIO) Reports to the company president. Develops the MIS software and hardware agenda. Provides system strategies to compliment company objectives. Maintains all software and hardware used for data handling.

Client Services, Sales, Account Manager(s). Reports to the company president. Establishes and maintains communications between clients and the laboratory pertaining to client requirements which are related to sample analysis and data deliverables. Initiates client orders and supervises sample login operations.

Quality Assurance Officer *(on location)*. Reports to the Laboratory Director. Develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities, monitors the quality system, and provides quality system feedback to management to be used for process improvement. In the event of prolonged absence QAO also designated a Deputy Technical Director, unless otherwise specified by internal memo from Laboratory Director.

Manager Client Services *(on location)*. Reports to the Laboratory Director. Establishes and maintains communications between clients and the laboratory pertaining to client requirements which are related to sample analysis and data deliverables. Initiates client orders and supervises sample login operations.

Manager, Organics. Reports to the laboratory director. Directs the operations of the organics group, consisting of organics preparation and instrumental analysis. Establishes daily work schedule. Supervises method implementation, application, and

data production. Responsible for following Quality Program requirements. Maintains laboratory instrumentation in an operable condition.

Manager, Sample Management. Reports to the laboratory director. Develops, maintains and executes all procedures required for receipt of samples, verification of preservation, and chain of custody documentation. Responsible for maintaining and documenting secure storage, delivery of samples to laboratory units on request, and disposal following completion of all analytical procedures. *Manager of Sample Management and Manager of Field Services are combined in Accutest-SE location.*

Group Leader, Wet Chemistry. Reports to the laboratory director. Directs the operations of the wet chemistry group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production Supervises the analysis of samples for wet chemistry parameters using valid, documented methodology. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements. . Responsible for following Quality Program requirements.

Group Leader, Metals. Reports to the laboratory director. Directs the operations of the metals group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples for metallic elements using valid, documented methodology. Documents all procedures and data production activities. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements. . Responsible for following Quality Program requirements

Group Leader, Organic Preparation. Reports to the organics manager. Executes the daily sample preparation schedule. Performs the extract of multi-media samples for organic constituents using valid, documented methodology. Prepares documentation for extracted samples. Assumes custody until transfer for analysis.

Volatile and Semivolatie Group leaders, Organics. Reports to the organic manager. Oversees all instrument maintenance and new equipment installation. Conducts method development and implementation tasks. Expedites the analysis of samples and sample extracts. Executes daily analysis schedule. Supervises the analysis of samples for organic parameters using valid, documented methodology. Documents all data and data production activities. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements.

4.2 ***Employee Screening, Orientation, and Training***.

All potential laboratory employees are screened and interviewed by human resources and technical staff prior to their hire. The pre-screen process includes a review of their qualifications including education, training and work experience to verify that they have adequate skills to perform the tasks of the job. Minimum qualifications for non-technical personnel require High School diploma (couriers also shall posses clean driving record), technical personnel must also demonstrate basic laboratory

experience, such as balance and syringe use, aseptic practices, etc. College-level science coursework is favored.

Newly hired employees receive orientation training beginning the first day of employment by the Company. Orientation training consists of initial health and safety training and a detailed review of the personal protection policies, technical ethics training and data integrity procedures and quality assurance program training (including Company's goals, objectives, mission, and vision).

All technical staff receives training to develop and demonstrate proficiency for the methods they perform. New analysts work under supervision until the supervisory staff is satisfied that a thorough understanding of the method is apparent. Organics/Inorganics analysts are required to demonstrate method proficiency through a precision and accuracy study. Data from the study is compared to method acceptance limits. If the data is unacceptable, additional training is required. The analyst must also demonstrate the ability to produce acceptable data through the analysis of an independently prepared proficiency sample.

Proficiency is demonstrated annually. Data from initial and continuing proficiency demonstration is archived in the individual's training folder. In the instance where analyte can not be spiked in the clean matrix, such as TSS or pH, the results of an external Performance Evaluation (PE) sample may be used to document analyst's proficiency.

Minimum training required for administrative staff consists of laboratory safety and ethical conduct.

4.3 ***Training Documentation***. The QA Officer prepares a training file for every new employee. All information related to qualifications, experience, external training courses, and education are placed into the file. Verification documentation for orientation, health & safety, quality assurance, and ethics training is also included in the file.

Additional training documentation is added to the file as it occurs. This includes data for initial and continuing demonstrations of proficiency, performance evaluation study data and notes and attendance lists from group training sessions.

The Quality Assurance Department maintains the employee training database. This database is a comprehensive inventory of training documentation for each individual employee. The database enables supervisors to obtain current status information on training data for individual employees on a job specific basis. It also enables the management staff to identify training documentation in need of completion.

Employee specific database records are created by human resources on the date of hire. Data base fields for job specific requirements such as SOP documentation of understanding and annual demonstration of analytical capability are automatically generated when the supervisor assigns a job responsibility. Employees acknowledge that their SOP responsibilities have been satisfied using a secure electronic process,

which updates the database record. Reports are produced which summarize the qualifications of individual employees or departments.

5.0 SIGNATORY APPROVALS

Requirement: Procedures are required for establishing the traceability of data and documents. The procedure consists of a signature hierarchy, indicating levels of authorization for signature approvals of data and information within the organization. Signature authority is granted for approval of specific actions based on positional hierarchy within the organization and knowledge of the operation that requires signature approval. A log of signatures and initials of all employees is maintained for cross-referencing purposes.

5.1 *Signature Hierarchy*.

President/Chief Executive Officer. Authorization for contracts and binding agreements with outside parties. Approval of final reports, quality assurance policy, SOPs, project specific QAPs, data review and approval in lieu of technical managers. Contract signature authority resides with Company Officers only, which include the President/CEO, CFO and VP Administration.

Vice President, Operations/Laboratory Director. Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. Technical policy.

Technical Director (on location): Approval of final reports and quality assurance policy in the absence of the Laboratory Director. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. Technical policy review. In the event of prolonged absence Technical Director also designated a Deputy QAO, unless otherwise specified by internal memo from Laboratory Director.

Director, Quality Assurance. Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers.

Quality Assurance Officer (on location). Approval of final reports and quality assurance policy in the absence of the Laboratory Director. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. In the event of prolonged absence QAO also designated a Deputy Technical Director, unless otherwise specified by internal memo from Laboratory Director.

Director, Management Information Systems (MIS). Department specific supplies purchase. MIS policy.

Manager, Sample Management. Initiation of laboratory sample custody and acceptance of all samples. Approval of department policies and procedures. Department specific supplies purchase. Waste manifesting and disposal.

Manager Client Services. QAP and sampling and analysis plan approval. Project specific contracts, pricing, and price modification agreements. Approval and acceptance of incoming work, Client services policy.

Managers, Technical Departments. Methodology and department specific QAPs. Data review and approval, department specific supplies purchase. Technical approval of SOPs.

Team Leaders, Technical Departments. Data review approval, purchasing of expendable supplies.

5.2 ***Signature Requirements***. All laboratory activities related to sample custody and generation or release of data must be approved using either initials or signatures. The individual, who applies his signature or initial to an activity or document, is authorized to do so within the limits assigned to them by their supervisor. All signatures and initials must be applied in a readable format that can be cross-referenced to the signatures and initials log if necessary.

5.3 ***Signature and Initials Log****. The QA Officer maintains a signature and initials log. New Employee signatures and initials are appended to the log on the first day of employment. Signature of individuals no longer employed by the company are retained***.**

6.0 DOCUMENTATION

Requirement: Document control policies have been established which specify that any document used as an information source or for recording analytical or quality control information must be managed using defined document control procedures. Accordingly, policies and procedures required for the control, protection, and storage of any information related to the production of analytical data and the operation of the quality system to assure its integrity and traceability have been established and implemented in the laboratory. The system contains sufficient controls for managing, archiving and reconstructing all process steps which contributed to the generation of an analytical test result. Using this system, an audit trail for reported data can be produced, establishing complete traceability for the result.

6.1 Administrative Records. Administrative (non-analytical) records are managed by the quality assurance department. These records consist of electronic documents which are retained in a limited access electronic directory or paper documents, which are released to the technical staff upon specific request.

Form Generation & Control. The quality assurance group approves all forms used as either stand-alone documents or in logbooks to ensure their traceability. Forms are generated as computer files only and maintained in a limited access master directory. Approved forms must display the date of current revision and initials of person who revised the form. Modifications to existing forms are approved by QA, obsolete forms moved to archive directory and retained for five years.

New forms must include Accutest SE identification and appropriate spaces for signatures of approvals and dates. Further design specifications are the responsibility of the originating department.

Technical staff is required to complete all forms to the maximum extent possible. If information for a specific item is unavailable, the analyst is required to cross out the information block. The staff is also required to cross out the uncompleted portions of a logbook or logbook form if the day's analysis does not fill the entire page of the form.

Logbook Control. All laboratory logbooks are controlled documents that are comprised of approved forms used to document specific processes. Logbook control is maintained by quality assurance.

New logs are numbered and issued to a specific individual who is assigned responsibility for the log. Supervisor performs periodical review of the logbooks. Old logs are returned to QA for entry into the document archive system where they are retained for minimum of five (5) years. Laboratory staff may hold a maximum of two consecutively dated logbooks of the same type in the laboratory, not including the most recently issued book to simplify review of recently completed analysis.

Controlled Documents. Key laboratory documents are designated for controlled document status to assure that identities of individuals receiving copies and the

number of copies that have been distributed are known. Controlled status simplifies document updates and retrieval of outdated documents. Control is maintained through a document numbering procedure and document control logbook designating the individual receiving the controlled document. Document control is also maintained by pre-designating the numbers of official copies of documents that are placed into circulation within the laboratory.

Quality Systems Manual (QSM). All QSMs are assigned a number prior to distribution. The number, date of distribution, and identity of the individual receiving the document are recorded in the document control logbook. The numbering system is continuous.

Standard Operating Procedures (SOPs). SOPs are maintained by pre-designating the numbers of official copies of documents that are placed into circulation within the laboratory. Official documents are printed and placed into the appropriate laboratory section as follows:

Sample Management: One or two copies for the sample management file, depending on the area.
Organics Laboratories: One for the affected laboratory area.
Inorganics Laboratories: One for the affected laboratory area.

The original, signed copy of the SOP is maintained in the master SOP binder by the QA staff.

Documents are controlled using an "Official Copy" stamp in red ink. Additional copies could be issued to individuals for training purposes. Distribution is documented on SOP cover page. Superceded copies collection is conducted accordingly to cover page distribution list.

6.2 ***Technical Records***. All records related to the analysis of samples and the production of an analytical result are archived in secure document storage or on electronic media and contain sufficient detail to produce an audit trail, which re-creates the analytical result. These records include information related to the original client request, bottle order, sample login and custody, storage, sample preparation, analysis, data review and data reporting.

Each department involved in this process maintains controlled documents, which enable them to maintain records of critical information relevant to their department's process.

6.3 ***Quality Assurance Directory.*** All Quality Assurance documentation and quality control limit data is stored in a restricted QA directory on the network server. The directory has been designated as read only. The QA staff, technical director and the laboratory director have write capability in this directory. Information on this directory is backed-up daily.

This directory contains all current and archived quality system manuals, SOPs, control limits, MDL studies, precision and accuracy data, internal and external audit reports, official forms, Health and Safety materials, PT scores, State Certifications and metrics calibration information.

6.4 ***Analytical Records***. All data related to the analysis of field samples are retained as either paper or electronic records that can be retrieved to compile a traceable audit trail for any reported result. All information is linked to the client job and sample number, which serves as a reference for all sample related information tracking.

Critical times in the life of the sample from collection through analysis to disposal are documented. This includes date and time of collection, receipt by the laboratory, preparation times and dates, analysis times and dates and data reporting information. Analysis times are calculated in hours for methods where holding time is specified in hours (≤72 hours).

Sample preparation information is recorded in a separate controlled logbook. It includes sample identification numbers, types of analysis, preparation and cleanup methods, sample weights and volumes, reagent lot numbers and volumes and any other information pertinent to the preparation procedure.

Information related to the identification of the instrument used for analysis is permanently attached to the electronic record. The record includes an electronic data file that indicates all instrument conditions employed for the analysis, including the type of analysis conducted. The analyst's identification is electronically attached to the record. The instrument tuning and calibration data is electronically linked to the sample or linked though paper logs, which were used in the documentation of the analysis. Quality control and performance criteria are permanently linked to the paper archive or electronic file.

Paper records for the identity, receipt, preparation and evaluation of all standards and reagents used in the analysis are documented in prepared records and maintained in controlled documents or files. Lot number information linking these materials to the analysis performed is recorded in the logbooks associated with the samples in which they were used.

Manual calculations or peak integrations that were performed during the data review are retained as paper or scanned documents and included as part of the electronic archive. Signatures for data review are retained on paper or as scanned versions of the paper record for the permanent electronic file.

6.5 ***Confidential Business Information (CBI).*** Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between Accutest and the receiving organization or

individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as Adobe Acrobat .PDF format only.

6.6 ***Software Change Documentation & Control.*** Changes to software are documented as text within the code of the program undergoing change. Documentation includes a description of the change, reason for change and the date the change was placed into effect. Documentation indicating the adequacy of the change is prepared following the evaluation by the user who requested the change.

6.7 ***Report and Data Archiving***. Accutest Laboratories maintains electronic image file copies of original reports in archive for a period of five (5) years. After five years, the files are automatically discarded unless contractual arrangements exist which dictate different requirements. Client specific data retention practices are employed for government organizations such as the Department of Defense Agencies and MA DEP that require a retention period of ten (10) years.

Accutest archives the original report (organized by job number) and the organic and inorganic support data. Organic support data is archived according to instrument batch numbers. All organics data is backed up to the tape or archive drive via Networker Backup software and/or AccuBack backup software. Data from the archive drive is then written to tape at periodic intervals.

Wet chemistry support data is archived by Analytical Batch (GN). Metals support data is archived by instrument batch (MA…). Metals digestion data is archived as digestion logbooks.

The reports generation group electronically scans completed reports and stores them by job number on the document server. The document server is backed up daily to a digital tape. Copies of these files remain active on the document server for easy review access. The digital tapes remain in secure storage for the remainder of the archive period.

6.8 ***Training***. The company maintains a training record for all employees that documents that they have received instruction on administrative and technical tasks that are required for the job they perform. Training records for individuals employed by the company are retained for a period of five years following their termination of employment.

Training File Origination. The Quality Assurance Officer (QAO) initiates training files. Quality Assurance officer retains the responsibility for the maintenance and tracking of all training related documentation in the file. The file is begun on the first day of employment. Information required for the file includes a copy of the individual's most current resume, detailing work experience and a copy of any college diplomas or transcript(s). Information added on the first day includes documentation of health and safety training and a signed Ethics and Data Integrity agreement. These two constitute minimal necessary training for Project Management and Administrative staff. Training documentation, training requirements, analyst proficiency information and other training related support documentation is tracked using a customized database

application. Database extracts provide an itemized listing of specific training requirements by job function. Training status summaries for individual analysts portray dates of completion for job specific training requirements.

Technical Training. The supervisor of each new employee is responsible for developing a training plan for each new employee. The supervisor updates the outline, adding signatures and dates as training elements are completed at regular frequency. Supporting documentation, such as precision and accuracy studies, which demonstrate analyst capability for a specific test, are added as completed. When analyte can not be spiked, such as pH or TSS, external PE sample is purchased and analyzed. Where no external PE sample is available, sample duplicates must be successfully analyzed. Method review records are retained where analysis of duplicates is not possible. Employees and supervisors verify documentation of understanding (DOU) for all assigned standard operating procedures in the training database. Certificates or diplomas for any off-site training are added to the file.

7.0 REFERENCE STANDARD TRACEABILITY

Requirement: Documented procedures, which establish traceability between any measured value and a national reference standard, must be in place in the laboratory. All metric measurements must be traceable to NIST reference weights or thermometers that are calibrated on a regular schedule. All chemicals used for calibration of a quantitative process must be traceable to an NIST reference that is documented by the vendor using a certificate of traceability. The laboratory maintains a documentation system that establishes the traceability links. The procedures for verifying and documenting traceability must be documented in standard operating procedures.

7.1 ***Traceability of Metric Measurements - Thermometers***. Accutest uses NIST thermometers to calibrate commercially purchased thermometers prior to their use in the laboratory. If necessary, thermometers are assigned correction factors that are determined during their calibration using an NIST thermometer as the standard. The correction factor is documented in a thermometer log and on a tag attached to the thermometer. The correction factor is applied to temperature measurements before recording the measurement in the temperature log. The NIST thermometer is checked for accuracy by an outside vendor minimum every five (5) years following the specifications for NIST thermometer calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005. Currently the NIST thermometer is verified by outside vendor on triennial basis due to contract-specific requirements. Certificate(s) of calibration are maintained on file with QAO.

7.2 ***Traceability of Metric Measurements – Calibration Weights***. Accutest uses calibrated weights, which are traceable to NIST standard weights to calibrate all balances used in the laboratory. Balances must be calibrated to specific tolerances within the intended use range of the balance. Calibration checks are required on each day of use. If the tolerance criteria are not achieved, corrective action specified in the balance calibration SOP must be applied before the balance can be used for laboratory measurements. All weights are recalibrated by outside vendor every five years following the specifications for weight calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005. Certificate(s) of calibration are maintained on file with QAO. Balances are inspected and maintained by professional service technicians annually. Certificate(s) of inspection are maintained with QAO.

7.3 ***Traceability of Chemical Standards and Reagents***. All chemicals and reagents, with the exception of bulk dry Na2SO4 and solvents purchased as reference standards for use in method calibration must establish traceability to NIST referenced material through a traceability certificate. Process links are established that enable a calibration standard solution to be traced to its NIST reference certificate. Solvents are being tested to verify their suitability for the analytical process.

7.4 ***Assignment Of Reagent and Standard Expiration Dates.*** Expiration date information for all purchased standards and reagents is provided to Accutest with all prepared standard solutions and unstable reagents as a condition of purchase. Neat materials and inorganic reagents are not required to be purchased with expiration dates. Certified prepared solutions are labeled with the expiration date provided by the manufacturer. In-house prepared solutions are assigned expiration dates that are consistent with the method that employs their use unless documented experience indicates that an alternate date can be applied. If alternate expiration dates are employed, their use is documented in the method SOP. Expiration dates for prepared inorganic reagents, which have not exhibited instability are established at two years form the date of preparation for tracking purposes. All containers shall be labeled with the date of preparation and expiration date clearly indicated.

The earliest expiration date is always the limiting date for assigning expiration dates to prepared solutions. Expiration dates that are later than the expiration date of any derivative solution or material are prohibited.

7.5 ***Documentation of Traceability***. Traceability information is documented in individual logbooks designated for the measurement process in use. The QA Officer maintains calibration documentation for metric references in pertinent folders and logbooks.

Balance calibration verification is documented in logbooks that are assigned to each balance. The individual conducting the calibration is required to initial and date all calibration activities. Any defects that occur during calibration are also documented along with the corrective action applied and a demonstration of return to control. Annual service reports and certificates retained on file with QA staff.

Temperature control is documented in logbooks assigned to the equipment being monitored. A calibrated thermometer is assigned to each individual item. Measurements are recorded along with date and initials of the individual conducting the measurement on a daily or as used basis. Corrective action, if required, is also documented including the demonstration of return to control.

Initial traceability of chemical standards and reagents is documented via a vendor-supplied certificate (see also 7.3) that includes lot number and expiration date information. Solutions prepared using the vendor supplied chemical standard are documented in logbooks assigned to specific analytical processes. Alternatively, documentation may be entered into the electronic standards and reagent tracking log The documentation includes links to the vendors lot number, an internal lot number, dates of preparation, and the preparer's initials. Supervisors conduct regular reviews of logbooks, which are verified using a word "rev'd", signature and date. QA Staff monitors the process and documents it in the same manner.

8.0 TEST PROCEDURES, METHOD REFERENCES, AND REGULATORY PROGRAMS

Requirements: The laboratory must use client specified or regulatory agency approved methods for the analysis of environmental samples. The laboratory maintains a list of active methods, which specifies the type of analysis performed, and cross-references the methods to applicable environmental regulation. Routine procedures used by the laboratory for the execution of a method must be documented in a standard operating procedure. Method performance and sensitivity must be demonstrated annually where required. Defined procedures for the use of method sensitivity for data reporting purposes must be established by the Director of Quality Assurance and used consistently for all data reporting purposes.

8.1 ***Method Selection.*** Accutest employs methods for environmental sample analysis that are consistent with the client's application, which are appropriate and applicable to the project objectives. Accutest informs the client if the method proposed is inappropriate or outdated and suggests alternative approaches.

Accutest employs documented, validated regulatory methods in the absence of a client specification and informs the client of the method selected. These methods are available to the client and other parties as determined by the client. Documented and validated in-house methods may be applied if they are appropriate to the project. The client is informed of the method selection.

8.2 ***Method Validation***. Standard methods from regulatory sources are primarily used for all analysis. Standard methods do not require validation by the laboratory. Non-standard, in-house methods are validated prior to use. Validation is also performed for standard methods applied outside their intended scope of use. Validation is dependent upon the method application and may include analysis of quality control samples to develop precision and accuracy information for the intended use. A final method validation report is generated, which includes all data in the validation study. A statement of adequacy and/or equivalency is included in the report. A copy of the report is archived in the quality assurance directory of the company server.

Non-standard methods are validated prior to use. This includes the validation of modified standard methods to demonstrate comparability with existing methods. Demonstrations and validations are performed and documented prior to incorporating technological enhancements and non-standard methods into existing laboratory methods used for general applications. The demonstration includes method specific requirements for assuring that significant performance differences do not occur when the enhancement is incorporated into the method. Validation is dependent upon method application and may include the analysis of quality control samples to develop precision and accuracy information for intended use.

The study procedures and specifications for demonstrating validation include comparable method sensitivity, calibration response, method precision, method accuracy and field sample consistency for several classes of analytical methods are

detailed in this document. These procedures and specifications may vary depending upon the method and the modification.

8.3 ***Standard Operating Procedures***. Standard operating procedures (SOP) are prepared for routine methods executed by the laboratory and processes related to sample or data handling. The procedures describe the process steps in sufficient detail to enable an individual, who is unfamiliar with the procedure to execute it successfully. SOPs are reviewed annually and edited if necessary. SOPs can be edited on a more frequent basis if systematic errors dictate a need for process change or the originating regulatory agency promulgates a new version of the method. Procedural modifications are indicated using a revision number. SOPs are available for client review at the Accutest facility upon request.

8.4 ***Method Detection Limit Determination***. Annual method detection limit (MDL) studies are performed as appropriate for routine methods used in the laboratory. MDL studies are also performed when there is a change to the method that affects how the method is performed or when an instrumentation change that impacts sensitivity occurs. The procedure used for determining MDLs is described in 40 CFR, Part 136, Appendix B. Studies are performed for each method on water, soil and air matrices for every instrument that is used to perform the method. MDLs are established at the instrument level. The highest MDL of the pooled instrument data is used to establish a laboratory MDL. MDLs are experimentally verified through the analysis of spiked quality control samples at 2-4 times the concentration of the experimental MDL. The verification is performed on every instrument used to perform the analysis. The quality assurance staff manages the annual MDL determination process and is responsible for retaining MDL data on file. Approved MDLs are appended to the LIMS and used for data reporting purposes.

8.5 ***Method Reporting Limit.*** The method reporting limit is established at the lowest concentration calibration standard in the calibration curve. The low calibration standard is selected by department managers as the lowest concentration standard that can be used while continuing to meet the calibration linearity criteria of the method being used. The validity of the Method Reporting Limits is confirmed via analysis of a spiked quality control sample at the Method reporting limit concentration.

By definition, detected analytes at concentrations below the low calibration standard cannot be accurately quantitated and must be qualified accordingly.

8.6 ***Reporting of Quantitative Data.*** Analytical data for all methods is reported without qualification to the reporting limit established for each method. Data may be reported to MDL depending upon the client's requirements provided that all qualitative identification criteria for the parameter have been satisfied. All parameters reported at concentrations between the reporting limit and MDL are qualified as an estimated concentration.

Measured concentrations of detected analytes that exceed the upper limit of the calibration range are either diluted into the range and reanalyzed or qualified as an estimated value. The only exception to this applies to ICP and ICP/MS analysis, which

can be reported to the upper limit of the experimentally determined linear range without qualification.

8.7 ***Estimated Uncertainty.*** A statement of the estimated uncertainty of an analytical measurement accompanies the test result when required. Estimated uncertainty is derived from the performance limits established for spiked samples of similar matrices. The degree of uncertainty is derived from the negative or positive bias for spiked samples accompanying a specific parameter. When the uncertainty estimate is applied to a measured value, the possible quantitative range for that specific parameter at that measured concentration is defined. Well recognized regulatory methods that specify values for the major sources of uncertainty and specify the data reporting format do not require a further estimate of uncertainty.

8.8 ***Precision and Accuracy Studies***. Annual precision and accuracy (P&A) studies, which demonstrate the laboratories ability to generate acceptable date, are performed for all routine methods used in the laboratory. The procedure used for generating organic P&A data is referenced in the majority of the regulatory methodology in use. The procedure requires quadruplicate analysis of a sample spiked with target analytes at a concentration in the working range of the method. This data may be compiled from a series of existing blank spikes or laboratory control samples. Accuracy (percent recovery) of the replicate analysis is averaged and compared to established method performance limits. Values within method limits indicate an acceptable performance demonstration. (See also Sec 4, Training, Demonstration of capability)

8.9 ***Method Sources, References and Update Mechanism.*** The Quality Assurance Staff maintains a list of active methods used for the analysis of samples. This list includes valid method references such as EPA, American Society of Testing and Materials (ASTM) or Standard Methods designations and the current version and version date.

Updated versions of approved reference methodology are placed into use as changes occur. The Quality Assurance Director informs operations management of changes in method versions as they occur. The operations management staff selects an implementation date. The operations staff is responsible for completing all method requirements prior to the implementation date. This includes modification to SOPs, completion of MDL and precision and accuracy studies and staff training. Documentation of these activities is provided to the QA staff who retains this information on file. The updated method is placed into service on the implementation date and the old version is de-activated.

Multiple versions of selected methods may remain in use to satisfy client specific needs. In these situations, the default method version becomes the most recent version. Client specific needs are communicated to the laboratory staff using method specific analytical codes method, which clearly depict the version to be used. The old method version is maintained as an active method until the specified client no longer requires the use of the older version.

Accutest will not use methodology that represents significant departures from the reference method unless specifically directed by the client. In cases where clients

direct the laboratory to use a method modification that represents a significant departure from the reference method, the request will be documented in the project file. The LQSM lists active methods used for the analysis of samples in Table 8.1. This list includes valid method references from sources such as USEPA, ASTM or Standard Methods designations and the current version and version date.

8.10 ***Analytical Capabilities***. Table 8.1 provides a detailed listing of the methodology employed for the analysis of test samples.

Table 8.1 – Analytical Capabilities and Method References

Method Type	Method Number	Regulatory Program
Organics – GC/MS:		
Volatile Organics	EPA 624	Clean Water Act
Semi-Volatile Organics	EPA 625	Clean Water Act
Volatile Organics	SW846 – 8260B	RCRA
Semi-Volatile Organics	SW846 – 8270C	RCRA
Organics – GC:		
EDB and DBCP – DW	EPA 504.1	Safe Drinking Water Act
Purgeable Halocarbons	EPA 601	Clean Water Act
Purgeable Aromatics	EPA 602	Clean Water Act
Chlorinated Pesticides & PCBs	EPA 608	Clean Water Act
Poly-Aromatic Hydrocarbons	EPA 610	Clean Water Act
Gasoline Range Organics	SW-846 – 8015B	RCRA
Diesel Range Organics	SW-846 – 8015B	RCRA
Alcohols	SW-846 – 8015B	RCRA
Dissolved Gases	RSK SOP 147-175	RCRA
Volatile Aromatic/Halocarbons	SW-846 – 8021B	RCRA
Organochlorine Pesticides	SW-846 – 8081A	RCRA
Polychlorinated Biphenyls	SW-846 – 8082	RCRA
Nitroaromatics	SW-846 – 8091	RCRA
Chlorinated Herbicides	SW-846 – 8151	RCRA
Total Petroleum Hydrocarbons	FLPRO	RCRA
Tennessee EPH	TN-EPH	RCRA
Tennessee GRO	TN-GRO	RCRA
Wisconsin DRO	WI-DRO	RCRA
Petroleum Hydrocarbons	Iowa OA-1	RCRA
Petroleum Hydrocarbons	Iowa OA-2	RCRA
Volatile Petro. Hydrocarbons	Massachusetts VPH	MCP
Volatile Petro. Hydrocarbons	Missouri Gasoline Range Organics	RCRA
Extractable Hydrocarbons	Missouri Diesel Range Organics	RCRA
Extractable Hydrocarbons	Missouri Oil Range Organic	RCRA
Extractable Petro. Hydrocarbons	Massachusetts EPH	MCP
Polynuclear-Aromatic Hydrocarbons	SW-846 – 8310	RCRA
Explosives	SW-846 – 8330 and 8332	RCRA
Thiodiglycol	Accutest in-house method (HPLC)	RCRA

Method Type	Method Number	Regulatory Program
Prep Methods:		
Liquid/Liquid Extraction, Water	SW846 – 3510C	RCRA
Solids Extraction by Sonication	SW846 – 3550B	RCRA
Acid/Base Partitioning	SW846 – 3650B	RCRA
Sulfur Cleanup of Extracts	SW846 – 3660B	RCRA
Sulfuric Acid Cleanup	SW846 – 3665A	RCRA
Purge & Trap - Aqueous	SW846 – 5030B	RCRA
Purge & Trap – Solids	SW846 – 5035	RCRA
Metals:		
Total Recov. Metals Digestion	EPA 200.7	Clean Water Act
Non-Pot. Water Digest: ICP	SW846 3010A,	RCRA
Digestion of Soils for ICP	SW846 3050B	RCRA
ICP: General – EPA WW	EPA 200.7, 1983	Clean Water Act
ICP (General – SW846 update)	SW846 6010B	RCRA
Cold Vapor Mercury – EPA WW	EPA 245.1, 1983	Clean Water Act
Cold Vapor Mercury – EPA DW	EPA 245.1, 1994	Safe Drinking Water Act
Cold Vapor Mercury – AQ	SW846 7470A	RCRA
Cold Vapor Mercury – Soils	SW846 7471A	RCRA
General Chemistry:		
Organic Matter – Loss on Ignition	AASHTO T267-86M	AASHTO Method
Oxidation-Reduction Potential	ASTM D1498-76, mod. for solids	ASTM Standard
Percent Ash (dry basis)	ASTM D2974-87, D482-91	ASTM Standard
Grain Size (hydrometer)		ASTM Standard
Sieve Testing	ASTM D422-63	ASTM Standard
Specific Gravity	ASTM D1298-85	ASTM Standard
Acidity	EPA 305.1	Clean Water Act
Alkalinity	EPA 310.1/SM18 2320B	Clean Water Act
CBOD		Clean Water Act
COD		Clean Water Act
BOD	EPA 405.1	Clean Water Act
Color, Apparent	EPA 110.2	Clean Water Act
Dissolved Oxygen	EPA 360.1	Clean Water Act
Fluoride	EPA 340.2	Clean Water Act
Hardness	EPA 130.2	Clean Water Act
Ion Chromatography (Bromide, Fluoride, Chloride, Sulfate, Nitrite, Nitrate,) – Aqueous	EPA 300.0	Clean Water Act

Method Type	Method Number	Regulatory Program
Mineral Suspended Solids	EPA 160.2/160.4	Clean Water Act
Total Kjeldahl Nitrogen	EPA 351.3	Clean Water Act
Ammonia	EPA 350.2	Clean Water Act
Nitrogen, Nitrite	EPA 354.1/SM18 4500NO2B	Clean Water Act
Odor	EPA 140.1	Clean Water Act
Oil & Grease, Gravimetric – AQ	EPA 9070	RCRA
Oil & Grease, Gravimetric – AQ	EPA 1664	Clean Water Act
Orthophosphate	EPA 365.2/SM18 4500PE	Clean Water Act
Organophosphonic Acids	Accutest in-house method (IC)	RCRA
Percent Solids	EPA 160.3	Clean Water Act
pH by electrode (Waters)	EPA 150.1	Clean Water Act
Phenols – chloroform extraction	EPA 420.1	Clean Water Act
Settleable Solids	EPA 160.5	Clean Water Act
Specific Conductance	EPA 120.1	Clean Water Act
Sulfate (Turbidimetric)	EPA 375.4	Clean Water Act
Sulfide	EPA 376.1	Clean Water Act
Total Dissolved Solids	EPA 160.1/SM18 2540C	Clean Water Act
Total Mineral Solids	EPA 160.4	Clean Water Act
Total Organic Carbon	EPA 415.1	Clean Water Act
Total Organic Carbon	SW-846 9060MOD	RCRA
Total Residual Chlorine	EPA 330.5/SM18 4500CLF	Clean Water Act
Total Solids	EPA 160.3	Clean Water Act
Total Suspended Solids	EPA 160.2	Clean Water Act
Total Volatile Solids	EPA 160.4	Clean Water Act
Turbidity	EPA 180.1	Clean Water Act
Volatile Suspended Solids	EPA 160.2/160.4	Clean Water Act
Total CN	EPA 335.1/2, SW846 9020	CWA or RCRA
CÑ Amenable to Chlorination	EPA 335.1/2, SW846 9020	CWA or RCRA
Perchlorate	EPA 314	DWA, CWA
Waste Ignitability	SW846 1010	CWA or RCRA
Bicarbonate, Carbonate, CO2	SM18 4500 CO2D	None – Standard Method
Calcium Hardness by Calculation	SM18 2340B	None – Standard Method
Ferrous Iron	SM18 3500 FE-D	None – Standard Method
Hardness, Total by Calculation	SM18 2340B	None – Standard Method
MBAS (Anionic Surfactants as)	SM18 5540C	None – Standard Method
Salinity	SM18 2520B	None – Standard Method
Total Nitrogen by calculation (TKN + NO32)	SM18 4500N	None – Standard Method

Method Type	Method Number	Regulatory Program
Total Organic Nitrogen by calculation (TKN – AMN)	SM18 4500N	None – Standard Method
Hexavalent Chromium/soils	SW846 3060/7196A (NJDEP)	None-NJDEP Modification
Ion Chromatography (Bromide, Fluoride, Chloride, Sulfate, Nitrite, Nitrate, Sulfate) – Solids	SW846 9056	RCRA
Corrosivity & pH – aqueous	SW846 9040B	RCRA
Corrosivity & pH – solid	SW846 9045B	RCRA
Waste Corrosivity	SW846 1110	CWA or RCRA
Hexavalent Chromium - soil	SW846 3060A/7196A	RCRA
Hexavalent Chromium - water	SW846 7196A	RCRA
Ignitability	SW846 Chp 7, SW1010, ASTM D93-90	RCRA
Oil & Grease, Gravimetric (Soils)	SW846 9071A M-Solids	RCRA
Paint Filter Test	SW846 9095	RCRA
Phenols with distillation	SW846 9066	RCRA
Synthetic Precipitation Leaching Procedure (SPLP)	SW846 1312	RCRA
Sulfide/Cyanide Reactivity	SW846 Chapter 7	RCRA
Toxicity Characteristics Leaching Procedure (TCLP)	SW846 1311	RCRA

9.0 SAMPLE MANAGEMENT, LOGIN, CUSTODY, STORAGE AND DISPOSAL

Requirement: A system to ensure that client supplied product is adequately evaluated, acknowledged, and secured upon delivery to the laboratory must be practiced by the laboratory. The system must assure that chain of custody is maintained and that sample receipt conditions and preservation status are documented and communicated to the client and internal staff. The login procedure must assign, document, and map the specifications for the analysis of each unique sample to assure that the requested analysis is performed on the correct sample and enables the sample to be tracked throughout the laboratory analytical cycle. The system must include procedures for reconciling defects in sample condition or client provided data, which occur at sample arrival. The system must specify the procedures for proper sample storage, transfer to the laboratory, and disposal after analysis. The system must be documented in a standard operating procedure.

9.1 ***Order Receipt and Entry***. New orders are initiated and processed by the client services group (See Chapter 14, Procedures for Executing Client Specifications). The new order procedure includes mechanisms for providing sampling containers to clients. These containers must meet the size, cleanliness, and preservation specifications for the analysis to be performed.

For new orders, the project manager prepares a bottle request form, which is submitted to sample management. This form provides critical project details to the sample management staff, which are used to prepare and assemble the sample bottles for shipment to the client prior to sampling.

The bottle order is assembled using bottles that meet USEPA specifications for contaminant free sample containers. Accutest-SE uses a combination of pre-cleaned bottles, which are purchased from commercial suppliers and bottles that are checked for cleanliness. Precleaned bottle certificates are reviewed by both the analyst and sample management technician. Results of bottle analyses are retained for 5 years.

All preservative solutions are prepared in the laboratory and are checked to assure that they are free of contamination from analytes of interest before being released for use. Sample management department retains a copy of the documentation of in-house contamination checks.

Reagent water for trip and field blanks is poured into appropriately labeled containers. All bottles are packed into ice chests with blank chain of custody forms and the original bottle order from. Completed bottle orders are delivered to clients using Accutest couriers or commercial carriers for use in field sample collection.

9.2 ***Sample Receipt and Custody***. Samples are delivered to the laboratory using a variety of mechanisms including Accutest couriers, commercial shippers, and client self-delivery. Documented procedures are followed for arriving samples to assure that custody and integrity are maintained and that handling and preservation requirements are documented and continued.

Sample custody documentation is initiated when the individual collecting the sample collects field samples. Custody documentation includes all information necessary to provide an unambiguous record of sample collection, sample identification, and sample collection chronology. Initial custody documentation employs either Accutest or client generated custody forms.

Accutest generates a chain of custody in situations where the individuals who collected the sample did not generate custody documentation in the field.

Accutest defines sample custody as follows:

- The sample is in the actual custody or possession of the assigned responsible person,

- The sample is in a secure area.

The Accutest facility is defined as a secure facility. Perimeter security has been established, which limits access to authorized individuals only. Visitors enter the facility through the building lobby and must register with the receptionist prior to entering controlled areas. While in the facility, visitors must be accompanied by their hosts at all times. After hours, building access is controlled using a computerized pass-key reader system. This system limits building access to individuals with a pre-assigned authorization status. After hours visitors are not authorized to be in the building. Clients delivering samples after hours must make advanced arrangements through client services and sample management to assure that staff is available to take delivery and maintain custody.

Upon arrival at Accutest, the sample custodian reviews the chain of custody Or Sample Receipt Confirmation form for the samples received to verify that the information on the form corresponds with the samples delivered. This includes verification that all listed samples are present and properly labeled, checks to verify that samples were transported and received at the required temperature, verification that the sample was received in proper containers, verification that sufficient volume is available to conduct the requested analysis, and a check of individual sample containers to verify test specific preservation requirements including the absence of headspace for volatile compound analysis.

Sample conditions and other observations are documented on the chain of custody by the sample custodian prior to completing acceptance of custody. The sample custodian accepts sample custody upon verification that the custody document is correct. Discrepancies or non-compliant situations are documented, flagged and communicated to the Accutest project manager, who contacts the client for resolution. The resolution is documented and communicated to sample management for execution.

9.3 ***Sample Tracking Via Status Change.*** An automated, electronic LIMS procedure records sample exchange transactions between departments and changes in analytical status. This system tracks all preparation, analytical, and data reporting procedures to which a sample is subjected while in the possession of the laboratory. Each individual receiving samples must acknowledge the change in custody and operational status in the LIMS. This step is required to maintain an accurate electronic record of sample status, dates of analytical activity, and custody throughout the laboratory.

Sample tracking is initiated at login where all chronological information related to sample collection dates and holding times are entered into the LIMS. This information is entered on an individual sample basis

9.4 ***Sample Acceptance Policy***. Incoming samples must satisfy Accutest's sample acceptance criteria before being logged into the system. Sample acceptance is based on the premise that clients have exercised proper protocols for sample collection. This includes sufficient volume, proper chemical preservation, temperature preservation, sample container sealing and labeling, and appropriate shipping container packing.

The sample management staff will make every attempt to preserve improperly preserved samples upon arrival. However, if preservation is not possible, the samples may be refused unless the client authorizes analysis. No samples will be accepted if holding times have been exceeded or will be exceeded before analysis can take place unless the client authorizes analysis.

Sample acceptance criteria include proper custody and sample labeling documentation. Proper custody documentation includes an entry for all physical samples delivered to the laboratory with an identification code that matches the sample bottle and a date and signature of the individual who collected the sample and delivered them to the laboratory.

Accutest reserves the right to refuse any sample which in its sole and absolute discretion and judgement is hazardous, toxic and poses or may pose a health, safety or environmental risk during handling or processing. The company will not accept samples for analysis using methodology that is not performed by the laboratory or for methods that lab does not hold valid accreditation unless arrangements have been made to have the analysis conducted by a qualified subcontractor.

9.5 ***Assignment of Unique Sample Identification Codes***. Unique identification codes must be assigned to each sample bottle to assure traceability and unambiguously identify the tests to be performed in the laboratory.

The sample identification coding process begins with the assignment of a unique alphanumeric job number. A job is defined as a group of samples received on the same day, from a specific client pertaining to a specific project. A job may consist of groups of samples received over multi-day period. The first character of the job number is an alpha-character that identifies the laboratory facility. The next characters are numeric and sequence by one number with each new job.

Unique sample numbers are assigned to each bottle collected as a discrete entity from a designated sample point. This number begins with the job number and incorporates a second series of numbers beginning at one and continuing chronologically for each point of collection. The test to be performed is clearly identified on the bottle label.

Alpha suffixes may be added to the sample number to identify special designations such as subcontracted tests, in-house QC checks, or re-logs. Multiple sample bottles for a specific analysis are labeled Bottle 1, Bottle 2, etc.

9.6 ***Subcontracted Analysis***. Subcontract laboratories are employed to perform analysis not performed by Accutest. The quality assurance staff evaluates subcontract laboratories to assure their quality processes meet the standards of the environmental laboratory industry prior to engagement. Throughout the subcontract process, Accutest follows established procedures to assure that sample custody is maintained and the data produced by the subcontractor meets established quality criteria.

Accutest network laboratories are considered primary subcontractors.

*Subcontracting Procedure***.** Subcontracting procedures are initiated through several mechanisms, which originate with sample management. Samples for analysis by a subcontractor are logged into the Accutest system using regular login procedures. If subcontract parameters are part of the project or sample management has received subcontracting instructions for a specific project, a copy of the chain of custody is given to the appropriate project manager with the subcontracted parameters highlighted. This procedure triggers the subcontract process at the project management level. The Sample Management supervisor contacts an approved subcontractor to place the subcontract order.. Subcontract chain of custody is processed in Sample Management Department and copy is filed with the original CoC. Sample management signs the subcontract chain of custody and ships the sample(s) to the subcontractor. The subcontract COC is filed with the original COC and the request for subcontract. Copies are distributed to the login department, the project manager, and sample management.

Client is verbally notified by Project Manager of the requirement to subcontract to the outside laboratory as soon as need Is identified by the Accutest staff. Client notification must be verified in writing, i.e. by e-mail. Client notification may take place during the initial project set-up, or at the time of sample receipt and login.

Subcontractor data packages are reviewed by the QA Staff to assess completeness and quality compliance. If completeness defects are detected, the subcontractor is asked to immediately upgrade the data package. If data quality defects are detected, the package is forwarded to the QA staff for further review. The QA staff will pursue a corrective action solution before releasing data to the client.

Approved subcontract data is entered into the laboratory information management system (LIMS) if possible and incorporated into the final report. All subcontract data is footnoted to provide the client with a clear indication of its source. Copies of original

subcontract data are included in the data report depending on the reporting level specified by the client.

*Subcontract Laboratory Evaluation***.** The QA staff evaluates subcontract laboratories prior to engagement. The subcontract laboratory must provide Accutest with a valid certification to perform the requested analysis, and a copy of the laboratory Quality Systems Manual. Certification verification must be submitted to Accutest annually. Qualification of a subcontract laboratory may be bypassed if the primary client directs Accutest to employ a specific subcontractor.

9.7 ***Sample Storage***. Following sample custody transfer, samples are assigned to various refrigerated storage areas by the sample management staff depending upon the test to be performed and the matrix of the samples. The location (refrigerator and shelf) of each sample is entered into sample location database on the line corresponding to each sample number. Samples remain in storage until the laboratory technician retrieves them into the laboratory for analysis.

Samples for volatile organics analysis are placed in storage in designated refrigerators by the sample management staff and immediately transferred to the organics group control. Sample custody is transferred to the VOC department staff. These samples are segregated according to matrix to limit opportunities for cross contamination to occur.

Organics staff is authorized to retrieve samples from these storage areas for analysis. When analysis is complete, the samples are placed back into storage.

9.8 ***Sample Login***. Following sample custody transfer to the laboratory, the documentation that describes the clients analytical requirements are delivered to the sample login group for coding and entry to the Laboratory Information management System (LIMS). This process translates all information related to collection time, turnaround time, sample analysis, and deliverables into a code which enables client requirements to be electronically distributed to the various departments within the laboratory for scheduling and execution.

The technical staff is alerted to client or project specific requirements through the use of a unique project code that is electronically attached to the job during login. The unique project code directs the technical staff to controlled specifications documents detailing the unique requirements.

9.9 ***Sample Retrieval for Analysis***. It is a responsibility of individual analyst to retrieve samples for analysis. Sample Management employs a program to facilitate sample placement and retrieval. Sample is traced around the laboratory using Status feature of LIMS.

After sample analysis has been completed, the analyst places the sample back into the storage and updates sample status.

9.10 ***Sample Disposal***. Accutest retains all samples under proper storage for a minimum of 30 days following completion of the analysis report. Longer storage periods are accommodated on a client specific basis if required. Samples may also be returned to the client for disposal.

Accutest disposes of all laboratory wastes following the requirements of the Resource Conservation and Recovery Act (RCRA). The Company has obtained and maintains a waste generator identification number, FLR00001263309002 (FLR designates State of Florida).

Sample management generates a sample disposal dump sheet from the LIMS tracking system each week, which lists all samples whose holding period has expired. Data from each sample is compared to the hazardous waste criteria established by the Florida Department of Environmental Protection (FDEP).

Samples containing constituents at concentrations above the criteria are labeled as hazardous and segregated into six separate waste categories for disposal as follows:

* Organic extracts: Chlorinated solvents
* Mixed non-chlorinated (flammable) solvents (hexane, acetone, toluene, acetonitrile, ether)
* Waste oil
* Soil (solids)
* Aqueous
* Corrosive waste

Non-hazardous aqueous samples are diluted, neutralized, and disposed directly into the laboratory sink.

Non-hazardous solids are drummed and disposed of by waste company. Sample bottles are disposed of as recyclable waste in order to crush the bottles and destroy the labels.

Laboratory wastes are collected by waste stream in designated areas throughout the laboratory. Waste streams are consolidated twice a week by the waste custodian and transferred to stream specific drums for disposal through a permitted waste management contractor. Filled, consolidated drums are tested for hazardous characteristics and scheduled for removal from the facility for appropriate disposal based on the laboratory data.

10.0 LABORATORY INSTRUMENTATION AND MEASUREMENT STANDARDS

Requirement: Procedures, which assure that instrumentation is performing to a pre-determined operational standard prior to the analysis of any samples, must be established by the laboratory. In general, these procedures will follow the regulatory agency requirements established in promulgated methodology. The instrumentation selected to perform specified analysis is capable of providing the method-specified uncertainty and sufficient sensitivity of measurement needed. These procedures must be documented and incorporated into the standard operating procedures for the method being executed.

10.1 ***Mass Tuning – Mass Spectrometers***. The mass spectrometer tune and sensitivity must be monitored to assure that the instrument is assigning masses and mass abundances correctly and that the instrument has sufficient sensitivity to detect compounds at low concentrations. This is accomplished by analyzing a specific mass tuning compound at a fixed concentration. If the sensitivity is insufficient to detect the tuning compound, corrective action must be performed prior to the analysis of standards or samples. If the mass assignments or mass abundances do not meet criteria, corrective action must be performed prior to the analysis of standards or samples.

10.2 ***Wavelength Verification – Spectrophotometers***. Spectrophotometer detectors are checked on a regular schedule to verify proper response to the wavelength of light needed for the test in use. If the detector response does not meet specifications, corrective action (detector adjustment or replacement) is performed prior to the analysis of standards or samples.

10.3 ***Inter-element Interference Checks (Metals)***. Inductively Coupled Plasma Emission Spectrophotometers (ICP) are subject to a variety of spectral interferences, which can be minimized or eliminated by applying interfering element correction factors and background correction points. Interfering element correction factors are checked on a specified frequency through the analysis of check samples containing high levels of interfering elements. Analysis of single element interferent solutions is also conducted at a specified frequency.

If the check indicates that the method criteria has not been achieved for any element in the check standard, the analysis is halted and data from the affected samples are not reported. Sample analysis is resumed after corrective action has been performed and the correction factors have been re-calculated.

New interfering element correction factors are calculated and applied whenever the checks indicate that the correction factors are no longer meeting criteria. At a minimum, correction factors are replaced once a year.

10.4 ***Calibration and Calibration Verification***. Many tests require calibration using a series of reference standards to establish the concentration range for performing quantitative analysis. Method specific procedures for calibration are followed prior to any sample analysis.

Calibration is performed using a linear or quadratic regression calculation or calibration factors calculated from the curve. The calibration must meet method specific criteria for linearity or precision. If the criteria are not achieved, corrective action (instrument maintenance or re-calibration) is performed. The instrument must be successfully calibrated before analysis of samples can be conducted.

Initial calibration for metals analysis performed using inductively coupled plasma (ICP) employs the use of two standards and a calibration blank to establish linearity. The calibration blank contains all reagents that are placed into the calibration standard with the exception of the target elements. Valid calibration blanks must not contain any target elements.

Initial calibrations must be initially verified using a single concentration calibration standard from a second source (i.e. separate lot or different provider). The continuing validity of an existing calibration must be regularly verified using a single concentration calibration standard. The response to the standard must meet pre-established criteria that indicate the initial calibration curve remains valid. If the criteria are not achieved corrective action (re-calibration) is performed before any additional samples may be analyzed.

10.5 ***Linear Range Verification and Calibration (ICP Metals)*** A linear range verification is performed for all ICP instrumentation. The regulatory program or analytical method specifies the verification frequency. A series of calibration standards are analyzed over a broad concentration range. The data from these analyses are used to determine the valid analytical range for the instrument..

Some methods or analytical programs require a low concentration calibration check to verify that instrument is sufficient to detect target elements at the reporting limit. The analytical method or regulatory program defines the criteria used to evaluate the low concentration calibration check. If the low calibration check fails criteria, corrective action is performed and verified through reanalysis of the low concentration calibration check before continuing with the field sample analysis.

In accordance with NELAC standards minimum number of calibration points in the absence of method-specific requirements is two calibration points and a blank.

10.6 ***Retention Time Verification (GC/HPLC/IC)***. Chromatographic retention time windows are developed for all analysis performed using gas chromatographs with conventional detectors. An initial experimental study is performed, which establishes the width of the retention window for each compound. The retention time range of the window defines the time ranges for elution of specified target analytes on the primary and

confirmation columns. Retention time windows are established upon initial calibration, applying the retention time range from the initial study to each target compound. Retention times are regularly confirmed through the analysis of an authentic standard during calibration verification. If the target analytes do not elute within the defined range during calibration verification, the instrument must be recalibrated and new windows defined. New studies are performed when major changes, such as column replacement are made to the chromatographic system.

10.7 ***Equipment List***.

Table 10.7 Accutest Laboratories Equipment List

Organics			
Instrument	**Model**	**Serial #**	**Year**
GC/MS	Agilent 5975 MSD/Agilent 7683 AS	US53921303	2005
GC/MS	Agilent 5973 MSD/OI 4552/4560 Archon	CN10417014	2004
GC/MS	Agilent 5973 MSD/OI 4552/4560 Archon	CN10418032	2004
GC/MS	Agilent 5973 MSD/OI 4552/4560 Archon	US21843765	2002
GC/MS	Agilent 5973 MSD/OI 4552/4560 Archon	US21844034	2002
GC/MS	Agilent 5973 MSD/OI 4552/4560 Archon	US02440350	2000
GC/MS	Agilent 5973 MSD/OI 4552/4560 Archon	US94240108	1999
GC/MS	Agilent 5973 MSD/Agilent 7683 AS	US82311290	1998
GC/MS	Agilent 5973 MSD/Agilent 7683 AS	US81211109	1998
GC/MS	Hewlett-Packard 5970 MSD/OI 4552/4560 Archon	3034A12782	1989
GC/MS	Hewlett-Packard 5970 MSD/OI 4552/4560 Archon	2905A11904	1987
GC/MS	Hewlett-Packard 5970 MSD/OI 4552/4560 Archon	2716A10454	1987
GC	Agilent 6890N/Dual ECD/7683 AS	US10613003	2006
GC	Agilent 6890/PID/PID/OI 4552/4560 Archon	CN10421047	2004
GC	Agilent 6890/PID/FID/OI 4552/4560 Archon	US10239007	2002
GC	Agilent 6890N/Dual FID/HP 7683 AS	CN10425061	2004
GC	Agilent 6890N/Dual ECD/HP 7683 AS	US10333015	2003
GC	Agilent 6890/Dual ECD/HP 7683 AS	US00036916	2000
GC	Agilent 6890/Dual ECD/HP 7683 AS	US00028304	1999
GC	Hewlett-Packard 5890/Dual FID/HP 7673 AS	3336A61096	1995
GC	Hewlett-Packard 5890/PID/ELCD/ OI 4552/4560 Archon	3336A60617	1995
GC	Hewlett-Packard 5890/Dual FID/HP 7673 AS	3336A59489	1995
GC	Hewlett-Packard 5890/PID/FID/ OI 4552/4560 Archon	3336A51045	1995
GC	Hewlett-Packard 5890/PID/ELCD/OI 4552/4560 Archon	3203A41646	1992
GC	Hewlett-Packard 5890/Dual FID/HP 7673 AS	3126A51085	1991
GC	Hewlett-Packard 5890/Dual ECD/HP 7673 AS	2921A24618	1990
GC	Hewlett-Packard 5890/PID/FID/ dual MPM 16	3029A29748	1990
GC	Hewlett-Packard 5890/FID	2843A20183	1988

GC	Hewlett-Packard 5890/Dual ECD/HP 7673 AS	2728A14096	1987
HPLC	Agilent 1100 Automated LC System	DE91604217	2004
HPLC	Agilent 1100 Automated LC System	DE83700740	1999
HPLC	Agilent 1100 Automated LC System	DE01608404	2000
HPLC	Hewlett-Packard 1050Q Automated LC System	3149G01430	1996
O-Prep	TurboVap 4 units		2001
O-Prep	TurboVap 3 units		2004
O-Prep	Sonicator 4 units		2001
O-Prep	Sonicator 2 units		2004
O-Prep	Midi-Vap 2000 Kontes	479200-2000	2000
Data System	Hewlett-Packard/MS ChemStation		1999

Inorganics			
Instrument	**Model**	**Serial #**	**Year**
ICP	TJA EnviroTrace 61E Simultaneous	348490	1994
ICP	TJA EnviroTrace 61E Simultaneous	470790	1997
Mercury Analyzer	Leeman Hydra AA	2022	2002
TOC Analyzer	Shimadzu	H51404235007	2004
IC	Dionex DX-500	99030664	1999
IC	Dionex	04070250	2004
Centrifuge	CentraCL2	42613052	2003
Spectrophotometer	Milton-Roy Spectronic 200		2000
Auto Analyzer	QuickChem 8500	050500000130	2005

LIMS		
Instrument	**Model**	**Year**
LIMS	HP True 64	1999

11.0 INSTRUMENT MAINTENANCE

***Requirement*.** Procedures must be established for equipment maintenance. The procedure may include a maintenance schedule if required or documentation of daily maintenance related activities. All instrument maintenance activities must be documented in instrument specific logbooks.

11.1 ***Routine, Daily Maintenance***. Routine, daily maintenance is required on an instrument specific basis. It is performed each time the instrument is used. Daily maintenance traditionally includes activities to insure a continuation of good analytical performance. In some cases, they include performance checks that indicate whether non-routine maintenance is required. If the performance check indicates a need for higher level maintenance, the equipment is taken out of service until maintenance is performed. Analysis cannot be continued until the performance checks meet established criteria. Document return to control. Daily maintenance is the responsibility of the individual assigned to the instrument used for the analysis he is performing.

11.2 ***Non-routine Maintenance***. Non-routine maintenance is reserved for catastrophic occurrences such as instrument failure. The need for non-routine maintenance is indicated by failures in general operating systems, that result in an inability to conduct required performance checks or calibration. Equipment in this category are taken out of service and repaired before attempting further analysis. Analysis cannot continue until the instrument meets all performance check criteria and is capable of being calibrated. Section supervisors are responsible for identifying non-routine maintenance episodes and initiating repair activities to bring the equipment on-line. This may include initiating telephone calls to maintenance contractors if necessary. They are also responsible for documenting all details related to the occurrence and the repair.

11.3 ***Scheduled Maintenance***. Modern laboratory instrumentation rarely requires regular preventative maintenance. Where required, the equipment is placed on a schedule, which dictates when maintenance is required. Examples include annual balance calibration by an independent provider and optical alignment of the ICP. Section supervisors are responsible for initiating scheduled maintenance on equipment that requires scheduled preventative attention. Scheduled maintenance is documented using routine documentation practices.

11.4 ***Maintenance Documentation***. Routine and non-routine maintenance activities are documented in logbooks assigned to instruments and equipment used for analytical measurements. The logbooks contain preprinted forms, which specify the maintenance activities required with each use. Accutest Laboratories Southeast has adopted a problem – action – follow-up format to conduct instrument maintenance. The analyst or supervisor who performs or initiates the maintenance activity is required to check the activity upon its completion and initial the form. Non-routine maintenance (i.e. repairs, upgrades, etc.) is documented in a separate service log.

12.0 QUALITY CONTROL PARAMETERS, PROCEDURES, AND CORRECTIVE ACTION

Requirement: All procedures used for test methods must incorporate quality control parameters to monitor elements that are critical to method performance. Each quality parameter includes acceptance criteria that have been established by regulatory agencies for the methods in use. Criteria may also be established through client dictates or through the accumulation and statistical evaluation of internal performance data. Data obtained from these parameters must be evaluated by the analyst, and compared to established method criteria. If the criteria are not achieved, the procedures must specify corrective action and conformation of control before proceeding with sample analysis. QC parameters, procedures, and corrective action must be documented within the standard operating procedures for each method. In the absence of client specific objectives the laboratory must define qualitative objectives for completeness and representativeness of data.

12.1 ***Procedure***. Bench analysts are responsible for methodological quality control and sample specific quality control. Each method specifies the control parameters to be employed for the method in use and the specific procedures for incorporating them into the analysis. These control parameters are analyzed and evaluated with every designated sample group (batch).

The data from each parameter provides the analyst with critical decision making information on method performance. The information is used to determine if corrective action is needed to bring the method or the analysis of a specific sample into compliance. These evaluations are conducted throughout the course of the analysis. Each parameter being indicative of a critical control feature. Failure of a methodological control parameter is indicative of either instrument or batch failure. Failure of a sample control parameter is indicative of control difficulties with a specific sample or samples.

Sample Batch. All samples analyzed in the laboratory are assigned to a designated sample batch, which contains all required quality control samples and a defined maximum number of field samples that are prepared and/or analyzed over a defined time period. The maximum number of field and quality control samples in the batch is 20. Accutest has incorporated the NELAP batching policy as the sample-batching standard. This policy incorporates the requirement for blanks and spiked blanks as a time based function as defined by NELAP. The typical batch contains a blank, laboratory control sample (LCS or spiked blank), matrix spike and matrix spike duplicate. Batch documentation includes lot specifications for all reagents and standards used during preparation of the batch.

12.2 ***Methodological Control Parameters and Corrective Action***. Prior to the analysis of field sample the analyst must determine that the method is functioning properly. Specific control parameters indicate whether critical processes meet specified requirements before continuing with the analysis. Method specific control parameters must meet criteria before sample analysis can be conducted. Each of these

parameters is related to processes that are under the control of the laboratory and can be adjusted if out of control.

Method Blank. A method blank is analyzed during the analysis of any field sample. The method blank is defined as a sample. It contains the same standards (internal standards, surrogates, matrix modifiers, etc.) and reagents that are added to the field sample during analysis, with the exception of the sample itself. If the method blank contains target analytes(s) at concentrations that exceed method or client requirements (typically defined as method detection limit concentrations), the source of contamination is eliminated before proceeding with sample analysis. Systematic contamination is documented for corrective action and resolved following the established corrective action procedures. In specific cases, contamination detected in the method blank may be acceptable if the concentrations do not exceed regulatory limits or client defined reporting limits.

Laboratory Control Samples (LCS or Spiked Blanks). A laboratory control sample (spiked blank or commercially prepared performance evaluation sample) is analyzed along with field samples to demonstrate that the method accuracy is within acceptable limits. These spike solutions are derived from different sources than the solutions used for method calibration. The performance limits are derived from published method specifications or from statistical controls generated from laboratory method performance data. Spiked blanks are blank matrices (reagent water or clean sand) spiked with the targeted parameters and analyzed using the same method used for samples. Accuracy data is compared to laboratory experimentally derived limits to determine if the method is in control. Laboratory control samples (LCS) are commercially prepared spiked samples in an inert material. Performance criteria for recovery of spiked analytes is pre-established by the commercial entity preparing the sample. This sample is analyzed in the laboratory as an external reference.

Accuracy data is compared to the applicable performance limits. If the spike accuracy exceeds the performance limits, corrective action, as specified in the SOP for the method is performed and verified before continuing with a field sample analysis. In some cases, decisions are made to continue with sample analysis if performance limits are exceeded; provided the unacceptable result has no negative impact on the sample data.

Marginal exceedance (ME) values are calculated for methods containing more than eleven (11) targeted analytes. The ME is calculated as $\pm$ 4 standard deviations about the mean. MEs are considered for multi-analyte methods because of the increased likelihood of LCS failure as the number of analytes in the method increase. The number of allowable MEs is based on the number of target analytes in the method. Analytes that regularly fall into the ME category are treated as systematic problems, which are resolved using established trend monitoring and corrective action procedures. Marginal Exceedances are not applied to parameters that are detected in field samples. Routine corrective action is initiated for all cases where LCS spike

accuracy criteria is beyond the established control limits and the parameter is detected in field samples corresponding to the unacceptable LCS.

Blanks and spikes are routinely evaluated before samples are analyzed. However, in situations where sample analysis is performed using an autosampler, they may be evaluated after sample analysis has occurred. If the blanks and spikes do not meet criteria, sample analysis is repeated.

Proficiency Testing. Performance evaluation samples (PEs) are single or double blind spikes, introduced to the laboratory to assess method performance. PEs may be introduced as double blinds submitted by commercial clients, single or double blinds from regulatory agencies, or internal blinds submitted by the QA group.

A minimum of two single blind studies must be performed each year for every parameter in aqueous and solid matrices for each field of testing for which the laboratory maintains accreditation. Proficiency samples must be purchased as blinds from an NIST NVLAP accredited vendor. Data from these studies are provided to the laboratory by the vendor and reported to accrediting agencies. If unsatisfactory performance is noted, corrective action is performed to identify and eliminate any sources of error. A new single blind must be analyzed to demonstrate continuing proficiency.

PE samples performed for accrediting agencies or clients, which do not meet performance specifications, require a written summary that documents the corrective action investigation, findings, and corrective action implementation.

Single or double blind proficiency test samples are employed for self-evaluation purposes. Data from these analyses are compared to established performance limits. If the data does not meet performance specifications, the system is evaluated for sources of acute or systematic error. If required, corrective action is performed and verified before initiating or continuing sample analysis.

Trend Analysis for Control Parameters. Accuracy data for selected spiked parameters from the laboratory control sample (LCS) is statistically evaluated daily for trends. Data from selected LCS parameters and surrogates are pooled on a method, matrix, and instrument basis. This data is evaluated by comparison to existing control and warning limits. Trend analysis is performed automatically as follows:

- Any point outside the control limit
- Any three consecutive points between the warning and control limits
- Any eight consecutive points on the same side of the mean
- Any six consecutive points increasing or decreasing

The results of the trend analysis are printed for supervisory evaluation prior to sample analysis. Trends that indicate the potential loss of statistical control are further evaluated to determine the impact on data quality and to determine if corrective action

is necessary. If corrective action is indicated, the supervisor informs the analysts of the corrective actions to be performed. Return to control is demonstrated before analysis resumes.

12.3 ***Sample Control Parameters and Corrective Action***. The analysis of samples can be initiated following a successful demonstration that the method is operating within established controls. Additional controls are incorporated into the analysis of each sample to determine if the method is functioning within established specifications for each individual sample. Sample QC data is evaluated and compared to established performance criteria. If the criteria are not achieved the method or the SOP specifies the corrective action required to continue sample analysis. In many cases, failure to meet QC criteria is a function of sample matrix and cannot be remedied. Each parameter is designed to provide quality feedback on a defined aspect of the sampling and analysis episode.

Duplicates. Duplicate sample analysis is used to measure analytical precision. This can also be equated to laboratory precision for homogenous samples. Precision criteria are method dependent. If precision criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

Laboratory Control Duplicate, Spikes & Spiked Duplicates. Spikes and spiked duplicates are used to measure analytical precision and accuracy for the sample matrix selected. Precision and accuracy criteria are method dependent. If precision and accuracy criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

Serial Dilution (Metals). Serial dilutions of metals samples are analyzed to determine if analytical matrix effects may have impacted the reported data. If the value of the serially diluted samples does not agree with the undiluted value within a method-specified range, the sample matrix may be causing interference, which may lead to either a high or low bias. If the serial dilution criterion is not achieved, it must be flagged to indicate possible bias from matrix effects. *Accutest-SE uses this procedure as opposed to post-digestion spike unless contractual obligations absolutely require latter*

Post Digestion Spikes. Digested samples are spiked and analyzed to determine if matrix interferences are creating biases in the results. It may also be used to determine potential interferences per client's specification. Spike concentration is determined as per analytical method. No action is necessary if the post digestion spike is outside of the method criteria, unless a preparation problem is suspected with the spike, in which case the post digestion spike should remade and reanalyzed.

Surrogate Spikes (Organics). Surrogate spikes are organic compounds that are similar in behavior to the target analytes but unlikely to be found in nature. They are added to all quality control and field samples to measure method performance for each

individual sample. Surrogate accuracy limits are derived from published method specifications or by statistical evaluation of laboratory generated surrogate accuracy data. Accuracy data is compared to the applicable performance limits. If the surrogate accuracy exceeds performance limits, corrective action, as specified in the method or SOP is performed before sample data can be reported.

Internal Standards (Organic Methods). Internal standards are retention time and instrument response markers added to every sample to be used as references for quantitation. Their response is compared to reference standards and used to evaluate instrument sensitivity on a sample specific basis. Internal standard retention time is also compared to reference standards to assure that target analytes are capable of being located by their individual relative retention time.

If internal standard response criteria are not achieved, corrective action or additional action may be required. The recommended action must be completed before sample data can be reported.

If the internal standard retention time criteria are not achieved corrective action or additional action may be required. This may include re-calibration and re-analysis. Additional action must be completed before sample data is reported.

Internal Standards (ICP Metals). Internal standards are used on ICP instruments to compensate for variations in response caused by differences in sample matrices. This adjustment is performed automatically during sample analysis. The internal standard response of replicated sample analysis is monitored to detect potential analytical problems. If analytical problems are suspected, then the field samples are reanalyzed.

12.4 ***Laboratory Derived Quality Control Criteria.*** Control criteria for in-house methods and client specific modifications that exceed the scope of published methodology are defined and documented prior to the use of the method. The Quality Assurance staff identifies the responsibility for control criteria needs. Control parameters and criteria, based on best technical judgement are established using input provided by the operations staff. These control parameters and criteria are documented and incorporated into the method.

The laboratory derived criteria are evaluated for technical soundness on spiked samples prior to the use of the method on field samples. The technical evaluation is documented and archived by the Quality Assurance staff.

When sufficient data form the laboratory developed control parameter is accumulated, the data is statistically processed and the experimentally derived control limits are incorporated into the method.

12.5 ***Bench Review & Corrective Action***. The bench chemists are responsible for all QC parameters. Before proceeding with sample analysis, they are required to

successfully meet all instrumental QC criteria. They have the authority to perform any necessary corrective action before proceeding with sample analysis. Their authority includes the responsibility for assuring that departures from documented policies and procedures do not occur.

The bench chemists are also responsible for all sample QC parameters. If the sample QC criteria are not achieved, they are authorized and required to perform the method specified corrective action before reporting sample data.

***Data Qualifiers*.** An alpha character coding system is employed for defining use limitations for reported data. These limitations are applied to analytical data by the analyst to clarify the usefulness of the reported data for data user. Accutest Laboratories Southeast qualifies data in accordance with program-specific requirements, such as State of Florida DEP, AFCEE, etc., and these qualifiers are hard-coded in the LIMS on project level. Definitions of common qualifiers could be found at the bottom of the sample report form.

12.6 ***QA Monitoring***. The QA staff prior to client release conducts a spot review of completed data packages. This review includes an examination of QC data for compliance and trends indicative of systematic difficulties. If non-conformances are detected, the QA staff places an immediate stop on the release of the data and initiates corrective action to rectify the situation. The data package is released when the package becomes compliant with all quality requirements.

If the review reveals trends indicative of systematic problems, QA initiates an investigation to determine the cause. If process defects are detected, a corrective action is implemented and monitored for effectiveness.

Performance Limits*.* The Technical Director is responsible for compilation and maintenance of all precision and accuracy data used for performance limits. Quality control data for all test methods are accumulated and stored in the laboratory information management system (LIMS). Parameter specific QC data is extracted annually and statically processed to eliminate outliers and develop laboratory specific warning limits and confidence limits. The new limits are reviewed and approved by the supervisory staff prior to their use for data assessment. The new limits are used to evaluate QC data for compliance with method requirements for a period of one year. Laboratory generated limits appear on all data reports unless method specifies hard-coded limits (mostly General Chemistry and Metals)

12.7 ***Data Package Review***. Accutest employs multiple levels of data review to assure that reported data has satisfied all quality control criteria and that client specifications and requirements have been met. Production departments have developed data review procedures which must be conducted before data is released to the client.

Analytical Review. The analyst conducts the primary review of all data. This review begins with a check of all instrument and method quality control and progresses

through sample quality control concluding with a check to assure that the client's requirements have been executed. Analyst checks focuses on a review of qualitative determinations and checks of precision and accuracy data to verify that existing laboratory criteria have been achieved. Checks at this level may include comparisons with project specific criteria if applicable. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter or nonconformance at this stage of review.

Secondary data reviews are performed at the peer level by analysts who have met the qualification criteria for the method in use. Qualification requirements include a valid demonstration of capability and demonstrated understanding of the method SOP. Section supervisors may perform secondary review in-lieu of a peer review Secondary review is performed on 100% of the data produced by their department. It includes a check of all manual calculations; an accuracy check of manually transcribed data from bench sheets to the LIMS, a check of all method and instrument QC criteria, baseline manipulations (if applicable) and a comparison of the data package to client specified requirements. Also included are checks to assure the appropriate methodology was applied and that all anomalous information was properly flagged for communication in the case narrative. Supervisors have the authority to reject data and initiate re-analysis, corrective action, or reprocessing.

All laboratory data requiring manual entry into LIMS system is double checked by the analysts performing initial data entry and the section supervisor. Verification of supervisory review is indicated on the raw data summary by the supervisor's initials and date.

Electronic data that is manually edited at the bench by the primary analysts is automatically flagged by the instrument data system indicating an override by the analyst. All manual overrides must be verified and approved by a supervisor who initials and dates all manual changes.

Hard copies of manually integrated chromatographic peaks are printed that clearly depict the manually drawn baseline. The hard copy is reviewed and approved by the reviewer (initialed and dated) and included in the data package of all full tier reports or the archived batch records of commercial report packages.

Electronic data that has been committed to the LIMS can only be edited by a manager or supervisor. These edits may be required if needs for corrections are indicated during the final review. An audit record for all electronic changes in the LIMS is automatically appended to the record.

The group manager performs a tertiary review on a spot check basis. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

Report Generation Review. The report generation group reviews all data and supporting information delivered by the laboratory for completeness and compliance with client specifications. Missing deliverables are identified and obtained from the laboratory. The group also reviews the completed package to verify that the delivered product complies with all client specifications. Non-analytical defects are corrected before the package is sent to the client.

Project Management/Quality Assurance Review. Spot-check data package reviews are performed by the project manager. Project management reviews focus on project specifications. If the project manager identifies defects in the product prior to release, he initiates immediate corrective action to rectify the situation.

The QA Staff reviews approximately 10% of the data produced. The QA review focuses on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification. QA reviews at this step in the production process are geared towards systematic process defects, which require procedural changes to effect a corrective action. However, if defects are identified that can be corrected prior to data release, the QA staff returns the package to the laboratory for corrective action. QA data review cannot be used in lieu of a peer level review or a supervisory review.

Data Reporting. Analytical data is released to clients following secondary departmental review. Data release at this stage of the process is limited to electronic information, which is released to clients through a secure, encrypted, password protected, Internet connection.

Hard copy support data is compiled by the report generation group and assembled into the final report. The report is sent to the client following reviews by report generation, and spot-check by QA staff.

All data reports include specified information, which is required to identify the report and its contents. This information includes a title, name and address of the laboratory, a unique report number, total number of pages in the report, clients name and address, analytical method identification, arriving sample condition, sample and analysis dates, test results with units of measurement, authorized signature of data release, statement of applicability, report reproduction restrictions and NELAC requirements certification. Subcontracted data is clearly identified.

12.8 ***Electronic Data Reduction.*** Raw data from sample analysis is entered into the laboratory information management system (LIMS) using automated processes or manual entry. Final data processing is performed by the LIMS using procedures developed by the Company.

All LIMS programs and internally developed software (including Excel spreadsheets) are tested and validated prior to use to assure that they consistently produce correct results. Validation testing is performed by the Information Technology Staff. The

testing procedures are documented in an SOP. Programs are not approved for use until they have demonstrated that they are capable of performing the required calculations.

12.9 ***Representativeness***. Data representativeness is based on the premise that qualitative and quantitative information developed for field samples is characteristic of the sample that was collected by the client and analyzed in the laboratory. The laboratory objective for representativeness defines data as representative if the criteria for all quality parameters associated with the analysis of the sample are achieved.

12.10 ***Comparability***. Analytical data is defined as comparable when data from a sample set analyzed by the laboratory is representatively equivalent to other sample sets analyzed separately regardless of the analytical logistics. The laboratory will achieve 100% comparability for all sample data which meets the criteria for the quality parameters associated with its analysis using the method requested by the client.

13.0

14.0 CORRECTIVE ACTION SYSTEM

Requirement. The laboratory must have polices and procedures for correcting defective processes, systematic errors, and quality defects, which enables the staff to systematically improve product quality. The system must include procedures for communicating items requiring corrective action, corrective action tracking procedures, corrective action documentation, monitoring of effectiveness, and reports to management. The system must be documented in a standard operating procedure.

14.1 ***Procedure***. Corrective action is the step that follows the identification of a process defect. The type of defect determines the level of documentation, communication, and training necessary to prevent re-occurrence of the defect or non-conformance.

Routine Corrective Action. Routine corrective action is defined as the procedures used to return out of control analytical systems back to control. This level of corrective action applies to all analytical quality control parameters or analytical system specifications.

Bench analysts have full responsibility and authority for performing routine corrective action. The resolution of defects at this level does not require a procedural change or staff re-training. The analyst is free to continue work once corrective action is complete and the analytical system has been returned to control. Documentation of routine corrective action is limited to bench logbook or maintenance logbook comment.

Process Changes. Corrective actions in this category require procedural modifications. They may be the result of systematic defects identified during audits, the investigation of client inquiries, failed proficiency tests, product defects identified during data review, or method updates. Resolution of defects of this magnitude requires formal identification of the defect, development and documentation of a corrective action plan, and staff training to communicate the procedural change.

Technical Corrective Action. Technical corrective action encompasses routine corrective action performed by bench analysts for out of control systems and corrective actions performed for data produced using out of control systems. Technical corrective action for routine situations is conducted using the procedures detailed above.

Non-routine corrective actions apply to situations where the bench analysts failed to perform routine corrective action before continuing analysis. Supervisors and Department Managers perform corrective action in these situations. Documentation of all non-routine corrective actions is performed using the corrective action system.

Sample re-analysis is conducted if sufficient sample and holding time remain to repeat the analysis using an in-control system. If insufficient sample or holding time remains,

the data is processed and qualifiers applied that describe the out of control situation. The occurrence is further documented in the case narrative and in the corrective action response. The corrective action must include provisions for retraining the analysts who failed to perform routine corrective action.

14.2 ***Documentation & Communication***. Routine corrective actions are documented as part of the analytical record. Notations are made in the comments section of the analytical chronicle or data sheet detailing the nonconformance. Continuation of the analysis indicates that return to control was successful.

Corrective actions for process changes are documented, tracked and monitored for effectiveness. Corrective actions may be initiated by any supervisor or senior staff member by completing the corrective action form in Corrective Action database

The corrective action database is an Access application. The initiator generates the corrective action investigation form, which is documented, tracked, distributed to responsible parties and archived through the application. The application assigns a tracking number initiation data and due date to each corrective action initiated and copies the corrective action form to the corrective action database. The application also distributes an E-mail message containing the form to the responsible parties for resolution.

Corrective Action system employs Deficiency – Root Cause – Immediate Fix – Corrective action approach, further divided into categories of Analytical Error, Omission Error, Random Error, Systemic Error and Training Issue.

The responsible party develops and implements the procedural change. Existing documentation such as SOPs are edited to reflect the change. The affected staff is informed of the procedural change through a formal training session. The training is documented and copies are placed into individual training files. The corrective action form is completed and closed in CA database.

Initial and completed corrective action forms are maintained in the Corrective Action directory. This information is archived daily. Copies of training records describing corrective actions are appended to the involved individuals training files.

Monitoring. The QA Staff monitors the implemented corrective action until it is evident that the corrective action has been effective and the systematic deficiency has been eliminated. The corrective action database is updated by QA to reflect closure of the corrective action. The QA staff also assigns an error code to the corrective action for classification of the type of errors being committed.

If QA determines that the corrective action procedure has not effectively remedied the deficiency, the process continues with a re-initiation of the corrective action. Corrective action continues until the defective process is eliminated. If another procedural change is required, it is treated as a new corrective action, which is documented and monitored using established procedures.

Client Notification. Defective processes, systematic errors, and quality defects, detected during routine audits may have negative impacts on data quality. In some cases, data that has been released to clients may be affected. If defective data has been released for use, Accutest will notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.

15.0 PROCEDURES FOR EXECUTING CLIENT SPECIFICATIONS

***Requirement*.** Systems must be established for evaluating and processing client specifications for routine and non-routine analytical services. The systems must enable the client services staff to identify, evaluate, and document the requested specifications to determine if adequate resources are available to perform the analysis. The system must include procedures for communicating the specifications to the laboratory staff for execution and procedures for verifying the specifications have been executed.

15.1 ***Client Specific Requirements***. The project manager is the primary contact for clients requesting laboratory services. Client specifications are communicated using several mechanisms. The primary source of information is the client's quality assurance project plan (QAPP) which details analytical and quality control specifications for the project. In the absence of a QAPP, projects specifications can also be communicated using contracts, letters of authorization, or letters of agreement, which may be limited to a brief discussion of the analytical requirements and the terms and conditions for the work. These documents may also include pricing information, liabilities, scope of work, in addition to the analytical requirements. QAPPs include detailed analytical requirements and data quality objectives, which supersede those found in the referenced methods. This information is essential to successful project completion.

The client services staff provides additional assistance to clients who are unsure of the specifications they need to execute the sampling and analysis requirements of their project. They provide additional support to clients who require assistance in results interpretation as needed, provided they possess the expertise required to render an opinion.

The project manager is responsibility for obtaining project documents, which specify the analytical requirements. Following project management review, copies are distributed to the QA Director and the appropriate departmental managers for review and comment. The original QAPP is numbered with a document control number and filed in a secure location.

15.2 ***Requirements for Non-Standard Analytical Specifications***. Client requirements that specify departures from documented policies, procedures, or standard specifications must be submitted to Accutest in writing. These requirements are reviewed and approved by the technical staff before the project is accepted. Once accepted, the non-standard requirements become analytical specifications, which follow the routine procedure for communicating client specifications. Departures from documented policies, procedures, or standard specifications that do not follow this procedure are not permitted.

Exception Policy: With respect to the quality system, incoming non-conforming product refers to received samples that do not meet requirements of custody documentation, are improperly packaged or stored or are contaminated. An internal non-conformance

refers to a problem, caused internally due to improper handling of samples, improper sampling methods, and equipment malfunction or data management errors. The individual who identifies the incoming non-conformance is responsible for notifying the project manager. The project manager resolves the issue with the client. The individual who recognizes an internal non-conformance is responsible for initiating corrective action

Departures from standard practices, policies and specifications are reviewed and approved by Technical Director, QA Officer and by Project Manager of the project affected.

Corrective & Preventative Action: Once a quality problem has been identified, the analytical or review process stops, until the reason is identified. Primary responsibility for identifying the cause of the problem rests with the instrument operator. Other staff may be called on to assist in reaching the root cause. The problem prevention tracking system, using Corrective Action Tracking Records, provides a method to track systemic problems until resolved/removed. The QA Officer is responsible for the record management with respect to the disposition of problems.

Deviations that do not limit themselves to a single department and/or client are cited on Corrective Action Record. This may include but not limited to: sample arrival outside of EPA specified holding time, analysis completion outside of EPA specified holding time (with explanation of the reason), inconsistencies between chain of custody and cooler contents, including labeling errors, improper preservation, etc.

Deviations from analytical methods' SOP's are reported by the Analyst to the Section Leader. Single occurrences warrant completion of Corrective Action Tracking record, repetitive occurrences may indicate that either an additional training session is in order, or the SOP does not reflect proper laboratory practice. Training session is conducted by the Technical Director or by QA Officer. In case where SOP does not reflect current laboratory practice, SOP review and correction process may be initiated.

15.3 ***Evaluation of Resources.*** A resource evaluation is completed prior to accepting projects submitted by clients. The evaluation is initiated by the client services staff receives project requirements (usually in the form of QAPjP) and distrubutes these requirements to the laboratory departments affected. The specifications are evaluated by the department managers from a scheduling and hardware resources perspective. The project is not accepted unless the department managers have the necessary resources to execute the project according to client specifications.

15.4 ***Documentation***. New projects are initiated using a project set up form, which is completed prior to the start of the project. This form details all of the information needed to correctly enter the specifications for each client sample into the laboratory information management system (LIMS, see example). The form includes data reporting requirements, billing information, data turnaround times, QA level, state of

origin, and comments for detailing project specific requirements. The project manager is responsible for obtaining this information from the client and completing the form prior to sample arrival and login.

Sample receipt triggers project creation and the login process. The information on the set-up form is entered into the LIMS immediately prior to logging in the first sample. The set up form may be accompanied by a quotation, which details the analytical product codes and sample matrices. These details are also entered into the LIMS during login.

Special information is distributed to the laboratory supervisors and login department in electronic or hardcopy format upon project setup. All project specific information is retained by the project manager in a secure file. The project manager maintains a personal telephone log, which details conversations with the client regarding the project.

14.5 ***Communication***. A pre-project meeting is held between client services and the operations managers to discuss the specifications described in the QAPjP and/or related documents. Project logistics are discussed and finalized and procedures are developed to assure proper execution of the client's analytical specifications and requirements. Questions, raised in the review meeting, are discussed with the client for resolution. Exceptions to any requirements, if accepted by the client, are documented and incorporated into the QAPjP or project documentation records.

Non-standard specifications for individual clients are documented in the LIMS at the client account level. Once entered into the LIMS, these specifications become memorialized for all projects related to the client account. Upon sample arrival, these specifications are accessed through a terminal or printed as a hard copy and stored in a binder for individuals who require access to the specification. Specifications that are not entered into the LIMS are prohibited unless documented in an interdepartmental memo, which clearly identifies the project, client and effective duration of the specification.

14.6 ***Operational Execution***. A work schedule is prepared for each analytical department on a daily basis. Analytical specifications from recently arrived samples have now been entered into the LIMS database. The database is sorted by analytical due date and holding time, into product specific groups. Samples are scheduled for analysis by due date and holding time. The completed schedule, which is now defined as a work list, is printed. The list contains the client requested product codes and specifications required for the selected sample(s). Special requirements are communicated to the analyst using the comments section or relayed through verbal instructions provided by the supervisor. The bench analyst assumes full responsibility for performing the analysis according to the specifications printed on the work sheet.

14.7 ***Verification***. Prior to the release of data to the client, laboratory section managers and the report generation staff review the report and compare the completed product to the

client specifications documentation to assure that all requirements have been met. Project managers perform a spot check of projects with unique requirements to assure that the work was executed according to specifications.

15.0 CLIENT COMPLAINT RESOLUTION PROCEDURE

Requirement. A system for managing and reconciling client complaints must be implemented in the laboratory. The system must include procedures for documenting client complaints and communicating the complaint to the appropriate department for resolution. The system must also include a quality assurance evaluation to determine if the complaint is related to systematic defects requiring process changes.

15.1 ***Procedure***. Client complaints are communicated to client services representatives, quality assurance staff, or senior management staff for resolution. The individual receiving the complaint retains the responsibility for documentation and communicating the nature of the complaint to the responsible department(s) for resolution. The responsible party addresses the complaint. The resolution is communicated to quality assurance (QA) and the originator for communication to the client. QA reviews the complaint and resolution to determine if systematic defects exist. If systematic defects are present, QA works with the responsible party to develop a corrective action that eliminates the defect.

Documentation. Client's complaints are documented by the client service representative receiving the complaint. A record of the telephone conversation is maintained by client services Complaint is communicated to the production departments concerned via e-mail. The complaint resolution is documented on the e-mail by the responsible party and returned to the originator. QA Officer is copied on the correspondence.

15.2 ***Corrective Action***. Responses to data queries are required from the responsible party. At a minimum, the response addresses the query and provides an explanation to the complaint. Corrective action may focus on the single issue expressed in the complaint. Corrective action may include job case narrative generation, reprocessing of data, editing of the initial report, and re-issue to the client. If the QA review indicates a systematic error, process modification is required. The defective process at the root of the complaint is changed. SOPs are either created or modified to reflect the change. The party responsible for the process implements process changes.

15.3 ***QA Monitoring***. Process changes, implemented to resolve systematic defects, are monitored for effectiveness by QA. If monitoring indicates that the process change has not resolved the defect, QA works with the department management to develop and implement an effective process. If monitoring indicates that the defect has been resolved, monitoring is slowly discontinued. Continued monitoring is incorporated as an element of the annual system audit.

16.0 CONTROL OF NONCONFORMING PRODUCT

Requirement: Policies and procedures have been developed and implemented that describe the procedures employed by the laboratory when any aspect of sample analysis or data reporting do not conform to established procedures or client specifications. These procedures include steps to ensure that process defects are corrected and affected work is evaluated to assess its impact to the client.

Procedure. Nonconforming product is identified through routine internal review and audit practices or through client inquiry. The individuals who identify the nonconformance or receiving a nonconformance inquiry immediately inform the Laboratory Director and the QA Officer. The Laboratory Director initiates an evaluation of the nonconformance through the QA Officer and takes full responsibility for managing the process and identifying the course of action to take, initiating corrective action and mitigating the impact of the nonconformance to the client.

16.1 ***Corrective Action.*** The outcome of the evaluation dictates the course of action. This may include at a minimum client notification, but may also include corrective action. Immediate corrective action is performed using the SOP-specified procedures. However, additional action may be required including cessation of analysis and withholding and/or recalling data reports. If the evaluation indicates that nonconforming data may have been issued to clients, the client is immediately notified and data may be recalled following the procedures specified in respective SOPs. If work has been stopped because of a nonconformance, the Laboratory Director is the only individual authorized to direct a resumption of analysis.

Nonconformances caused by systematic process defects require retraining of the personnel involved as an element of the corrective action solution.

17.0 CONFIDENTIALITY PROTECTION PROCEDURES

Requirements: Policies and procedures are required to protect client data from release to unauthorized parties or accidental release of database information through accidental electronic transmission or illegal intrusion. These policies must be communicated to clients and staff. Electronic systems must be regularly evaluated for effectiveness.

17.1 ***Client Anonymity***. Information related to the Company's clients is granted to employees on a "need to know" basis. An individual's position within the organization defines his "need to know". Individuals with "need to know" status are given password access to systems that contain client identity information and access to documents and document storage areas containing client reports and information. Access to client information by individuals outside of the Company is limited to the client and individuals authorized by the client.

Individuals outside of the Company may obtain client information through subpoena issued by a court of valid jurisdiction. Clients are informed when subpoenas are received ordering the release of their information.

17.2 ***Documents***. Access to client documents is restricted to employees in need to know positions. Copies of all client reports are stored in secure archive with restricted access. Reports and report copies are distributed to individuals who have been authorized by the client to receive them. Documents are not released to third parties without verbally expressed or written permission from the client.

17.3 **Confidential Business Information (CBI).** Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between Accutest and the receiving organization or individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as Adobe Acrobat .PDF format only. See also Sec. 6.5.

17.4 ***Electronic Data***.

Database Intrusion. Direct database entry is authorized for employees of Accutest only on a need to know basis. Entry to the database is restricted through a user specific multiple password entry system. Direct access to the database outside of the facility is possible through a VPN connection. A unique password is required for access to the local area network. A second unique password is required to gain access to the database. The staff receives read or write level authorization on a hierarchical privilege basis.

Internet Access. Access to client information is through an HTTP Web application only. It does not contain a mechanism that allows direct access to the database. Clients can gain access to their data only using a series of Accutest assigned, client and user specific passwords. The viewable data, which is encrypted during transmission, consists of an extraction of database information only.

Client Accessibility. Accessibility to client data delivered via electronic means follows strict protocols to insure confidentiality. Clients accessing electronic data are assigned a company account. The account profile, which is established by the MIS staff, grants explicit access to explicit information pertaining to the clients project activity. Passwords are assigned on an individual basis within a client account. These accounts can be activated or deactivated by the MIS staff only.

17.5 ***Information Requests***. Client specific data or information is not released to third parties without verbally expressed or written permission from the client. Written permission is required from third parties, who contact the Company directly for the release of information. Verbal requests will be honored only if they are received directly from the client. These requests must be documented in a record of communication maintained by authorized recipient.

17.6 ***Transfer of Records***. Archived data, which has previously been reported and transmitted to clients, is the exclusive property of Accutest Laboratories. In the event of a cessation of business activities due to business failure or sale, The Company's legal staff will be directed to arrange for the final disposition of archived data.

The final disposition of archived data will be accomplished using the approach detailed in the following sequence:

1. All data will be transferred to the new owners for the duration of the required archive period as a condition of sale.

2. If the new owners will not accept the data or the business has failed, letters will be sent to clients listed on the most recent active account roster offering them the option to obtain specific reports (identified by Accutest Job Number) at their own expense.

3. A letter will be sent to the NELAC accrediting authority with organizational jurisdiction over the company offering them the option to obtain all unclaimed reports at their own expense.

4. All remaining archived data will be recycled using the most expedient means possible.

18.0 QUALITY AUDITS AND SYSTEM REVIEWS

Requirement: The quality assurance group will conduct regularly scheduled audits of the laboratory to assess compliance with quality system requirements, technical requirements of applied methodology, and adherence to documentation procedures. The information gathered during these audits will be used to provide feedback to senior management and perform corrective action where needed for quality improvement purposes.

18.1 ***Quality Systems Review***. Quality system audits are performed annually by the Quality Assurance Director for the Company President. In this audit, the laboratory is evaluated for compliance with the Laboratory Quality Systems Manual (LQSM) and the quality system standards of the National Environmental Laboratory Accreditation Conference. Findings, which indicate non-compliance or deviation from the LQSM, are flagged for corrective action. Corrective actions require either a return to compliance or a plan change to reflect an improved quality process. The QA Officer is responsible for making and documenting changes to the LQSM. These changes are reviewed by the Laboratory Director and Technical Director prior to the approval of the revised system.

18.2 ***Quality System Audits.*** Quality system audits are conducted to evaluate the effectiveness and laboratory compliance with individual quality system elements. These audits are conducted on an established schedule. Audit findings are documented and communicated to the management staff and entered into the corrective action system for resolution. If necessary, retraining is conducted to assure complete understanding of the system requirements.

18.3 ***Technical Compliance Audits***. Technical compliance audits are performed throughout the year following the established schedule. Selected analytical procedures are evaluated for compliance with standard operating procedures (SOPs) and method requirements. If non-conformances exist, the published method serves as the standard for compliance. SOPs are edited for compliance if the document does not reflect method requirements. Analysts are trained to the new requirements and the process is monitored by quality assurance. Analysts are retrained in method procedures if an evaluation of bench practices indicates non-compliance with SOP requirements.

18.4 ***Documentation Audits***. Documentation audits are conducted periodically. This audit includes a check of measurement processes that require manual documentation and non-analytical logbook review. It also includes checks of data archiving systems and a search to find and remove any inactive versions of SOPs that may still be present in the laboratory and being accessed by the analysts. Non-conformances are corrected on the spot. Procedural modifications are implemented if the evaluation indicates a systematic defect.

18.5 ***Corrective Action Monitoring***. Defects or non-conformances that are identified during client or internal audits are shared with management and entered into CA

database for attention by the responsible party. Audit findings are corrected through process modifications and/or retraining. Once a corrective action has been designed and implemented, it is monitored for compliance on a regular basis by the QA staff. Monitoring of the corrective action continues until satisfactory implementation has been verified.

18.6 ***Preventive Action.*** Laboratory systems or processes, which may be faulty and pose the potential for nonconformances, errors, confusing reports or difficulties establishing traceability may be identified during internal audits. These items are highlighted for systematic change using the corrective action system and managed to resolution using appropriate procedures for corrective action.

18.7 ***Management Reports***. Formal reports of all audit activities are prepared for the management staff. These reports are prepared annually. The report details the status of the Quality System

The formal report also addresses the following topics:

- Status and results of internal and external audits,
- Status and results of internal and external proficiency testing,
- Identification of quality control problems in the laboratory,
- Discussion of corrective action program issues,
- Status of external certifications and approvals,
- Status of staff training and qualifications,
- Discussion of new quality system initiatives.
- Recommendations for further action on listed items are included in the report.

19.0 HEALTH AND SAFETY

Requirement. The company operates a formal health and safety program that complies with the requirements of the Occupational Health and Safety Administration. The program consists of key policies and practices that are essential to safe laboratory operation. All employees are required to receive training on the program elements. Job specific training is conducted to assure safe practices for specific tasks. All employees are required to participate in the program, receive initial and annual training, and comply with the program requirements. All plan and program requirements are detailed in the Health and Safety Program Manual.

19.1 **Policy.** Accutest Laboratories will provide a safe and healthy working environment for its employees and clients while protecting the public and preserving the Company's assets and property. The company will comply with all applicable government regulations pertaining to safety and health in the laboratory and the workplace.

The objective of the Accutest Health and Safety Program is to promote safe work practices that minimize the occurrence of injuries and illness to the staff through proper health and safety training, correct laboratory technique application and the use of engineering controls.

19.2 **Responsibilities.** The Health and Safety Program assists managers, supervisors and non-supervisory employees in control of hazards and risks to minimize the potential for employee and client injuries, damage to client's property and damage or destruction to Accutest's facility.

The Health and Safety Officer is responsible for implementing the Program's elements and updating its contents as necessary. He also conducts periodic audits to monitor compliance and assess the program's effectiveness and is also responsible for creating and administering safety training for all new and existing employees.

The employee is responsible for following all safety rules established for their protection, the protection of others and the proper use of protective devices provided by the Company. The employee is also expected to comply with the requirements of the program at all times. Department Managers and Supervisors are responsible for ensuring the requirements of the Safety Program are practiced daily. The Company President retains the ultimate responsibility for the program design and implementation.

19.3 **Program Elements.** The Accutest Health and Safety Program consists of key program elements that compliment the company's health and safety objective. These elements form the essence of the health and safety policy and assure that the objectives of the program are achieved.

Safety Education and Training and Communication. Training is conducted to increase the staff's awareness of laboratory hazards and their knowledge of the safety

practices and procedures required to protect them from those hazards. It is also used to communicate general safety procedures required for safe operation in a chemical laboratory.

Initial health and safety training for new employees is conducted during orientation. The training focuses on the Accutest Safety and Health Program and includes specific training for the hazards that may be associated with the employees duties. Training is also conducted for all program elements focusing on general, acceptable, laboratory safety procedures. Targeted training is conducted to address hazards or safety procedures that are specific to individual employee's work assignments. All training activities are documented and archived in individual training folders. A health and safety training inventory is maintained in the training database.

Accutest Laboratories Southeast maintains personnel trained in HAZWOPER, DOT and HazMat operations, as well as respirator certified.

Safety Committee. The safety committee provides the employee with an opportunity to express their views and concerns on safety issues in a forum where those concerns will be addressed. This committee meets monthly to assure that the interests of the company and the well being of the employee are protected. They also serve as a catalyst for elevating the level of safety awareness among their peers.

***Hazard Identification and Communication*.** The hazard communication program enables employees to readily identify laboratory hazards and the procedures to protect themselves from those hazards. This program complies with OSHA's Hazard Communication Standard, Title 29 Code of Federal Regulations 1910.1200 that requires the company to adopt and adhere to the following key elements:

- Material Safety Data Sheets (MSDS) must be available to any employee wishing to view them,
- The Company must maintain a Hazardous Chemicals Inventory (by location), which is updated on an annual basis,
- Containers are properly labeled,
- All employees must be provided with annual Personal Protection, Hazard Communication and Right to Know training,

Chemical Hygiene Plan. The Chemical Hygiene Plan complies with the requirements of the Occupational Safety and Health Administration's Occupational Exposure to Hazardous Chemicals in the Laboratory Standard, 29 CFR 1910.1450. This plan establishes procedures, identifies safety equipment, personal protective equipment, and work practices that protect employees from the potential health hazards presented by hazardous chemicals in the laboratory if properly used and/or applied.

Emergency Action & Evacuation Plan. The Emergency Action and Evacuation Plan details the procedures used to protect and safeguard Accutest's employees and property during emergencies. Emergencies are defined as fires or explosions, gas leaks, building collapse, hazardous material spills, emergencies that immediately threaten life and health, bomb threats and natural disasters such as floods, hurricanes or tornadoes. The plan identifies and assigns responsibility for executing specific roles in situations requiring emergency action.

Lockout/Tagout Plan. Lockout/tagout procedures have been established to assure that laboratory employees and outside contractors take steps to render equipment inoperable and/or safe before conducting maintenance activities. The plan details the procedures for conducting maintenance on equipment that has the potential to unexpectedly energize, start up, or release energy or can be operated unexpectedly or accidentally resulting in serious injury to employees. The plan ensures that employees performing maintenance render the equipment safe through lock out or tag out procedures.

Personal Protection Policy. Policies have been implemented which detail the personal protection requirements for employees. The policy includes specifications regarding engineering controls, personal protective equipment (PPE), hazardous waste, chemical exposures, working with chemicals and safe work practices. Safety requirements specific to processes or equipment are reviewed with the department supervisor or the Health and Safety Officer before beginning operations.

Emergency Preparedness Plan. This plan identifies the actions to be taken by Accutest Laboratory's staff in the event of terrorism or terrorist actions, to ensure the safety of the employees and the facility. The plan describes the building security actions coinciding with the "Alert Condition", designated by the Department of Homeland Security.

Appendix I

Glossary of Terms

GLOSSARY OF TERMS

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accreditation: the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Audit: a systematic evaluation to determine the conformance to quantitative *and qualitative* specifications of some operational function or activity.

Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Method: a defined technical procedure for performing a calibration.

Calibration Standard: a substance or reference material used to calibrate an instrument.

Certified Reference Material (CRM): a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.

Chain of Custody: an unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples.

Clean Air Act: the enabling legislation in 42 U.S.C. 7401 *et seq.*, Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and to enforce them.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 *et seq*., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601*et seq.*, to eliminate the health and environmental threats posed by hazardous waste sites.

Confirmation: verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors or, additional cleanup procedures.

Conformance: an affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

Federal Water Pollution Control Act (Clean Water Act, CWA): the enabling legislation under 33 U.S.C. 1251 *et seq.*, Public Law 92-50086 Stat. 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance.

Field of Testing: NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff).

Holding Times (Maximum Allowable Holding Times) the maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Matrix: the component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated a potable or potential potable water source. Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake. Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue, Biota: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Matrix Spike (spiked sample or fortified sample): a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, NELAC, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater.

National Environmental Laboratory Accreditation Conference (NELAC): a voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP.

National Environmental Laboratory Accreditation Program (NELAP): the overall National Environmental Laboratory Accreditation Program of which NELAC is a part.

NELAC Standards: the plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference.

Performance Audit: the routine comparison of independently obtained *qualitative and quantitative* measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

PT Fields of Testing: NELAC's approach to offering proficiency testing by regulatory or environmental program, matrix type, and analyte.

Proficiency Testing: a means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Quality Manual: a document stating the management policies, objectives, principles, oganizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user.

Range: the difference between the minimum and the maximum of a set of values.

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

Reagent Blank (method reagent blank or method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval.

Requirement: denotes a mandatory specification; often designated by the term “shall”.

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the “Cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal.

Safe Drinking Water Act (SDWA): the enabling legislation, 42 USC 300f *et seq.* (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis.

Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture.

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends.

Validation: the process of substantiating specified performance criteria.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.
NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: A defined group of analysts that together perform the method analysis. Members of the group and their specific functions within the work cell must be fully documented. A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation, extraction, or analysis. The entire process is completed by a group of capable individuals, each member of the work cell demonstrates capability for each individual step in the method sequence.

STANDARD OPERATING PROCEDURE

Subject: Chain of Custody Documentation - Paper

1. PURPOSE

The purpose of this procedure is to provide the requirements for completion of written Chain of Custody (COC) documentation and to provide a suggested Chain of Custody Form for project use.

2. SCOPE

This procedure is applicable to all Shaw E & I efforts where samples are transferred among parties, including to off-site testing facilities. Adherence to this procedure is not required whenever the same individual/team is performing the sampling and testing within the same workday, and transfer to the testing process is being documented by other means, e.g. sampling and then field-screening in a mobile laboratory.

3. REFERENCES

- U.S. Environmental Protection Agency, 1986, *Test Methods for Evaluating Solid Waste; Physical/Chemical Methods, SW-846*, Third Edition.
- U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3.
- Shaw E & I, 2002, *Sampler's Training Course Handout*.

4. DEFINITIONS

- **Custody**—The legal term used to define the control and evidence traceability of an environmental sample. A sample is considered to be in an individual's custody when it is in actual physical possession of the person, is in view of the person, is locked in a container controlled by the person, or has been placed into a designated secure area by the person.
- **Chain of Custody Form**—A form used to document and track the custody and transfers of a sample from collection to analysis or placement in a designated secure area within the testing facility.
- **COC Continuation Page**—Additional page(s) that may be included with a Chain of Custody form. The continuation page(s) contain the information on additional samples contained within the *same* cooler/shipping container associated with the cooler/shipping container Chain of Custody form.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

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5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1 Documentation

All Chain of Custody documentation must be completed in indelible ink. All corrections must be performed using standard single-line cross-out methods, and the initials of the individual making the change must be included beside the corrected entry.

6.2 Continuation Pages

Continuation pages may be utilized for shipping containers/coolers with sufficient samples/sample containers that all of the lines of the Chain of Custody form are used before the documentation of the cooler/shipping container is complete. The number of pages in total must be filled out. *All samples entered onto a Continuation Page must be included in the same cooler/shipping container as those on the Chain of Custody form itself.*

6.3 Header Information

- Each Chain of Custody form must be assigned a unique Reference Document Number–use the Project/proposal number followed by a unique numeric sequence or current date (if only one cooler sent per day). Continuation Pages should contain the same Document Reference Number as the Chain of Custody form that they are associated with. The project team should maintain a log of Chain of Custody Reference Document Numbers.
- The page identifier and total page count section must be completed. Total pages include the Chain of Custody form and any attached Continuation Pages.
- Project number, name, and location information must be completed for all forms.
- If available, the laboratory Purchase Order Number should be included on the appropriate line.
- The name and phone number of the *Project Contact* should be included; the Project Contact should be a responsible individual that the laboratory may access to address analytical issues. This person is usually the analytical lead for the project.
- The *Shipment Date* should be provided on the applicable lines.
- If shipping by carrier, the *Waybill/Airbill Number* must be included. Note: couriers will not sign custody documents. Therefore, inclusion of the waybill/airbill number on the Chain of Custody is the *only* means of documenting the transfer to the carrier.
- Laboratory Destination and Contact information should be provided.

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- The Sampler(s) names should be provided on the appropriate line. This line should include all persons whose initials appear on any of the sample containers, to provide the laboratory a means of cross-referencing containers.
- The "Send Report To" information should be completed. If multiple reports/locations are needed, the information should be provided on a separate page included with the Chain of Custody documents.

6.4 Sample Information Section–Including on Continuation Page(s)

During actual sampling, each sample must be entered on the COC form at the time of collection in order to document possession. The sampler must not wait until sampling is completed before entering samples on the COC.

- Complete the *Sample ID Number* for each line. If there are multiple container types for a sample, use additional lines to indicate the needed information.
- Ensure that the *Sample Description* matches the description on the sample label–the laboratory will use this information for cross-referencing.
- Provide the *Collection Date* and *Time*. These must match those on the sample label and Field Logbook/Logsheets.
- Indicate whether the sample is a Grab or Composite sample.
- Indicate the *Matrix* of the sample. Use the Matrix Codes listed on the Chain of Custody form.
- Indicate the *Number of Containers* and the *Container Type*. If a sample has multiple container types, use multiple lines and cross-out the information spaces to the left of the container blocks. *Failure to do this may cause the laboratory to log-in each container type as a separate sample/lab-ID, resulting in a confused report and invoice.*
  - Alternatively, if each sample has the same number/type container types, use "various" in the *Container Type* block and provide detail in the *Special Instructions* section, e.g., "Each sample consists of one 16-oz jar, two pre-weighed VOC w/DI water, and one pre-weighed VOC w/Methanol."
- Check the appropriate *Preservative* box for each line/container type.
- Write in and check the *Analyses Requested* boxes for each line/container type. The appropriate method number (e.g., EPA Method 8260C) must be written as well as the method name.
- Indicate the *Turn-around Time Requested* for each sample.
- Use the *Special Instructions* section to provide important information to the laboratory, e.g., samples that may require dilution or samples that will need to be composited by the laboratory. This section may also be used to inform the laboratory of additional information contained in attachments to the Chain of Custody package.
- Circle the appropriate *QC/Data Package Level* requested.

6.5 Custody Transfer Section

- The first *Relinquished By* space must be completed by the individual who will either transfer the samples or seal the shipping container.

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- If the samples will be transferred to a courier, write the courier/carrier company in the *Received By* box and enter the Date and Time that the shipping container was closed.
- All other transfers must be performed in person, and the Relinquisher must witness the signing by the Receiver.
- A copy of the Chain of Custody form and all associated Continuation Pages should be maintained in the project files.

7. ATTACHMENTS

None.

8. FORMS

- Shaw E & I Chain of Custody Form
- Shaw E & I COC Continuation Page

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FS003.1_0

Ref. Document # _____

Page _____ of ____

Shaw E & I Chain of Custody Form

Project Number: _____

Project Name / Location: _____

Purchase Order #: _____

Shipment Date: _____

Waybill/Airbill Number: _____

Lab Destination: _____

Lab Contact Name / ph. #: _____

Project Contact: _____
(Name & phone #)

Send Report To: _____

Phone/Fax Number: _____

Address: _____

City: _____

Sampler's Name(s): _____

Sample ID Number	Sample Description	Collection Information			Matrix	# of containers	Container type	Preservative						Analyses Requested									Turn Around Time Requested
		Date	Time	G/C				HCL	NaOH	HNO_3	H_2SO_4	Ice											

Special Instructions:

QC/Data Package Level Required:
I II III IV/Project Specific: _____

Relinquished By:	Date:	Received By:	Date:
	Time:		Time:
Relinquished By:	Date:	Received By:	Date:
	Time:		Time:
Relinquished By:	Date:	Received By:	Date:
	Time:		Time:

G/C Codes

C = Composite G = Grab

Matrix Codes

DW = Drinking Water	SO =Soil
GW = Ground Water	SL = Sludge
WW = Waste Water	CP = Chip Samples
SW = Surface Water	WP = Wipe Samples
LIQ = Other Liquid	SOL = Other Solid
AS = Air Sample	SED = Sediment

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FS003.1_0

Shaw E & I COC Continuation Page

COC Ref. Document # _____ Page _____ of _____

Project Number: _____ Shipment Date: _____

Project Name / Location: _____

Sample ID Number	Sample Description	Collection Information			Matrix	# of containers	Container type	Preservative						Analyses Requested										Turn Around Time Requested
		Date	Time	G/C				HCL	NaOH	HNO_3	H_2SO_4	Ice												

STANDARD OPERATING PROCEDURE

Subject: Custody Seals

1. PURPOSE

The purpose of this procedure is to provide the requirements for completion and attachment of Custody Seals on environmental samples and shipping containers.

2. SCOPE

This procedure is applicable to all Shaw E & I efforts where sample legal defensibility and custody integrity is desired. Adherence to this procedure is not required whenever the same individual/team is performing the sampling and testing within the same workday, and transfer to the testing process is being documented by other means, i.e. sampling and then field-screening in a mobile laboratory.

3. REFERENCES

- U.S. Environmental Protection Agency, 1986, *Test Methods for Evaluating Solid Waste; Physical/Chemical Methods, SW-846*, Third Edition.
- U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans, EM200-1-3*
- Shaw E & I, 2002, Sampler's Training Course Handout.

4. DEFINITIONS

- **Custody**—The legal term used to define the control and evidence traceability of an environmental sample. A sample is considered to be in one's custody if it is in actual physical possession of the person, is in view of the person, has been locked in a container controlled by the person, or has been placed into a designated secure area by the person.
- **Custody Seal**—Commercially available thin strips of adhesive paper with write-in lines for the date/time and identification of the using party. Custody seals are placed over the caps of sample containers and along the cover seals of shipping containers as a means to detect tampering before arrival at the testing facility. All Shaw E & I strategic alliance laboratories provide Custody Seals in their sample container supply kits.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

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5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1 Completing the Custody Seal Information

- All Custody Seals must be completed in indelible ink. All corrections must be made using standard single-line cross-out methods, and the initials of the individual making the change must be included beside the corrected entry.
- Each Custody Seal attached must be completed by writing the *Date*, at a minimum, and signing with *full signature* by the person responsible for the sealing of the sample.
- If a space is provided, the *Time* should also be added.

6.2 Attaching the Custody Seals

Whenever possible, custody seals should be attached over the sample container lids during actual sampling and not when the samples are packaged for shipment. This will provide confidence in legal custody and will demonstrate non-tampering during the sample collection process.

Do not attach custody seals to VOC sample containers, as contamination may occur. For these samples, the custody seal should be used to seal the folded plastic zip bag that holds the sample containers.

- For sample jars, the completed Custody Seal should be placed across the top of the lid with the edges below the lid/jar interface and attached to the jar material. This will require the visible breaking of the seal in order to open the container.
- Sample coolers and shipping containers should have Custody Seals attached in such a manner that the seal extends lengthwise from the top edge of the lid to the side of the cooler/container.

7. ATTACHMENTS

None.

8. FORMS

None.

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STANDARD OPERATING PROCEDURE

Subject: Sample Labeling

1. PURPOSE

The purpose of this procedure is to provide the requirements for completion and attachment of sample labels on environmental sample containers.

2. SCOPE

This procedure is applicable to all Shaw E & I projects/proposals where samples will be collected.

3. REFERENCES

- U.S. Environmental Protection Agency, 1986, *Test Methods for Evaluating Solid Waste; Physical/Chemical Methods*, SW-846, Third Edition.
- U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3
- Shaw E & I, 2002, Sampler's Training Course Handout.

4. DEFINITIONS

- **Sample Label**—Any writing surface with an adhesive backing that can be used to document sample identification information. The sample label is attached to the sample container as a means of identification and, in some commercially available or laboratory-supplied containers, may be pre-attached. All Shaw E & I strategic alliance laboratories provide sample labels or pre-labeled containers in their sample container supply kits.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

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6. PROCEDURE

- All sample labels must be completed in indelible ink. All corrections must be performed using standard single-line cross-out methods, and the initials of the individual making the change must be included beside the corrected entry.
- Sample labels should be completed and attached as samples are collected. Do not wait until final packaging to attach and/or complete the sample labels.
- Sample labels must be attached to the non-sealing portion of the container. Do not place labels on or across sample container caps.
- If the laboratory has provided pre-labeled containers, make sure to fill one for each parameter set needed. Laboratory pre-labeled containers are often bar-coded and it is important to provide a complete container set for each sample.
- The following information must be recorded on the Sample Label:
  - Sample Identification Number
  - Date and Time collected
  - Initials of person(s) responsible for collection
- If a space is provided, the *Analysis Requested* should also be added.
- If a *Description* is provided, remember it must match that on the Chain of Custody form for cross-referencing purposes.
- Cover the completed and attached label with clear plastic tape to prevent bleeding of the ink if it becomes wetted. *Do **not** perform this step for pre-weighed VOC vials, as the final weight values will be influenced by the mass of the tape. Protect these containers by enclosing the rack/holder in a plastic bag within the cooler.*

7. ATTACHMENTS

None.

8. FORMS

None.

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STANDARD OPERATING PROCEDURE

Subject: Sample Homogenization

1. PURPOSE

The purpose of this procedure is to establish the method for homogenizing samples prior to containerization. Proper homogenization is very important because it helps ensure that sample aliquots are representative of the whole collected sample and helps minimize sampling error so that other errors included in the measurement process, such as laboratory sample preparation and test measurement, can be better assessed.

2. SCOPE

This procedure applies to Shaw Environmental & Infrastructure (Shaw E & I) personnel responsible for the collection of environmental samples. The sample matrix must be amenable to mixing. This SOP applies to the collection of samples that are to be tested for all analytes except volatile analytes.

3. REFERENCES

- American Society for Testing and Materials (ASTM), 1998, Reducing Samples of Aggregate to Testing Size, C702.
- U.S. Army Corps of Engineers, Requirements for the Preparation of Sampling and Analysis Plans, EM 200-1-3, Section E-2, Homogenizing Techniques.

4. DEFINITIONS

- **Homogenize**—The use of physical mixing motions to make a uniform sample matrix.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be sent to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

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6. PROCEDURE

Sampling equipment materials shall be selected so as to minimize contamination of samples. Sampling equipment shall be either new (never used previously), documented to have been decontaminated, or dedicated to each specific sampling point. Samples for organic constituent/compound analysis should be collected and mixed using non-reactive material such as glass or stainless steel bowls, trowels, and/or spoons. Samples for metals analysis should be collected and mixed using equipment made of stainless steel, glass, or Teflon®.

Certain types of solid matrices may not be amenable to mixing using conventional techniques. For example, certain solids may require grinding and thorough mixing to ensure that the analytes of interest within the sample are homogeneously distributed. It is extremely important that soil and sediment samples be homogenized to ensure that the entire sample is as representative as possible of the media being sampled.

6.1 Solid Samples

The following two methods are examples for homogenizing solid samples. Other homogenization techniques may be employed using approved standard methods such as ASTM C702, Reducing Samples of Aggregate to Testing Size.

6.1.1 Quartering

- Place the sample on a hard, clean, level surface such as a pan. If such a surface is too small for the desired quantity, a clean sheet of plastic may be used.
- Mix the solid material by turning the entire quantity over three times with a trowel or shovel. For the third time, shovel the material into a cone-shaped pile.
- Carefully press down on the apex of the pile to create a soil layer of uniform thickness and diameter.
- Divide the material in the sample pan or on the plastic into quarters

  Option 1

  - Mix each quarter individually
  - Then mix two quarters to form halves
  - Mix each formed half and then fill the appropriate sample jars/containers

  Option 2

  - Remove two diagonally opposite quarters including any fine material
  - Mix the remaining material, build it into a cone, and press down to flatten as before
  - Divide the flattened material into quarters, discard two diagonally opposing sections, and repeat
  - Repeat the process until only enough sample remains to fill the required containers and proceed to fill the sample jars.

6.1.2 Mixing in a Bowl

- Place the sample in a bowl. Samples for organic constituent/compound analysis should be mixed using bowls and stirrers made of glass or stainless steel, while samples for metals analysis should be mixed using equipment made of glass, stainless steel, or hard plastic. Make sure the bowl is large enough to accommodate the sample, with extra volume to allow for mixing the sample.

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- Mix the sample with the stirrer. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over. High moisture samples are more difficult to homogenize. Use an adequate mixing motion for as long as needed to determine by visual observation that the sample media has taken on a uniform appearance.

6.2 Liquid Samples

Most aqueous samples do not require homogenization since water is well mixed due to diffusion and bulk convection. If the sample matrix is a viscous liquid, semi-solid, or an aqueous one with suspended solids, the sample will require mixing.

Do **not** shake the sample and do not agitate the sample in **any** way if collecting for volatile parameters. Volatile sample containers should be either filled directly from the sample source or if transferring from a large container, such as an automatic sampler reservoir, filled first and **without agitation**.

For non-volatile parameters, mix either using an appropriate stirrer or by gentle swirling and then immediately transfer the material into the appropriate containers. The sample should be mixed frequently during the container-filling step, in particular if there are a large number of containers, so that the condition of the bulk sampled fluid will be approximately the same when each parameter-specific sample container is filled.

7. ATTACHMENTS

None.

8. FORMS

None.

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STANDARD OPERATING PROCEDURE

Subject: Shipping and Packaging of Non Hazardous Samples

1. PURPOSE

The purpose of this procedure is to provide general instructions in the packaging and shipping of non-hazardous samples. The primary use of this procedure is for the transportation of samples collected on site to be sent off site for physical, chemical, and/or radiological analysis.

2. SCOPE

This procedure applies to the shipping and packaging of all non-hazardous samples. Non-hazardous samples are those that do not meet any hazard class definitions found in 49 CFR 107-178, including materials designated as Class 9 materials and materials that represent Reportable Quantities (hazardous substances) and/or materials that are not classified as *Dangerous Goods* under current IATA regulations.

In general most soil, air, and aqueous samples, including those that are acid or caustic preserved do **not** qualify as *hazardous materials* or *dangerous goods*. An exception is methanolic soil VOC vials: these containers are flammable in any quantity and **must** be packaged, shipped, and declared as *Dangerous Goods* whenever transported by air.

The Class 9 "Environmentally Hazardous" designation should only be applied to samples if they are known or suspected (via screening) to contain a sufficient concentration of contaminant to pose a health and/ or environmental risk if spilled in transport. Samples for which screening has shown a potential hazard (i.e. flammability) or those that are derived from a known hazard, including a site/facility with confirmed contamination by an *infectious substance* must also be shipped in accordance with the applicable DOT/IATA requirements. Refer to Shaw E & I SOP FS013.

Improper shipment of hazardous materials, especially willful misrepresentation and shipment as non-hazardous materials, is a violation of federal law and is punishable by fines and possible imprisonment of the guilty parties. It is also a violation of Shaw E & I policy and can result in disciplinary action up to and including termination of employment.

3. REFERENCES

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3, Washington, D.C.
- U.S. Department of Transportation Regulations, 49 CFR Parts 108-178
- International Air Transport Association (IATA), *Dangerous Goods Regulations*, current edition.

4. DEFINITIONS

- **Cooler/Shipping Container**—Any hard-sided insulated container meeting DOT's or IATA's general packaging requirements.
- **Bubble Wrap**—Plastic sheeting with entrained air bubbles for protective packaging purposes.

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5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be sent to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1 Packaging

- Use tape and seal off the cooler drain on the inside and outside to prevent leakage.
- Place packing material on the bottom on the shipping container (cooler) to provide a soft impact surface.
- Place a large (30-55 gallon or equivalent) plastic bag into the cooler (to minimize possibility of leakage during transit).
- Starting with the largest glass containers, wrap each container with sufficient bubble wrap to ensure the best chance to prevent breakage of the container.
- Pack the largest glass containers in the bottom of the cooler, placing packing material between each of the containers to avoid breakage from bumping.
- Double-bag the ice (chips or cubes) in gallon- or quart-sized resealable plastic freezer bags and wedge the ice bags between the sample bottles.
- Add bagged ice across the top of the samples.
- When sufficiently full, seal the inner protective plastic bag, and place additional packing material on top of the bag to minimize shifting of containers during shipment.
- Tape a gallon-sized resealable plastic bag to the inside of the cooler lid, place the completed chain of custody document inside, and seal the bag shut.
- Tape the shipping container (cooler) shut using packing tape, duct tape, or other tear-resistant adhesive strips. Taping should be performed to ensure the lid cannot open during transport.
- Place a custody seal on two separate portions of the cooler, to provide evidence that the lid has not been opened prior to receipt by the intended recipient.

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6.2 Labeling

- A “This Side Up” arrow should be adhered to all sides of the cooler, especially ones without obvious handles.
- The name and address of the receiver and the shipper must be on the top of the cooler.
- The airbill must be attached to the top of the cooler.

6.3 Shipping Documentation

- A Cooler Shipment Checklist (Attachment 1) should be completed and kept in the project file.

7. ATTACHMENTS

- Attachment 1, Shaw E & I Cooler Shipment Checklist

8. FORMS

None.

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Attachment 1
Shaw E & I Cooler Shipment Checklist

Shaw™
Shaw E & I, Inc.

Project Name _____ Project Number _____
Address _____ Date _____ Time _____
City, State, Zip _____ Fax No. _____
Site Contact No. _____

SAMPLE CHECKLIST	YES	NO	COMMENTS
SAMPLE LIDS ARE TIGHT AND CUSTODY SEALS IN PLACE?	❒	❒	_____
ARE ALL SAMPLE NUMBERS, DATES, TIMES AND OTHER LABEL INFORMATION LEGIBLE AND COMPLETE?	❒	❒	_____
HAVE ALL SAMPLE NUMBERS, DATES, TIMES AND OTHER SAMPLING DATA BEEN LOGGED INTO THE SAMPLE LOG BOOK?	❒	❒	_____
DO SAMPLE NUMBERS AND SAMPLE DESCRIPTIONS ON THE LABELS MATCH THOSE ON THE COC?	❒	❒	_____
HAVE THE SAMPLES BEEN PROPERLY PRESERVED?	❒	❒	_____
HAVE THE CHAIN OF CUSTODIES BEEN FILLED OUT COMPLETELY AND CORRECTLY?	❒	❒	_____
DOES THE ANALYTICAL SPECIFIED ON THE COC MATCH THE ANALYTICAL SPECIFIED IN THE SCOPE OF WORK?	❒	❒	_____
HAVE THE COC'S BEEN PROPERLY SIGNED IN THE TRANSFER SECTION?	❒	❒	_____

PACKAGING CHECKLIST	YES	NO	COMMENTS
HAS EACH SAMPLE BEEN PLACED INTO AN INDIVIDUAL PLASTIC BAG?	❒	❒	_____
HAS THE DRAIN PLUG OF THE COOLER BEEN TAPED CLOSED WITH WATER PROFF TAPE FROM THE INSIDE?	❒	❒	_____
HAVE ALL THE SAMPLES BEEN PLACED INTO THE COOLER IN AN UPRIGHT POSITION?	❒	❒	_____
IS THERE ADEQUATE SPACING OF SAMPLES SO THAT THEY WILL NOT TOUCH DURING SHIPMENT?	❒	❒	_____
HAVE AN ADEQUATE NUMBER OF BLUE ICE PACKS OR WATER ICE BEEN PLACED AROUND AND ON TOP OF THE SAMPLE?	❒	❒	_____
HAS FRESH BLUE ICE OR WATER ICE BEEN ADDED TO THE COOLER THE DAY OF THE SHIPMENT?	❒	❒	_____
HAS THE COOLER BEEN FILLED WITH ADDITIONAL CUSHIONING MATERIAL?	❒	❒	_____
HAS THE COC BEEN PLACE IN A ZIPLOCK BAG AND TAPED TO THE INSIDE OF THE LID OF THE COOLER?	❒	❒	_____
HAVE CUSTODY SEALS BEEN PLACED ONTO THE LID?	❒	❒	_____
HAS THE COOLER BEEN LABELED "THIS SIDE UP"?	❒	❒	_____
IF REQUIRED, HAS THE COOLER BEEN LABELED WITH THE DOT PROPER SHIPPING NAME, UN NUMBER AND LABEL?	❒	❒	_____
HAS THE LABORATORY PERFORMING THE ANALYSES BEEN NOTIFIED OF THE SHIPMENT OF SAMPLES?	❒	❒	_____

PROBLEMS/RESOLUTIONS: _____

_____

_____

PREPARED BY: _____ SIGNATURE _____

STANDARD OPERATING PROCEDURE

Subject: Packaging and Shipping of DOT/IATA-Hazardous Samples

1. PURPOSE

The purpose of this procedure is to provide general instructions for packaging and shipping of hazardous samples, as defined by DOT and/or IATA, including Class 9 "Environmentally hazardous substances." The primary use of this procedure is for the transportation of samples collected on site to be sent off site for physical, chemical, biological (*infectious substance*), and/or radiological analysis in accordance with applicable laws and regulations and without destroying sample integrity.

2. SCOPE

This procedure applies to the packaging and shipping of all DOT/IATA-hazardous samples. Samples must be packaged and shipped as hazardous materials if they meet any of the hazard class definitions in 49 CFR 107-178, including Reportable Quantities, and/or if they can be classified as a *Dangerous Good* under IATA. All IATA classified materials designated for air transport, even in Limited Quantities, **must** be declared, packaged, and shipped as *Dangerous Goods.* Examples include methanolic VOC soil samples and any samples from a project/facility known to be impacted by an *infectious substance.*

Improper shipment of hazardous materials, especially willful misrepresentation and shipment as non-hazardous materials, is a violation of federal law and is punishable by fines and possible imprisonment of guilty parties. It is also a violation of Shaw E & I policy and can result in disciplinary action up to and including termination of employment.

3. REFERENCES

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3, Washington, D.C.
- U.S. Department of Transportation Regulations, 49 CFR Part 107-178
- *Dangerous Goods Regulations*, current edition, International Air Transport Association (IATA)

4. DEFINITIONS

- ***Dangerous Goods* Airbill**—Form required when offering *Dangerous Goods* as defined in IATA regulations for air transport. The "*Dangerous Goods* Airbill" must be completed and signed by a responsible and qualified person. Some carriers require a typed or computer-generated form.
- **Inner packaging**—Packaging in immediate contact with the hazardous materials to be shipped, such as a sample jar or vial.
- **Limited Quantity**—In the IATA Tables, the maximum total amount of a *Dangerous Good* that can be transported without using UN-specification containers, such as a non-UN tested cooler.

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- **Outer packaging**—Packaging into which one or more inner packages can be placed, such as a sturdy plastic cooler meeting general packaging requirements or a 5-gallon UN-specification plastic pail.
- **Performance-Oriented Packaging**—Packaging designed for and tested to be used for shipment of DOT-hazardous materials. Also known as "UN-specification" packaging.
- **Qualified person**—An individual with appropriate DOT/IATA Hazardous Materials training, including General Awareness, Function-Specific, and Safety training, necessary to properly classify samples as hazardous materials and to complete all subsequent shipping steps.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for the maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for recording information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

A Qualified Person **must** perform or oversee the classification, packaging, and completion of all related declaration and shipping papers. *It is a violation of federal law to pre-complete these documents and provide them to an unqualified person without providing minimal training to that person.* This training **must** be documented and may take the form of a verbal discussion, hands-on demonstration, or detailed written instructions, including a task-specific SOP, with review provided by the Qualified Person.

The basic packaging and shipping procedures are as follows:

- Determine the traits of the material to be shipped and compare them to the specific hazard class definitions in the appropriate regulations. If the material falls within one or more hazard class definitions, it is deemed "hazardous". Select the most accurate proper shipping name and packing group combination, and prepare the package according to the prescribed requirements for quantity limitations, authorized packaging, marking, labeling, and documentation.
- Check the current IATA regulations to make sure the carrier accepts the material(s) and/or does not have its own special requirements for shipment.
- If shipping multiple inner packages that each meet a separate hazard class definition, consult the "Separation and Segregation" table in the appropriate regulations for guidance on

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packaging and prepare as an over-pack with individual marking and labeling on the outer packaging.

- If shipping multiple inner packages that meet the same hazard class(es) but represent both solid and liquid matrices, prepare as an over-pack with individual marking and labeling on the outer packaging.
- If shipping hazardous material that meets more than one hazard class definition, check the hazard precedence table in the appropriate regulations to determine primary and subsidiary classes.

6.1 Additional Inner Packaging Requirements

- Place each sample container into a resealable plastic baggie.
- Fold over and tape the bag seal onto the sample jar to prevent the closure from unsealing.
- Several IATA packing instructions require containerizing of glass/plastic sample jars into a sealed primary receptacle such as a metal can before placing them into outer-packaging, i.e. the cooler.
  - Wrap the bagged sample container with bubble-wrap or other packing material to prevent breakage against the sides of the primary receptacle, and place it into the primary receptacle.
  - Seal the primary receptacle and label it with the Sample ID and any hazard information and place it into a plastic bag to protect the label.

6.2 Additional Outer Packaging Requirements

- Samples that in total qualify as Excepted Quantities or Limited Quantities do not require the use of UN-specification packaging and may be shipped in sturdy coolers, pails, or any packaging that meets general packaging requirements.
- Samples that do not qualify as Excepted Quantities or Limited Quantities require UN-specification packaging. For such samples that also require cooling to meet sample preservation requirements, UN-specification coolers are available from several Haz-Mat packaging vendors.
- If using a cooler of any kind, seal off the cooler drain on the inside and outside with tape to prevent leakage.
- Place cushioning and/or absorbent material on the bottom of the outer packaging to provide a soft impact surface.
- Place a plastic bag into the container (to minimize the possibility of leakage during transit).
- Wrap glass inner packagings with sufficient bubble wrap to ensure the best chance to prevent breakage of the container.
- For methanolic soil VOC vials, place vials into the supplied rack/holder or box and then place it into a tied off plastic bag to keep out moisture.
- Pack the largest inner packagings in the bottom of the container with cushioning material between each to avoid breakage from bumping.
- If cooling is required, double-bag the ice (chips or cubes) in gallon- or quart-size freezer Ziploc-type resealable plastic bags, and wedge the ice bags between the inner packages and/or primary receptacles. Also add bagged ice across the top of the samples/receptacles.

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- When sufficiently full, seal the plastic bag that lines the outer packaging, and place additional cushioning material on top of the bag to minimize shifting of contents during shipment.
- Tape a gallon Ziploc-type bag to the inside of the container lid, place the completed chain of custody document inside, and seal the bag shut.
- Tape the outer packaging closed using packing tape, duct tape, or other tear-resistant adhesive strips.
- Place a custody seal on two separate portions of the outer packaging to provide evidence that the lid remains sealed during transit.

6.3 Marking and Labeling

- If the package contains any liquids, orientation arrows must be applied to two opposite faces of the package (front and back or both ends).
- The proper shipping name, UN number, and all other required markings, as well as the appropriate hazard class label, must be placed on the same face of the package in close proximity to each other.
- Consignor and consignee information should appear on some face of the package in addition to appearing on the shipping papers that are enclosed in a pouch attached to the package.

6.4 Shipping Documentation

- If a sturdy cooler is used, whether UN-specification or not, complete a Cooler Shipment Checklist (see Attachment 1) and keep it in the project file.
- A Dangerous Goods Airbill must be completed, inserted into an adhesive pouch, and attached to the package in close proximity to the proper shipping name and hazard class label.
- Many carriers require a typed or computer-generated Dangerous Goods Airbill.
- If the Dangerous Goods Airbill has an area specifically designated for a “24-Hour Emergency Response” telephone number, insert “800-424-9300” into that space. If it does not, write “24-Hour Emergency Response Telephone Number: 800-424-9300” in the “Additional Handling Information” section of the airbill. Immediately following the telephone number, write “ERG-*xxx*,” where *xxx* is the 3-digit Emergency Response Guidebook page number that corresponds to the hazardous material being shipped.
- The shipper must sign the certification on the airbill.
- Prior to carrier pickup, a copy of the Dangerous Goods Airbill must be faxed to CHEMTREC at 703-741-6037 with a Shaw coversheet addressed to “ITCR.”

7. ATTACHMENTS

- Attachment 1, Shaw E & I Cooler Shipment Checklist

8. FORMS

None.

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Attachment 1
Shaw E & I Cooler Shipment Checklist

Shaw™ Shaw E & I, Inc.

Project Name _____ Project Number _____
Address _____ Date _____ Time _____
City, State, Zip _____ Fax No. _____
Site Contact No. _____

SAMPLE CHECKLIST	YES	NO	COMMENTS
SAMPLE LIDS ARE TIGHT AND CUSTODY SEALS IN PLACE?	❒	❒	_____
ARE ALL SAMPLE NUMBERS, DATES, TIMES AND OTHER LABEL INFORMATION LEGIBLE AND COMPLETE?	❒	❒	_____
HAVE ALL SAMPLE NUMBERS, DATES, TIMES AND OTHER SAMPLING DATA BEEN LOGGED INTO THE SAMPLE LOG BOOK?	❒	❒	_____
DO SAMPLE NUMBERS AND SAMPLE DESCRIPTIONS ON THE LABELS MATCH THOSE ON THE COC?	❒	❒	_____
HAVE THE SAMPLES BEEN PROPERLY PRESERVED?	❒	❒	_____
HAVE THE CHAIN OF CUSTODIES BEEN FILLED OUT COMPLETELY AND CORRECTLY?	❒	❒	_____
DOES THE ANALYTICAL SPECIFIED ON THE COC MATCH THE ANALYTICAL SPECIFIED IN THE SCOPE OF WORK?	❒	❒	_____
HAVE THE COC'S BEEN PROPERLY SIGNED IN THE TRANSFER SECTION?	❒	❒	_____

PACKAGING CHECKLIST	YES	NO	COMMENTS
HAS EACH SAMPLE BEEN PLACED INTO AN INDIVIDUAL PLASTIC BAG?	❒	❒	_____
HAS THE DRAIN PLUG OF THE COOLER BEEN TAPED CLOSED WITH WATER PROFF TAPE FROM THE INSIDE?	❒	❒	_____
HAVE ALL THE SAMPLES BEEN PLACED INTO THE COOLER IN AN UPRIGHT POSITION?	❒	❒	_____
IS THERE ADEQUATE SPACING OF SAMPLES SO THAT THEY WILL NOT TOUCH DURING SHIPMENT?	❒	❒	_____
HAVE AN ADEQUATE NUMBER OF BLUE ICE PACKS OR WATER ICE BEEN PLACED AROUND AND ON TOP OF THE SAMPLE?	❒	❒	_____
HAS FRESH BLUE ICE OR WATER ICE BEEN ADDED TO THE COOLER THE DAY OF THE SHIPMENT?	❒	❒	_____
HAS THE COOLER BEEN FILLED WITH ADDITIONAL CUSHIONING MATERIAL?	❒	❒	_____
HAS THE COC BEEN PLACE IN A ZIPLOCK BAG AND TAPED TO THE INSIDE OF THE LID OF THE COOLER?	❒	❒	_____
HAVE CUSTODY SEALS BEEN PLACED ONTO THE LID?	❒	❒	_____
HAS THE COOLER BEEN LABELED "THIS SIDE UP"?	❒	❒	_____
IF REQUIRED, HAS THE COOLER BEEN LABELED WITH THE DOT PROPER SHIPPING NAME, UN NUMBER AND LABEL?	❒	❒	_____
HAS THE LABORATORY PERFORMING THE ANALYSES BEEN NOTIFIED OF THE SHIPMENT OF SAMPLES?	❒	❒	_____

PROBLEMS/RESOLUTIONS: _____

_____

_____

PREPARED BY: _____ SIGNATURE _____

STANDARD OPERATING PROCEDURE

Subject: Decontamination of Contact Sampling Equipment

1. PURPOSE

This procedure is intended to provide minimal guidelines for the decontamination of contact sampling equipment. Contact sampling equipment is equipment that comes in direct contact with the sample or the portion of a sample that will undergo chemical analyses or physical testing.

2. SCOPE

This procedure applies to all instances where non-disposable direct contact sampling equipment is utilized for sample collection and no project-specific procedure is in place. This procedure is not intended to address decontamination of peristaltic or other sampling pumps and tubing. The steps outlined in this procedure must be executed between each distinct sample data point.

3. REFERENCES

- U.S. Environmental Protection Agency, Region 4, 2001, *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, 980 College Station Road, Athens, Georgia. November.
- US Army Corp of Engineers, Washington, D.C., 2001, Requirements for the Preparation of Sampling and Analysis Plans (EM-200-1-3), February.

4. DEFINITIONS

- **Soap**—A standard brand of phosphate-free laboratory detergent, such as Liquinox®.
- **Organic Desorbing Agent**—A solvent used for removing organic compounds. The specific solvent would depend upon the type of organic compound to be removed. See Attachment 1 for recommendations.
- **Inorganic Desorbing Agent**—An acid solution for use in removing trace metal compounds. The specific acid solution would depend upon the type of inorganic compound to be removed. See Attachment 1 for recommendations.
- **Tap water**—Water obtained from any municipal water treatment system. An untreated potable water supply can be used as a substitute for tap water if the water does not contain the constituents of concern.
- **Distilled Water**—Water that has been purified via distillation. Distilled water can be purchased in most stores and is acceptable as a final rinse in non-trace analytical decontamination processes. Examples would include disposal profiling, HazCat, and other gross screening applications.
- **Analyte-free water**—Water that has been treated by passing through a standard deionizing resin column, and for organics either distillation or activated carbon units. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds, and/or no detectable organic compounds (i.e., at or above analytical detection limits). Type I and Type II Reagent Grade Water meet this definition as does most laboratory-supplied blank water.

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5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be sent to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

Wear appropriate eye protection including safety goggles when working with corrosive liquids, especially when diluting concentrated materials to create low-percentage solutions and follow all project Health and Safety requirements. Decontamination wastes are to be recovered and handled as impacted project waste materials and must be disposed of in accordance with regulatory requirements.

A decontamination area should be established. Implements can either be immersed in a 5-gallon bucket containing each solution/rinse or the solutions can be contained in hand-held units made of an inert and compatible material; such as a Teflon™ wash bottle. The analyte-free water needs to be placed in a container that will be free of any compounds of concern.

Consult Attachment 1 for the decontamination solutions/solvents appropriate to the task. The minimum steps for decontamination are as follows:

1. Remove particulate matter and other surface debris by brushing and/or dipping in the soap solution.
2. Rinse thoroughly with tap water.
3. If necessary, rinse with other applicable solutions/solvents. If hexane is used, be sure to follow it with isopropyl alcohol to allow for the final water rinses to properly mix and contact the surface.
4. Final rinse three times to make sure all residual solutions/solvents are removed.
5. Place decontaminated equipment on a clean surface appropriate for the compounds of concern and allow to air dry.

7. ATTACHMENTS

- Attachment 1, Recommended Decontamination Procedures.

8. FORMS

None.

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Attachment 1
Recommended Decontamination Procedures

Compound	Detergent Wash	Tap Water	Inorganic Desorbing Agent	Tap Water	Organic Desorbing Agent[1]	Final Water Rinse[4]	Air Dry
Organic Constituents							
Volatile Organic Compounds	✓	✓			Methanol Purge & Trap grade	✓	✓
Base Neutrals/Acid Extractables/PCBs/Pesticides	✓	✓			Hexane followed by Isopropyl Alcohol	✓	✓
Organic Bases[2]	✓	✓	1% nitric acid	✓	Isopropyl Alcohol	✓	✓
Organic Acids[3]	✓	✓	1% nitric acid		Isopropyl Alcohol	✓	✓
Inorganic Constituents							
Trace Metals and Radio Isotopes	✓	✓	10% Nitric acid -Trace metals grade	✓		✓	✓
Cations/Anions	✓	✓				✓	✓
Acidic Compounds	✓	✓				✓	✓
Basic Compounds (caustic)	✓	✓	1% nitric acid	✓		✓	✓

1 – All organic solvents must be Pesticide Grade or better. The selection of appropriate solvent rinses should first consider if a *known or suspected* contaminant requires removal from sampling equipment. Secondly, identify whether the subsequent analytical protocol would be impacted by the proposed solvent or an impurity thereof (e.g., residual acetone present in isopropyl alcohol would be measured with certain volatile organics analysis).
2 - Organic bases include amines, hydrazines.
3 - Organic acids include phenols, thiols, nitro and sulfonic compounds.
4- Use a grade of water appropriate to the application. For trace level analysis this must be Analyte Free Water. For non-trace applications store-bought distilled water is sufficient

Adapted from: Appendix E, Requirements for the Preparation of Sampling and Analysis Plans (EM-200-1-3), February 2001. US Army Corp of Engineers, Washington, D.C.

Revision 1- 3/2006

STANDARD OPERATING PROCEDURE

Subject: Trowel/Spoon Surface Soil Sampling

1. PURPOSE

The purpose of this document is to provide the methods and procedure for sampling of surface soils using trowels or spoons. Trowels or spoons can be used when matrices are composed of relatively soft and non-cemented formations and to depths of up to 12 inches into the ground surface, dependent on site conditions. Samples for Volatile Organic Compound (VOC) analysis should not be collected via trowel or spoon method. However, a trowel or spoon may be utilized to penetrate to and expose the undisturbed material at the desired depth for sampling by more applicable methods.

2. SCOPE

This procedure is applicable to all Shaw E & I projects where surface soil samples will be collected via trowel or spoon methods.

3. REFERENCES

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, Appendix C, SectionC.6, EM200-1-3, Washington, D.C.

4. DEFINITIONS

- **Trowel**—A sample collection device with a curved and pointed metal blade attached to a handle. All trace environmental samples should be collected using stainless steel blades.
- **Spoon**—A sample collection device with a round metal blade attached to a handle.
- **Surface Soil**—Soil that is removed from the surface no greater than 6 inches below grade after removing vegetation, rocks, twigs, etc.
- **Weathered Soil**—The top ⅛ to ¼ inch of soil impacted by heat from sun, rain, or foot traffic that could evaporate, dilute, or otherwise deposit contaminants from an adjacent location, thereby misrepresenting the actual soil characteristic.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for the maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

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For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1 Equipment

- Decontaminated trowel or spoon, stainless steel construction for trace environmental sampling. If samples will be collected at depth (0-6 inches), the trowel or spoon will require decontamination prior to collection of the targeted-depth sample. Alternatively, a different trowel or spoon can be used to remove the material to the targeted depth and the sample collected using a clean dedicated trowel or spoon.
- Engineers rule or stiff measuring tape
- Decontaminated stainless steel mixing bowl

6.2 Sampling

1. Don a pair of clean gloves.
2. If desired, place plastic sheeting around the targeted location to keep sampled material in place. Use a knife to cut an access hole for the sample location.
3. Remove any surficial debris (e.g. vegetation, rocks, twigs) from the sample location and surrounding area until the soil is exposed. Once exposed, the soil surface is designated as “at grade,” or 0 inches.
4. Use a trowel to scrape and remove the top 1/8 to 1/4 inch of weathered soil. (A spoon can be interchanged with trowel).
5. If collecting a sample that includes VOC analysis, collect the VOC sample aliquot first following more applicable methods.
6. With a new trowel, place the point of the blade on the ground. While holding the handle of the trowel, partially rotate the blade in a clockwise/counter-clockwise motion while pushing at a downward angle until the blade is inserted to the required depth or the blade is nearly covered. Be certain that the trowel is not inserted to a depth where the soil will touch the handle or other non-stainless steel portion of the trowel or the sampler’s hand.
7. With a prying motion lift up the trowel with soil on the blade and place soil into the stainless steel mixing bowl.
8. Repeat steps 6 and 7 until the required depth of soil is placed into the mixing bowl.
9. Measure the depth of the sample location with a rule or tape to verify the sampling depth and record in the field logbook.
10. Homogenize the non-VOC sample and transfer the sample directly into the sample container(s). Cap the sample container(s), label the containers, complete the documentation, and place the containers into the sample cooler.

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7. ATTACHMENTS

None.

8. FORMS

None.

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STANDARD OPERATING PROCEDURE

Subject: Soil Sampling using a Soil Probe or Core-Type Sampler

1. PURPOSE

The purpose of this document is to provide the methods and procedure for sampling of soils and other solids using soil probes and core-type devices. These samplers can be used when matrices are composed of relatively soft and non-cemented formations. They are utilized to collect near-surface core samples and can also be placed into boreholes at specified depths. Soil probe/corer samplers provide an intact depth-specific sample for geotechnical, chemical, radiological, or biological analysis

2. SCOPE

This procedure is applicable to all Shaw E & I projects where soil samples will be collected via hand-operated soil probe/corer methods and no project-specific procedure exists. This procedure is not applicable to drilling or direct push methods.

3. REFERENCES

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, EM-200-1-3.
- American Society for Testing and Materials, *Standard Practice for Soil Investigation and Sampling by Auger Borings*, D1452-80 (re-approved 2000).
- U.S. Environmental Protection Agency, 1994, *Soil Sampling*, EPA/ERT SOP 2012, November.

4. DEFINITIONS

- **Soil Corer**—A sample collection device consisting of extension rods, a T-handle, and a sampling head. The sampling head is a thin-walled two-piece metal tube, split lengthwise, into which a metal or plastic sleeve is placed. The tube halves are held together with screw-locked ends, the bottom one having a point. The sleeve fills with material as the sampler is forced downward, allowing for an undisturbed core to be collected
- **Soil Probe**—A core sample collection device consisting of a thin-walled metal tube with a cutting edge on the bottom. The tube is cut-away from its tip to approximately one-third of the way to its top to allow material to enter. The top of a soil probe is removable, and a plastic or metal sleeve is inserted through the top and is held in place by the reduced diameter of the tube at the top of the cutout. Soil probes can be attached to extension rods and T-handles or may be of one-length construction. Samples collected from a soil probe are almost always submitted to the laboratory intact.

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5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

The sampling procedure is as follows:

1. Assemble the sampler by inserting the appropriate sample tube and close the ends. If using extension rods, attach the sampler by its top to the bottom rod. Attach the T-handle either to the extension rod or directly to the sampler head.

2. If desired, place plastic sheeting around the targeted location to keep sampled material in place. Use a knife to cut an access hole for the sample location.

3. Don a pair of clean sample gloves.

4. Remove any surficial debris (e.g. vegetation, rocks, twigs) from the sample location and surrounding area.

5. If the sample will be collected from a depth beyond the surface, use a hand-auger to remove the overburden and expose the “target” sample depth. Measure the depth of the hole with a rule or stiff tape to confirm that the target depth has been reached.

6. If the sampling depth is below where the sampling device can be seen while sampling, measure the distance from the tip to top of the sampler and mark the extension rod at this distance plus the depth of the hole with tape as a reference.

7. Change sample gloves just prior to collecting the sample, especially if an auger was used to expose the target depth

8. To collect the sample using a Soil Corer, place the point of the assembled corer directly on the ground or in the auger hole and, while holding it vertical, push straight down into the soil. Do not twist. A slide hammer may be required for hard or stiff materials.

9. A Soil Probe should be placed into the location and pushed downward with a twisting motion to allow the cutting edge to work. Do not drive or hammer the sampler as this will damage the cutting tip.

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10. Continue to force the sampler downward until either the top joint is touching the ground or the reference mark is even with the top of the auger hole. This will ensure that the entire sleeve is filled with material.

11. Extract the sampler by pulling upward with a slight rocking or twisting motion until the head is fully out of the hole.

12. Wipe the sampler head with a cloth or towel and remove it from the T-handle or extension rod.

13. Disassemble the sampler and remove the sleeve. Also perform any field screening desired (e.g., PID screen).

14. For a Soil Probe sample, the sleeve will most likely be submitted intact. Wipe the outside of the sleeve and use a knife to cut off any material sticking from the end so that the ends are even. Place Teflon™ tape over the ends and cap both ends. Be sure to label the top and bottom of the sample interval.

15. A Soil Corer sample may be submitted intact, especially for geotechnical parameters. If this is the case, wipe the outside of the sleeve and use a knife to cut off any material sticking from the end so that the ends are even. Place Teflon™ tape over the ends and cap, labeling the sleeve and marking the top and bottom of the sample interval.

16. If the Soil Corer sample will be aliquotted into other containers, use a knife to split the sleeve lengthwise and remove the top section to expose the sample.

17. If sampling for Volatile Organic Compounds (VOCs), collect sample aliquots from the intact core first using an EnCore™ or other syringe-type device.

18. Place the remaining material directly into sample jars or into a mixing bowl for homogenization and containerization. Cap the sample container(s), label it/them, complete the documentation, and place the sample container(s) into the sample cooler.

19. Decontaminate the sampler.

7. ATTACHMENTS

None.

8. FORMS

None.

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STANDARD OPERATING PROCEDURE

Subject: Sampling for VOCs in Soil—Sealed-Cap (EnCore®) Sampler

1. PURPOSE

The purpose of this procedure is to provide general information about the procedure for using the Disposable EnCore® Sampler or other similar sealed-cap soil VOC samplers. These samplers are used to obtain and ship soil and clay samples for volatile organic compound (VOC) analysis, including GRO, in accordance with SW-846 Method 5035A and other related protocols.

2. SCOPE

This procedure applies to all instances where soils require sampling and shipment for VOC analysis using no headspace methods, including samples collected from drilling cores.

This procedure should not be used if collecting samples for pre-weighed vial VOC methods.

This procedure and these types of samplers are not applicable to non-elastic soils and non-compactable materials, such as loose sand, rocky soils, and gravel. Such materials should be sampled using alternative methods.

3. REFERENCES

3.1 U.S. Environmental Protection Agency, 1996, Method 5035A "Closed-System Purge and Trap for Volatile Organics in Soil and Waste," *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, Revised December, Update IV.

3.2 En Novative Technologies, Inc., "Disposable EnCore® Sampler Sampling Procedures—Using the EnCore® T-Handle," guide supplied with each case of samplers.

4. DEFINITIONS

4.1 Sealed-Cap VOC Sampler—A single-use volumetric sampling system designed to collect, store, and deliver soil samples for VOC methods that require no headspace.

4.2 EnCore® Sampler—A form of Sealed-Cap VOC Sampler designed and marketed by En Novative Technologies, Inc., of Green Bay, WI. The cartridges come in two sizes for sample volumes of approximately 25 or 5 grams.

4.3 EnCore® T-Handle—The specially machined holder for the EnCore® sampler sold separately by En Novative Technologies, Inc. The T-Handle provides the leverage needed to push the sampler into the soil, and should be used along with the sampler. In cases where a T-Handle is not available, it is possible though not recommended to grip the sampler by the sides, away from its sealing surfaces, with a pair of pliers or similar implement and push it into the soil.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be sent to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

For each sample location collected and for each applicable field or laboratory QC sample, a total of three samples will need to be obtained, as follows:

1. Open the sealed bag containing the sampler and, if using an EnCore®, push the plunger down until the small O-ring rests against the tabs.

2. If using an EnCore®, the locking lever on the T-Handle must be depressed as the cartridge is inserted. Line up the slots on the cartridge with the locking pins in the T-Handle. Plunger end first, insert cartridge into T-Handle with locking tabs aligned and twist the cartridge clockwise locking it in place.

3. Prepare the surface by removing grass, sticks, and other matter to allow the sampler to penetrate the intended location.

   - For hard pan soils and clays or excavations, scrape away the top few inches of the material to expose virgin and penetrable soil/clay for sampling.

   - When sampling subsurface cores, split the core cover lengthwise or push the core from the coring tube to expose the core and sample from points along the core.

4. Insert the cartridge device into the material being sampled with a downward twisting motion until full. If using the EnCore® system, observe the appropriate hole in the T-Handle and continue to push the sampler into the material being sampled until the small O-ring on the plunger is visible in the viewing hole (5g-bottom hole, 25g-top hole).

5. Withdraw sampling device from medium and use a fresh tissue to wipe off excess material from the outside of the cartridge body and especially the O-rings. If soil is protruding from the tube, carefully slice it off even with the open end using a clean knife or spatula.

6. For the EnCore® system, while the cartridge is still on the T-Handle, turn the T-Handle until the cartridge is facing upward and place the cap over the cartridge with the locking arms aligned with the flat surfaces of the locking ridge. Then gently push the cap onto the cartridge with even pressure, and twist the cap maintaining downward pressure until the arms lock against the ridge. Non-EnCore® systems must be sealed according to the manufacturer's instructions.

7. Inspect the cap and seal making sure that the cap is seated over the cartridge squarely and evenly. For the EnCore® system, both arms must be locked over the ridge or an imperfect seal will result, compromising the data.
8. Remove the capped sampler from its holder
9. For the EnCore® system, lock the plunger by rotating the plunger rod counterclockwise until the wings rest against the tabs
10. Complete and attach the label and seal the cartridge in the provided sampler bag.
11. Repeat steps 1-10 for the other two cartridges needed for the sample location, collecting each cartridge from undisturbed material as close as possible to the original location.
12. Place all three cartridges in the same bag and then label the outside of the bag per the project requirements
13. Place the labeled bag into a cooler with the project-required coolant (ice or dry ice).
14. Complete all required documentation and ship to the laboratory per the project plans

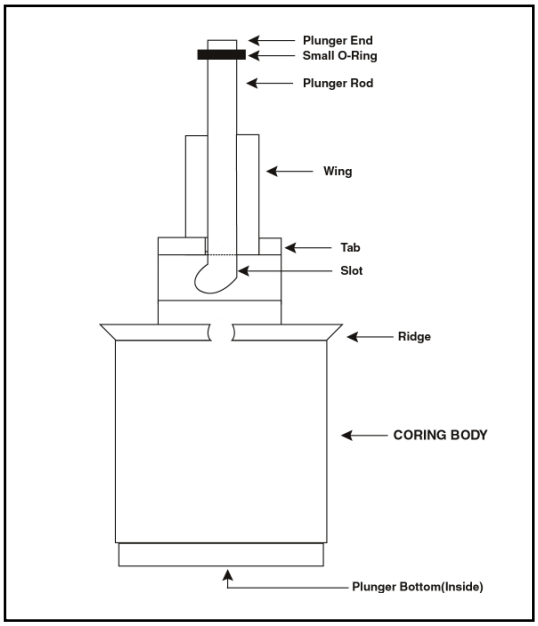
7. ATTACHMENTS

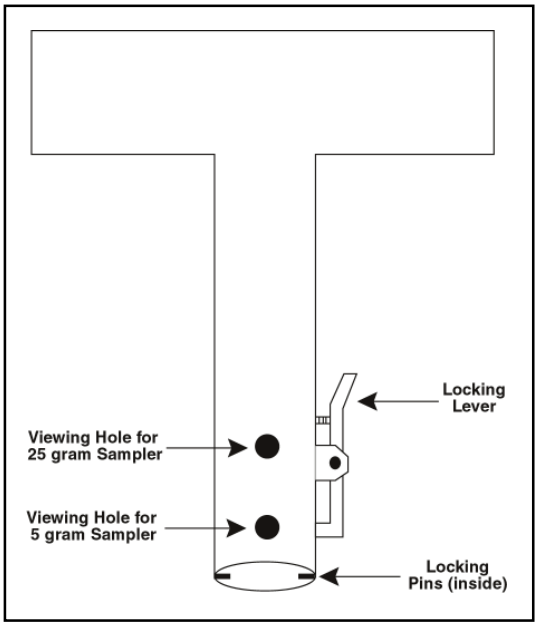
- **Attachment 1**—EnCore® Sampler Figures (from En Novative Technologies, Inc. web-site: http://www.ennovativetech.com).

8. FORMS

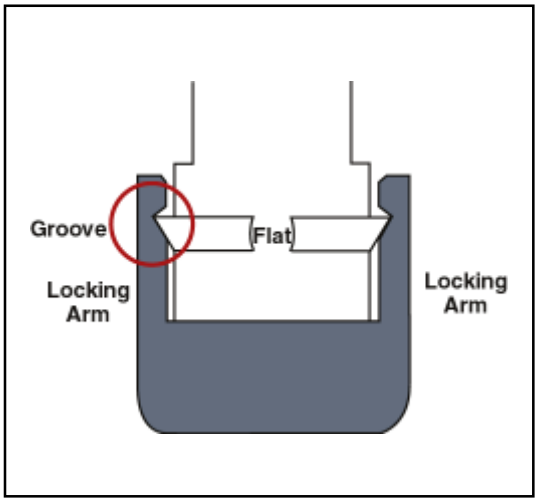
None.

ATTACHMENT 1
ENCORE® SAMPLER FIGURES

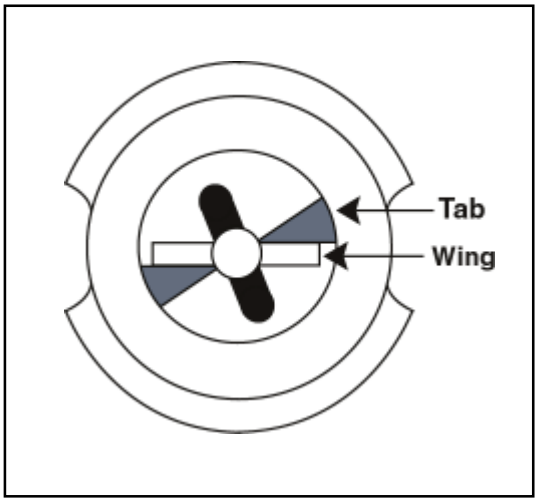




T-Handle



Sampler Properly Capped



Plunger Top view

STANDARD OPERATING PROCEDURE

Subject: Surface Water Sampling

1. PURPOSE

The purpose of this document is to provide methods, procedures, and guidance for sampling of surface waters or liquids in lakes, streams, pits, sumps, lagoons, and similar reservoirs for environmental analysis.

2. SCOPE

This procedure is applicable to all Shaw E & I projects where surface water sampling will be performed and where no project/program plan or procedure is in place to direct those activities.

The procedure presents two methods of sampling: direct immersion of sampling containers and use of a pond sampler.

3. REFERENCES

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, EM 200-1-3, Appendix C, Washington, D.C.
- U.S. Environmental Protection Agency, 1994, *Surface Water Sampling,* EPA/ERT SOP 2013.

4. DEFINITIONS

- **Pond Sampler**—A type of liquid sampling device consisting of an adjustable aluminum or fiberglass pole with an adjustable clamp to hold a container on one end. The pole allows for grab samples to be obtained at distances as far as 10 to 12 feet from the edge of the source without the need to contact the medium.
- **Grab Sample**—A single sample representative of a specific location at a given point in time.

5. RESPONSIBILITIES

5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager or designee is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations,

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reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

Safety Note: *Surface water sampling can sometimes require the use of boats for access into or across bodies of water. Observe all boating safety considerations in the HASP including donning of proper life jackets. If sampling from a bank, do not overreach; use a Pond Sampler whenever possible and do not attempt to remove the container from the clamp while still in contact or close proximity to the water body. Do not wade into a water body unless the depth is well known, currents are flowing at a safe speed, appropriate personnel have determined it is safe, and a spotter is available.*

6.1 Direct Immersion

The following procedure shall be used for direct immersion sampling:

- Don a pair of clean gloves.
- Obtain the required sample container(s).
- If entering the water body, always do so with as little bottom disturbance as possible and wait for the water around the planned sampling area to return to its undisturbed state (clarity) before sampling.
- Collect each liquid sample by slowly submerging the sample container with minimal surface disturbance. If sampling in a stream or current, make sure the open end of the sample container is pointed upstream.
- Withdraw the container from the liquid with minimal disturbance; cap and wipe the outside of the container with a towel or cloth.
- If collecting samples for VOC analysis, make sure that the VOA vial is slightly overfilled before capping, and check for bubbles or trapped air by inverting. If the sample integrity is compromised, discard the sample and repeat the collection process.
- Complete all required documentation, and place the sample containers into a cooler or other specified container.

6.2 Pond Sampler

The following procedure shall be used for sampling with a pond sampler:

- Don a pair of clean gloves.
- Place plastic sheeting around the area where the sampler will be emptied.
- Assemble the pond sampler and secure the sample container or collection jar/bottle/beaker in the adjustable clamp.
- If entering the water body, always do so with as little bottom disturbance as possible and wait for the water around the planned sampling area to return to its undisturbed state (clarity) before sampling.
- Collect each liquid sample by extending the container end outward and slowly submerging the sample container while holding the Pond Sampler handle with minimal surface disturbance. If sampling in a stream or current, make sure the open end is pointed upstream.

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- Retrieve the container with minimal surface disturbance, retract any extensions, transport the sample while still attached to the emptying area, and remove it from the clamp.
- Alternatively, if sampling with a partner, the partner can remove the collection container from the clamp and carry it to the transfer area.
- If the container is the one to be used for the sample, remove it from the clamp, cap, and label.
- If the sampler was used to collect a fill container, remove the lid(s) from the required sample containers and slowly transfer the sample into the appropriate containers; cap and label each one.
- Fill containers for VOC analysis first, making sure that the VOA vial is slightly overfilled before capping, and check for bubbles or trapped air by inverting. If the sample integrity is compromised, discard the sample and repeat the vial filling process.
- Complete all required documentation, and place the samples into a cooler or other specified container.
- After each use (i.e. between sample locations), the pond sampler must be disassembled and decontaminated, especially at the clamp area.

Sample jars or beakers are attached to the pole using the clamps for collecting the sample. With a pond sampler device, sample jars can be attached directly to the sample pole and the sample directly filled into the sample jar, or a sampling beaker can be attached to the pole and the collected sample then transferred to an appropriate sample jar. If sample jars are filled directly, they should be wiped clean prior to being placed in the cooler for shipment. If sampling beakers are used, they can be disposed of or decontaminated prior to reuse.

7. ATTACHMENTS

None.

8. FORMS

None.

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STANDARD OPERATING PROCEDURE

Subject: Jar Headspace Screening

1. PURPOSE

The purpose of this procedure is to provide the basic methods and guidance for volatile organic compound (VOC) screening of environmental samples using jar headspace techniques. Jar headspace analytical screening can be used to provide field data regarding the presence or absence of VOC vapors in environmental samples.

Field screening for VOC compounds can be useful for such environmental characterization purposes as discovery of site VOC contamination; selection of field samples to submit to a laboratory for analyses; selection of surface soil sampling locations; selection of boring locations; placement of groundwater monitoring wells; soil cutting (from drilling operations) screening for disposal characterization purposes; and purge water (from well purging/sampling tasks) screening for disposal characterization purposes.

2. SCOPE

This SOP is applicable to all Shaw E & I projects where VOC screening by the jar headspace method is employed. This procedure serves as general guidance on the proper methods for conducting jar headspace analytical screening. Users should always consult state-specific, program-specific, or project-specific requirements to ensure compliance with requirements when performing the activities of this SOP.

3. REFERENCES

- Massachusetts Department of Environmental Protection, *Interim Remediation Waste Management Policy for Petroleum Contaminated Soils*, #WCS-94-400.

4. DEFINITIONS

- **Flame Ionization Detector (FID)**—An organic compound detector based upon the ionization in a flame of compounds containing carbon-hydrogen bonds. The FID is a gross screening tool that detects the total organic content of the introduced sample. Its response is lower to halogenated compounds, and it will not respond to compounds lacking a carbon-hydrogen bond.

- **Ionization Potential (IP)**—The amount of energy required to remove an electron from the outer shell of a molecule or atom. The resultant molecule or atom will be a positively charged cation.

- **Photo Ionization Detector (PID)**—An organic compound detection system based upon the ionization of compounds via UV-radiation. A PID will respond only to those compounds with IP values less than or equal to the output of the UV-lamp. As such it is an indicator of aromatic and conjugated organic compounds. PID response is lower for halogenated compounds. PID systems are available with either a 10.2 or 11.7ev lamp.

5. RESPONSIBILITIES

5.1. Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

5.2. Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for recording information in sufficient detail to provide objective documentation (i.e., checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

6. PROCEDURE

6.1. Equipment

The equipment to be used for jar headspace screening includes the following:

- Field logbook(s)
- Volatile organic compound vapor meter (PID or FID)
- Field Data Forms - See Section 8
- Indelible markers
- Wide-mouth glass jars (16-oz preferred, 8 oz minimum size)
- Stainless steel laboratory spoons
- Aluminum foil

6.2. Field Gas Chromatography

If field GC is being employed, the following additional equipment will be required:

- Gas Chromatograph system
- Calibration standards and materials
- Gas-tight syringes

6.3. Procedure Steps

- Calibrate field screening equipment in accordance with the manufacturer's instructions and/or project-specific requirements.
- Obtain a soil sample from the sampling device (split spoon, spatula, shovel, etc.) immediately after removal from the ground. Groundwater samples can be collected from the inside of auger

flights using a disposable bailer. In order to reduce loss of the volatiles, take care to minimize handling of the sample and exposure to the air during transfer to the jar.

- Half-fill a clean glass jar with the sample to be analyzed. Quickly cover each open top with one or two sheets of clean aluminum foil and subsequently apply screw caps to tightly seal the jars. Sixteen-ounce (approximately 500 mL) soil or "mason" type jars are preferred. Do not use jars with less than 8 oz. (approximately 250 mL) total capacity.
- Allow sealed jar to sit for at least 10 minutes. Vigorously shake jars for 15 seconds at the beginning of the headspace development period. Where ambient temperatures are below 32°F (0°C), sample bottles should be placed within a heated vehicle or building for the prescribed period to allow the volatilization process to occur.
- Remove screw/lid and expose foil seal. Puncture foil seal with instrument sampling probe into the jar. Keep probe tip sufficiently above the media surface to avoid uptake of water droplets or soil particulates into the sample probe.
- As an alternative collection method or when using a field GC, use a gas-tight syringe to withdraw a measured volume of the headspace and inject into the probe inlet or calibrated GC.
- Following probe insertion through the foil seal and/or sample injection into the probe, the maximum (non-GC) instrument response should occur between 2 and 5 seconds. Record the highest meter response as the jar headspace concentration in the field log book or sheet. For GC analysis, determine and record the response/concentration of the target compound(s)
- Perform Duplicate QC and evaluate in accordance with the project plans
- Dispose of all wastes, including screened samples, in accordance with the project plans

7. ATTACHMENTS

- Attachment 1, Ionization Potentials for Common Volatile Contaminants

8. FORMS

- Jar Headspace Screening Results Log

Attachment 1
Ionization Potentials for Common Volatile Contaminants

Analyte	IP (eV)	Analyte type
Acetone	9.69	AAK
Acrolein	10.10	AAK
Allyl alcohol	9.67	SDO
Benzene	9.245	AC
Bromochloromethane (I.S.)	10.77	AH
Bromoform	10.51	AH
Bromomethane	10.53	AH
n-Butanol	10.04	AAETS
2-Butanone (MEK)	9.53	AAK
Carbon disulfide	10.08	AAETS
Carbon tetrachloride	11.47	AH
Chlorobenzene	9.07	AC
Chlorodibromomethane	10.59	AH
Chloroethane	10.98	AH
Chloroform	11.42	AH
Chloromethane	11.28	AH
1,2-Dibromoethane	10.19	AH
Dibromomethane	10.49	AH
1,2-Dichlorobenzene	9.07	AH
1,3-Dichlorobenzene	9.12	AH
1,4-Dichlorobenzene	8.94	AH
Dichlorodifluoromethane	12.31	AH
1,2-Dichloroethane	11.12	AH
trans-1,2-Dichloroethene	9.66	AH
1,2-Dichloropropane	10.87	AH
Diethyl ether	9.53	AAETS
Ethanol	10.48	AAETS
Ethyl acetate	10.11	AAE
Ethyl benzene	8.76	AC
Ethylene oxide	10.565	MM
2-Hexanone	9.34	AAK
Iodomethane	9.54	AH
Isopropylbenzene	8.69	AH
Methane	12.98	PC
Methanol	10.85	AAETS
Methylene chloride (DCM)	11.35	AH
4-Methyl-2-pentanone (MIBK)	9.30	AAK
Naphthalene	8.12	AC
Nitrobenzene	9.92	AC
Pyridine	9.32	HM
Styrene	8.47	AC

Analyte	IP (eV)	Analyte type
Tetrachloroethene	9.32	SDO
Toluene	8.82	AC
Trichloroethene	9.45	SDO
Trichlorofluoromethane	11.77	AH
Vinyl acetate	9.19	SDO
Vinyl chloride	9.995	SDO
o-Xylene	8.56	AC
m-Xylene	8.56	AC
p-Xylene	8.445	AC

PC = Paraffins and Cycloparaffins

AH = Alkyl Halides

AAETS = Aliphatic Alcohol, Ether, Thiol, and Sulfides

AAK = Aliphatic Aldehydes and Ketones

AAE = Aliphatic Acids and Esters

SDO = Some Derivatives of Olefins

HM = Heterocyclic Molecules

AC = Aromatic Compounds

MM = Miscellaneous Molecules

Draft Final

Explosives Safety Submission

Solid Waste Management Unit 4 (SWMU 4) and Area of Concern "R" (AOC R)

Former Naval Ammunition Support Detachment (NASD) Vieques, Puerto Rico

CTO Task Order 002
LANTDIV CLEAN III Program
Contract N62470-02-D-3052
Revision 1, August 2005

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Appendix

A Risk Evaluation – Mechanized Vegetation Removal Process

Acronyms and Abbreviations

AFWTF	Atlantic Fleet Weapons Training Facility
AOC	Area of Concern
ARS	Archives Record Search
CAL	Caliber
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLEAN	Comprehensive Long-Term Environmental Action Navy
CRP	Community Relations Plan
CSM	Conceptual Model
DDT	Deflagration-to-Detonation Transition
DERP	Defense Environmental Restoration Program
DoD	Department of Defense
DOI	Department of Interior
EBS	Environmental Baseline Survey
ECA	Eastern Conservation Area
EIS	Environmental Impact Statement
EMA	Eastern Maneuver Area
EOD	Explosive Ordnance Disposal
°F	Degrees Fahrenheit
FMFLANT	Fleet Marine Force, Atlantic
Ft	Feet/Foot
GIS	Geographic Information System
GPS	Global Positioning System
IAS	Initial Assessment Study
LANTDIV	Atlantic Division
LAW	Light Anti-Tank Weapon
LDGP	Low Drag General Purpose Bomb
LIA	Live Impact Area
LUMP	Land Use Management Plan
MC	Munitions Constituents
MEC	Munitions and Explosives of Concern
MPPEH	Material Potentially Presenting an Explosive Hazard
MSL	Mean Sea Level
NASD	Naval Ammunition Support Detachment
NAVFACENGCOM	Naval Facilities Engineering Command
Navy	U.S. Navy
NEW	Net Explosive Weight

NGFS	Naval Gunfire Support
NRHP	National Register of Historic Places
NTR	Navy Technical Representative
NWS	National Wildlife Service
OB	Open Burn
OD	Open Detonation
ODUSD	Office of the Deputy Under Secretary of Defense
OP	Observation Post
PA	Preliminary Assessment
PAOC	Potential Area of Concern
PI	Photo-Identified
PPE	Personal Protective Equipment
PRA	Preliminary Range Assessment
PRASA	Puerto Rico Aqueduct and Sewer Authority
PREQB	Puerto Rico Environmental Quality Board
QA	Quality Assurance
QC	Quality Control
RAC	Risk Assessment Code
RCRA	Resource Conservation and Recovery Act
RFA	RCRA Facility Assessment
RFI	RCRA Facility Investigation
SIA	Surface Impact Area
SWMU	Solid Waste Management Unit
TM	Technical Memorandum
UNITAS	United International Antisubmarine Warfare (Exercise)
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
USGS	U.S. Geological Survey
UXO	Unexploded Ordnance
VNTR	Vieques Naval Training Range

SECTION 1

Project Summary

1.1 Introduction

This Explosive Safety Submission (ESS) supplements work plans associated with the collection of environmental samples in the vicinity of 16 Munitions Response Sites (MRS) located at SWMU 4 at the Former NASD. These 16 MRS were initially identified as potential OB/OD pits

Additionally, this ESS addresses MEC avoidance and site inspection activities in the vicinity of AOC R in the Former NASD. Approximately 12 MEC items were discovered near AOC R in August 2004. The disposal/demilitarization of these items, and any similar items found during the site inspection is also covered under this ESS.

Finally, if resources are available and time permits, limited UXO removal actions will be conducted in areas near SWMU 4 most likely used by the public. These areas include beaches and foot trails used to access the beaches. This phase would be limited to detector-assisted surface clearance and associated MEC removal and disposal.

The ESS was prepared by CH2M HILL for the Naval Facilities Engineering Command (NAVFACENGCOM) Atlantic Division (LANTDIV) under Navy Contract N62470-02-D-3052, Navy Comprehensive Long-Term Environmental Action Navy (CLEAN), District III, Contract Task Order 002.

This ESS has been prepared in accordance with the requirements presented in Department of Defense (DoD) 6055.9-STD; DoD Ammunition and Explosives Safety Standards, Chapter 12; NAVSEA OP 5 Volume 1, the guidance presented in Department of Defense Explosive Safety Board (DDESB) Memorandum of 27 February 1998, and the NOSSA Guidelines for Preparing an Explosives Safety Submission, (NOSSA Instruction 8020.15, 8 March 2004).

1.2 Conclusions from Previous Studies and Reports

SWMU 4 covers an area of approximately 40 acres on the Former NASD. Within SWMU 4, 16 sites have been identified as potential OB/OD pits. U.S. Navy personnel disposed of unserviceable/excess (retrograde) munitions at SWMU 4 from 1969 to 1979. Unexploded ordnance from training exercises on the VNTR was also disposed of at SWMU 4.

A three-phased remedial investigation was conducted by CH2M HILL between January 2002 and July 2003. Nearly 2,000 MEC items were encountered during that investigation. A detailed inventory of MEC items recovered during the remedial investigation is included in Table 3-1 of this document.

AOC R was the subject of an Environmental Baseline Survey in 2000 and a Preliminary Assessment/Site Investigation in 2002. Neither of these studies addressed potential MEC

contamination. In August 2004, NAVFAC Atlantic submitted a Notification Report to NOSSA describing the MEC items discovered near AOC R.

1.3 Suspected Type and Amount of Ordnance Contamination

Residual munitions and munitions components from incomplete open burn procedures and "kick-out" munitions from open detonation operations are strewn throughout the site at SWMU 4.

The type of MEC expected to be encountered may include: unserviceable/expired munitions unsuccessfully or partially treated at the OB/OD pits. UXO brought to SWMU 4 for disposal following training exercises on the VNTR may also be encountered.

The assortment of ordnance treated at the OB/OD pits on the SWMU 4 site will likely result in a wide variety of MEC items found. A more detailed description of the types of ordnance anticipated to be encountered is provided in Section 3 of this ESS.

MEC items discovered at AOC R appear to be concrete-filled, MK 80-series LDGP bombs. Some of these bomb were modified for use as static-display or decorative items. Munitions related accessories, such as ammunition cans and aircraft-mounted rocket launchers, have also been found in a ravine just outside the boundary of AOC R. The ravine was apparently used as a disposal site for unwanted scrap metal.

1.4 Planned Future Use of Property

The US Fish and Wildlife Service (FWS) is in the process of preparing the Comprehensive Conservation Plan/Environmental Impact Statement for the Vieques National Wildlife Refuge. This document will provide long-term guidance for land management and use.

A preliminary land use plan for SWMU 4 has been developed by FWS and includes wildlife observation towers, nature trails, and public beaches along the southern boundaries of the site.

The Navy will maintain control of AOC R until all requirements of environmental characterization and cleanup are satisfied. However, AOC R will ultimately be returned to the Municipality of Vieques for development.

1.5 Remediation Goals

Remediation goals will be established following publication of the final version of the FWS land use plan. For this investigation environmental samples will be taken in and around the potential OB/OD pits at SWMU 4. If resources and scheduling permit, MEC removal actions may be conducted on beach areas and access paths to the beach areas identified for public access.

Remediation goals for AOC R will be determine based on results of site inspection and assessment as well as the land-use plans of the Municipality of Vieques.

1.6 Type of Munitions Response Action

The Puerto Rico Environmental Quality Board (EQB) provides oversight for work on the Former NASD. Although the site has not been officially placed under the domain of CERCLA or RCRA, the EQB has generally followed CERCLA guidelines for projects conducted near the Former NASD Area (west Vieques).

The munitions response action at SWMU 4 is primarily MEC avoidance to facilitate the safe collection of environmental samples. Future actions may include surface MEC removal, digital geophysical mapping (DGM), and subsurface MEC removal.

Actions at AOC R will include demilitarization and disposal of MEC items discovered in August 2004. An inspection and assessment for additional MEC items will also be conducted in AOC R. The inspection/assessment will consist of a magnetometer-assisted surface sweep.

Demilitarization/disposal procedures will be performed on any additional MEC items found. The requirement for future subsurface MEC investigation of AOC R will be based largely upon the results of the surface inspection/assessment.

SECTION 2

Maps

2.1 Regional Map

Figure 2-1: Regional Location Map

2.2 Activity Maps

Figure 2-2: Munitions Response Areas

Figure 2-3: Munitions Response Sites (Potential OB/OD Pits)

Figure 2-4: Munitions Response Sites (Beach Areas)

Figure 2-5: Munitions Response Sites (AOC R)

Figure 2-6: DOI FWS Preliminary Land Use Plan

Figure 2-7: SWMU 4 (OB/OD Pits) Intentional/Unintentional Detonation Exclusion Zones

Figure 2-8: SWMU 4 (Beach Areas) Intentional/Unintentional Detonation Exclusion Zones

Figure 2-9: AOC R Intentional/Unintentional Detonation Exclusion Zones

Figure 2-10: Security Fences SWMU 4 MEC Remedial Investigation

Figure 2-11: Traffic Control Points AOC R MEC Remedial Investigation

2.3 Q-D Map

Figure 2-12: Building 239 (Earth Covered Magazine) Explosive Safety Arcs

2.4 Soil & Groundwater Sampling Maps

Figure 2-13: Proposed Sampling Locations in SWMU 4

Figure 2-14: Proposed Sampling Locations in AOC R

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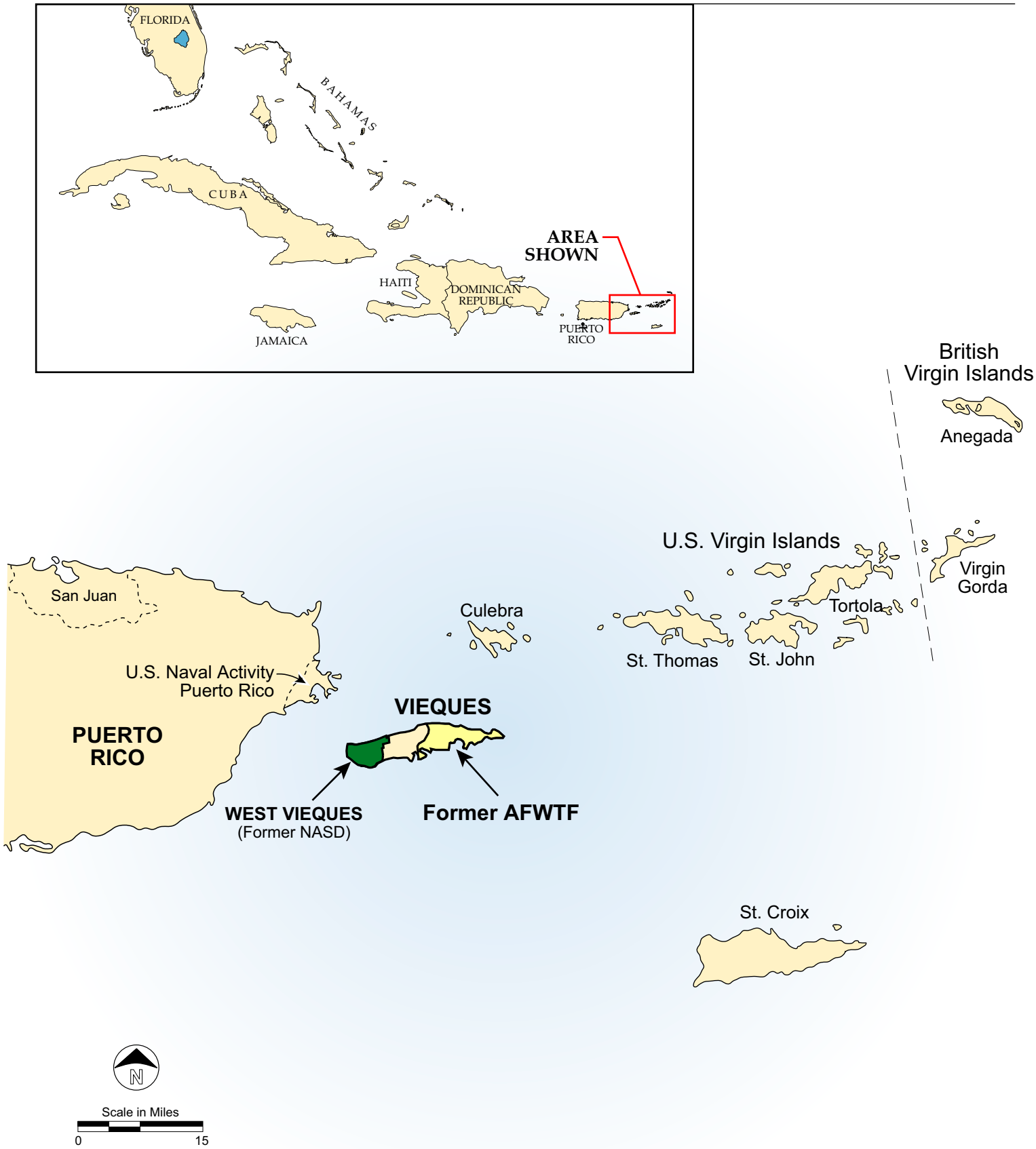

Figure 2-1
Regional Location Map
Former Atlantic Fleet Weapons Training Facility, Vieques, Puerto Rico

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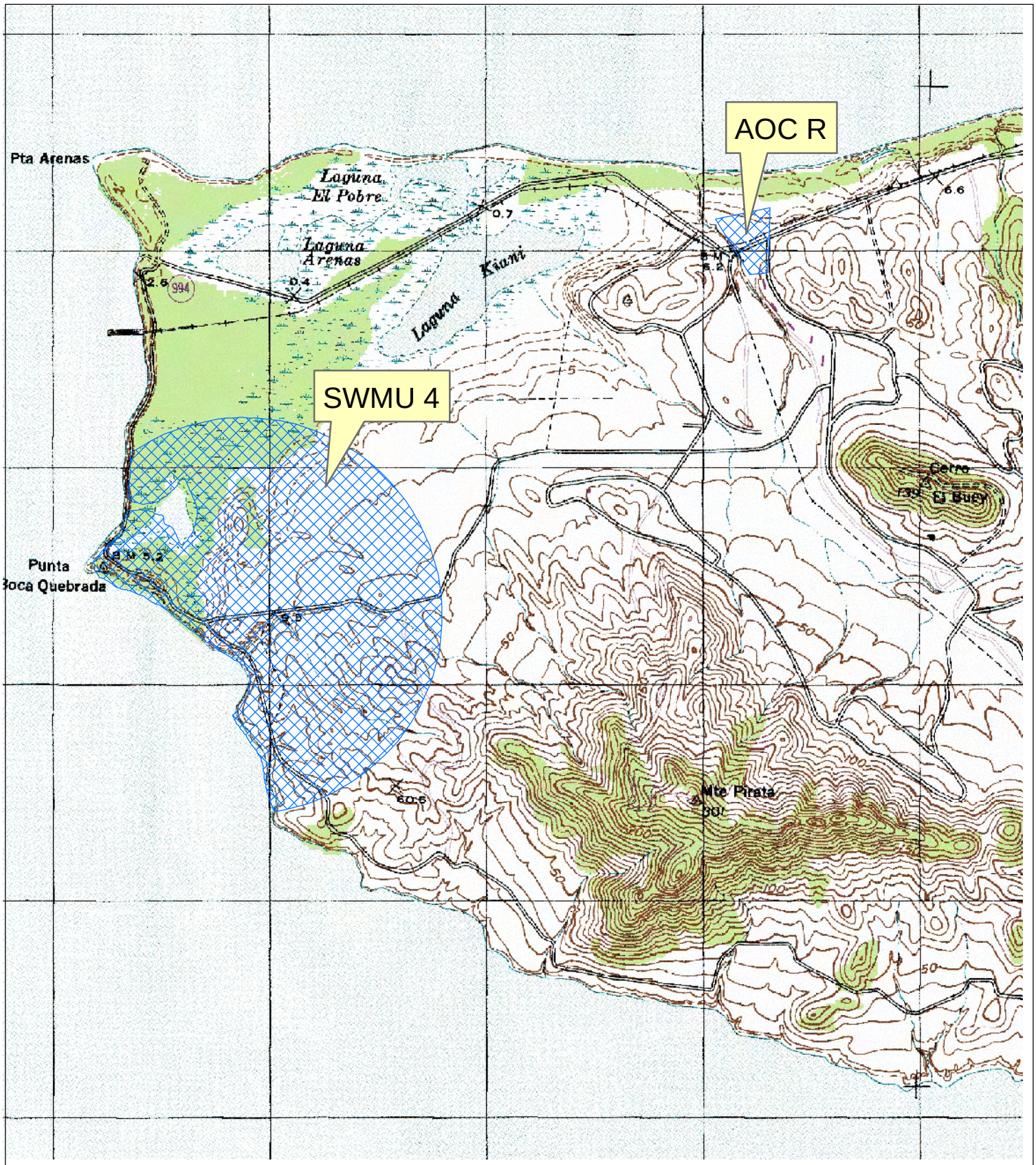

N

0 1,000 2,000 Feet

Figure 2-2
Munitions Response Areas
Former NASD, West Vieques, Puerto Rico

CH2M HILL

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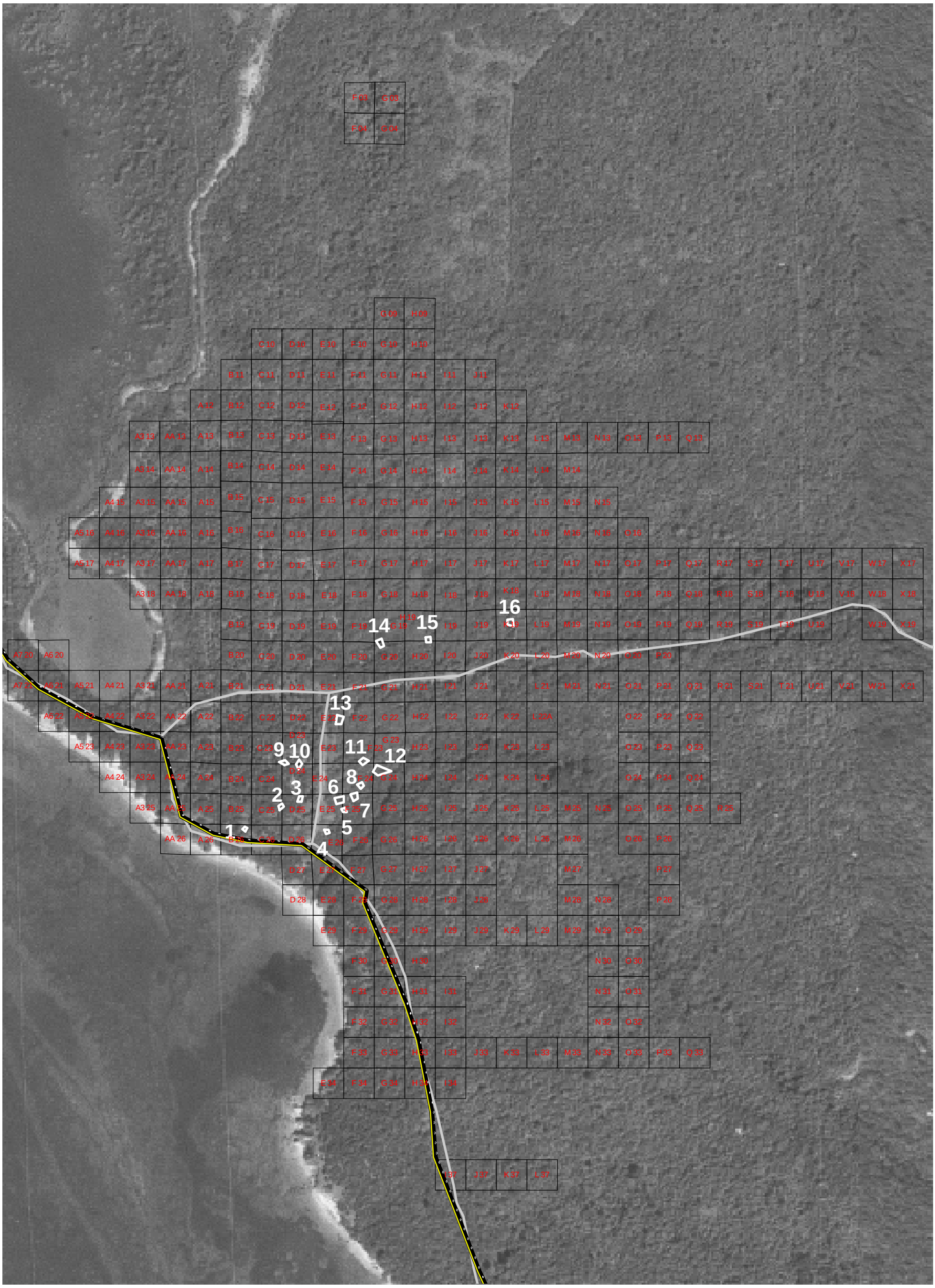

Fence Line

N
W
E
S

0 150 300 Feet

Figure 2-3
Munitions Response Sites
Potential OB/OD Pits
Former NASD, West Vieques, Puerto Rico

CH2MHILL

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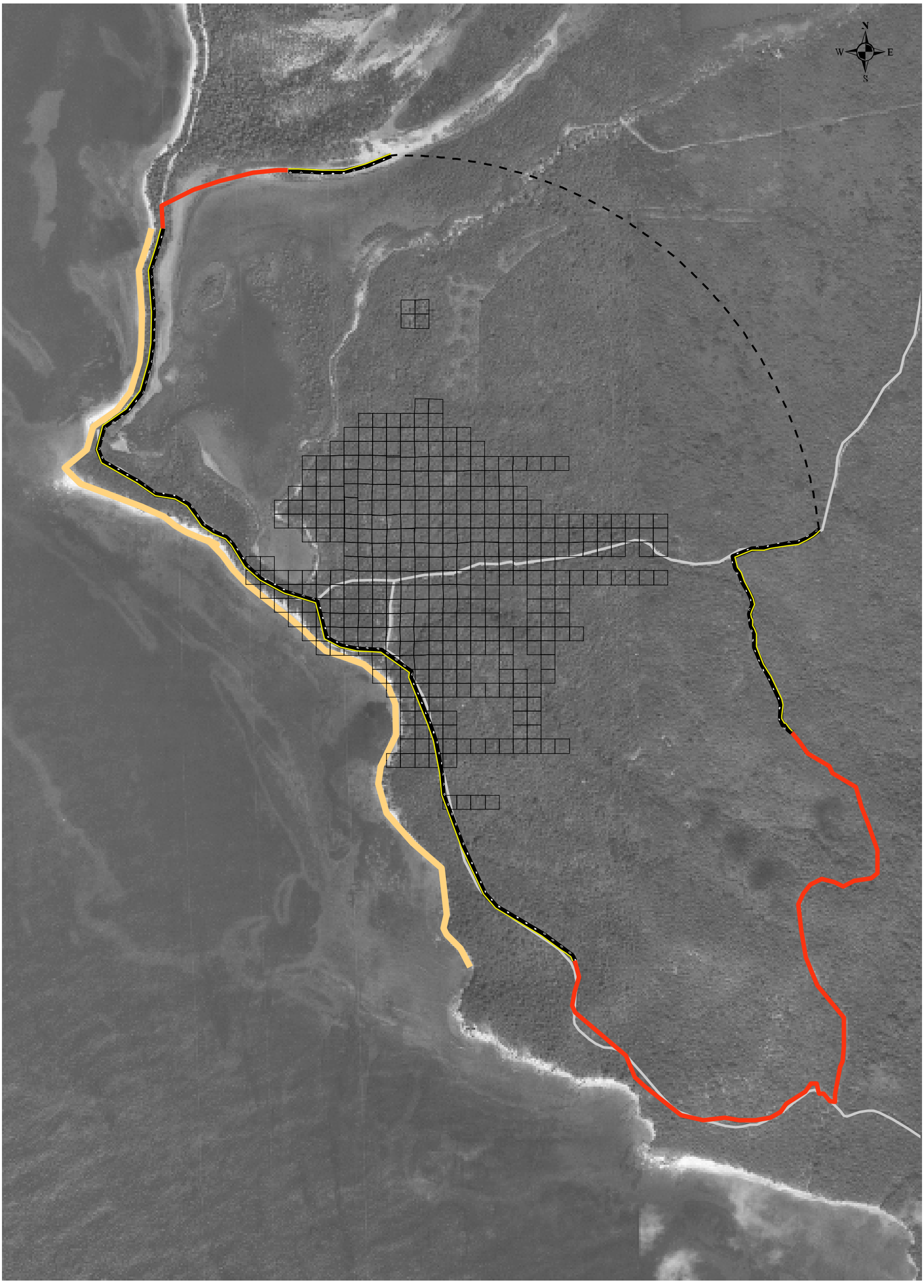

Legend

- OB/OD BOUNDARY
- FENCE LINE INSIDE OB/OD AREA
- FENCE LINE OUTSIDE OB/OD AREA
- Beach Areas

Feet
0 500 1,000

Figure 2-4
Munitions Response Sites
Beach Areas
Former NASD, West Vieques, Puerto Rico

CH2MHILL

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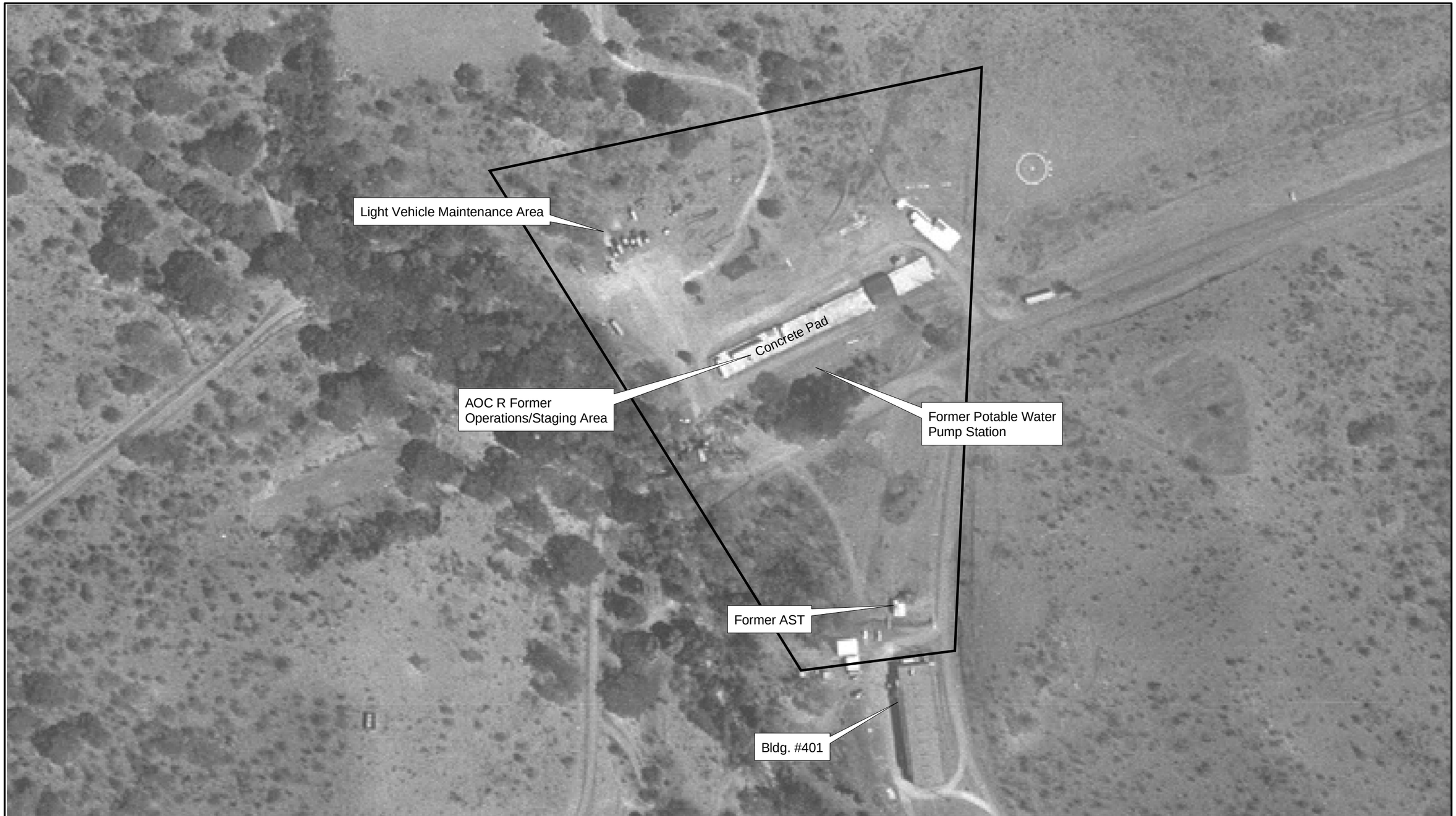


Source: 1967 Aerial

Access Restriction Boundary

N
W E
S

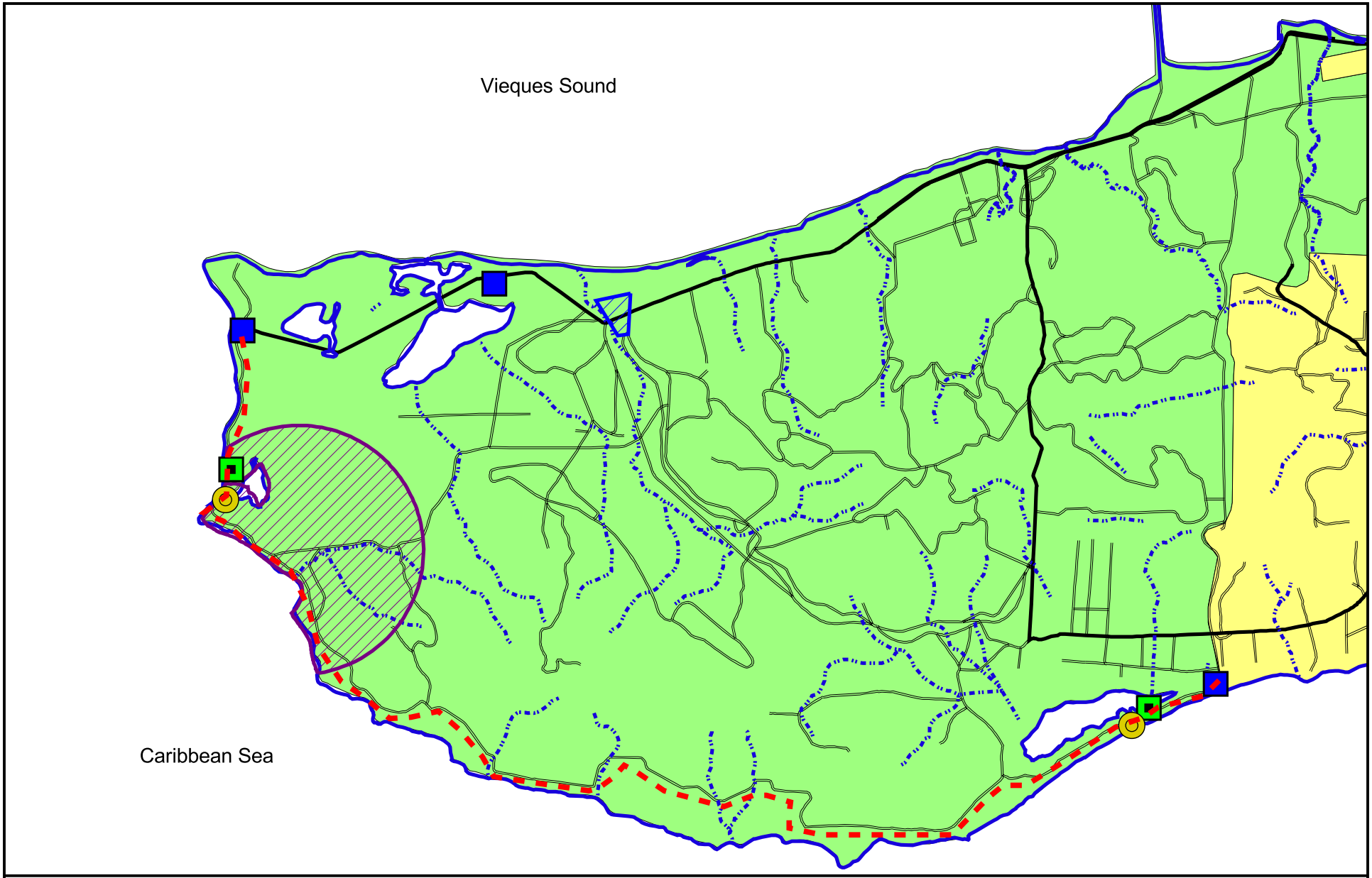
0 150 300 Feet

Figure 2-5
AOC R Munitions Response Site
Former NASD, Vieques, Puerto Rico

CH2MHILL

File Path: \\vbo20009180\usb drive\18gis\vieques\figures\mxd\figure2-5_aocr_mrs.mxd, Date: 9/28/04, User: munwin

NOTE: Original figure created in color



WOODEN BRIDGE
WILDLIFE OBSERVATION TOWERS
TRAILHEAD PARKING LOTS
FOOT TRAIL
SWMU 4
AOC R
STREAMS
MAJOR ROADS
MINOR ROADS

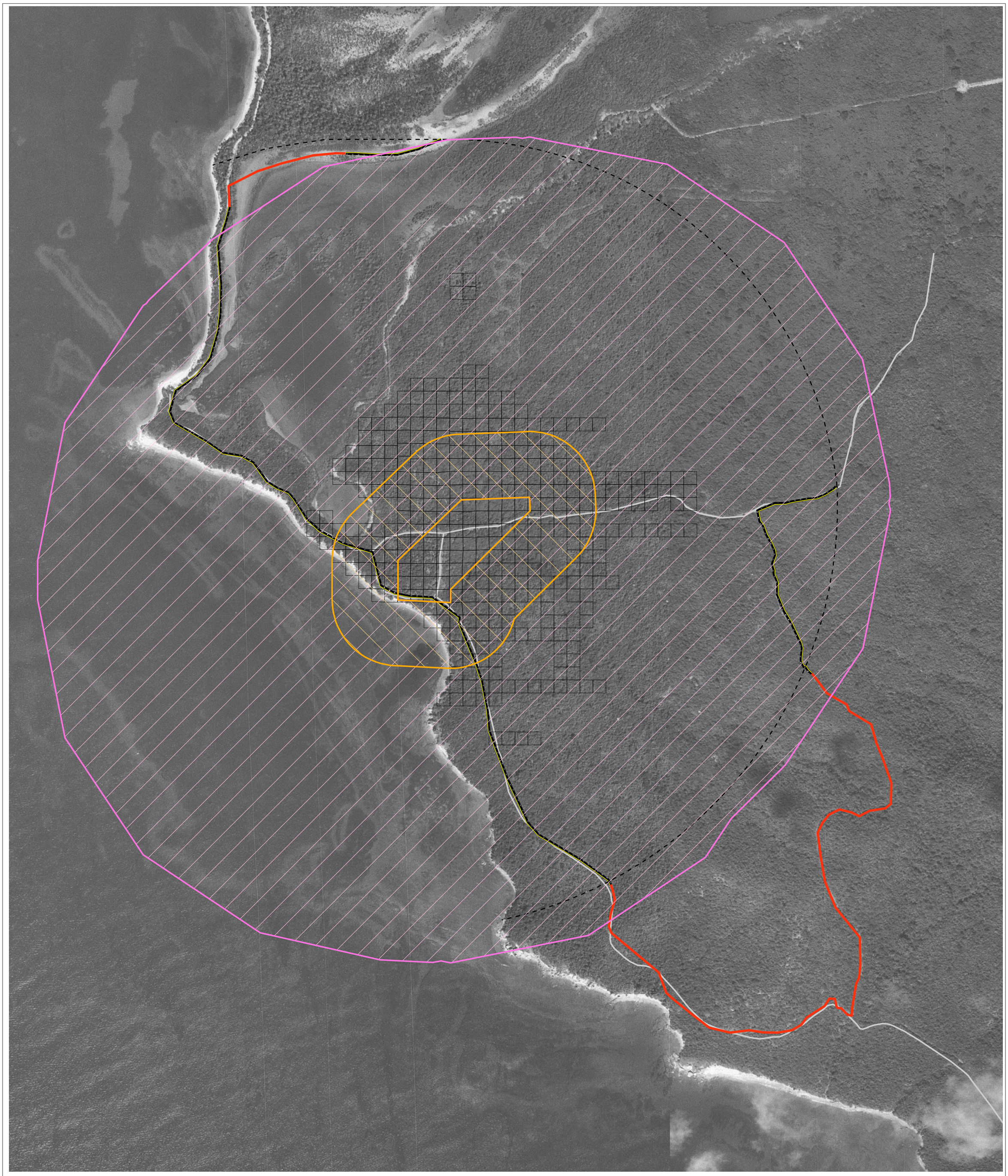
N

0 700 1400 Feet

Figure 2-6
DOI FWS Preliminary Land Use Plan
SWMU 4 MEC Remedial Investigation
Former NASD, West Vieques, Puerto Rico

CH2M HILL

File Path: (usbdrive) g:\18gis\vieques\figures\vieques_doi_fws.apr, Date: 28JUL04, User: munwin



Legend

- - - - OB/OD BOUNDARY
FENCE LINE INSIDE OB/OD AREA
FENCE LINE OUTSIDE OB/OD AREA
2772 ft EZ (Intentional Detonations)
506 ft EZ (Unintentional Detonations)

N
W E
S

Feet
0 500 1,000

Figure 2-7
SWMU 4 (OB/OD Pits) Exclusion Zones
Former NASD, West Vieques, Puerto Rico

CH2MHILL

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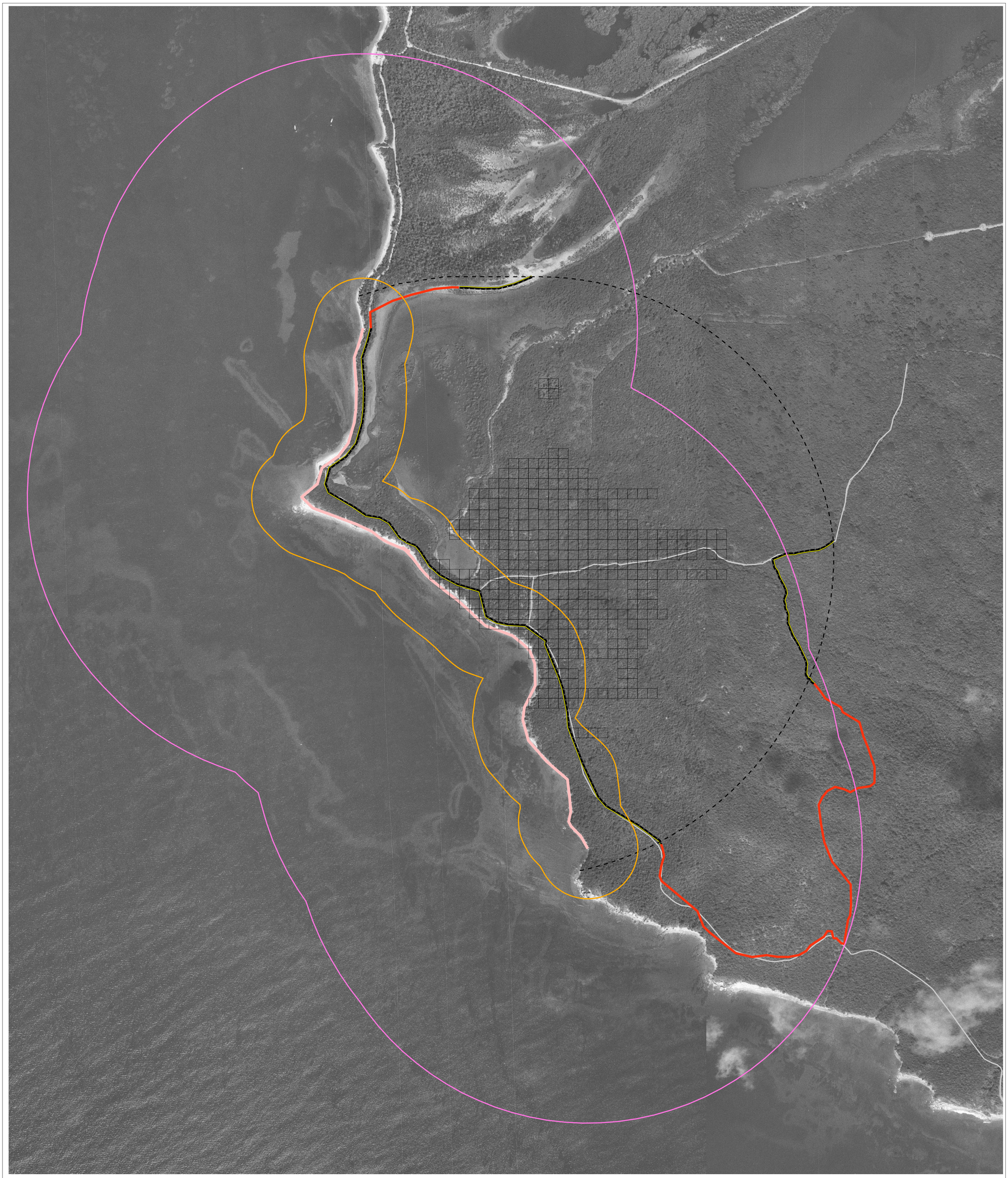


Legend

- OB/OD BOUNDARY
- FENCE LINE INSIDE OB/OD AREA
- FENCE LINE OUTSIDE OB/OD AREA
- BEACH AREAS
- 2772 ft EZ (Intentional Detonations)
- 506 ft EZ (Unintentional Detonations)

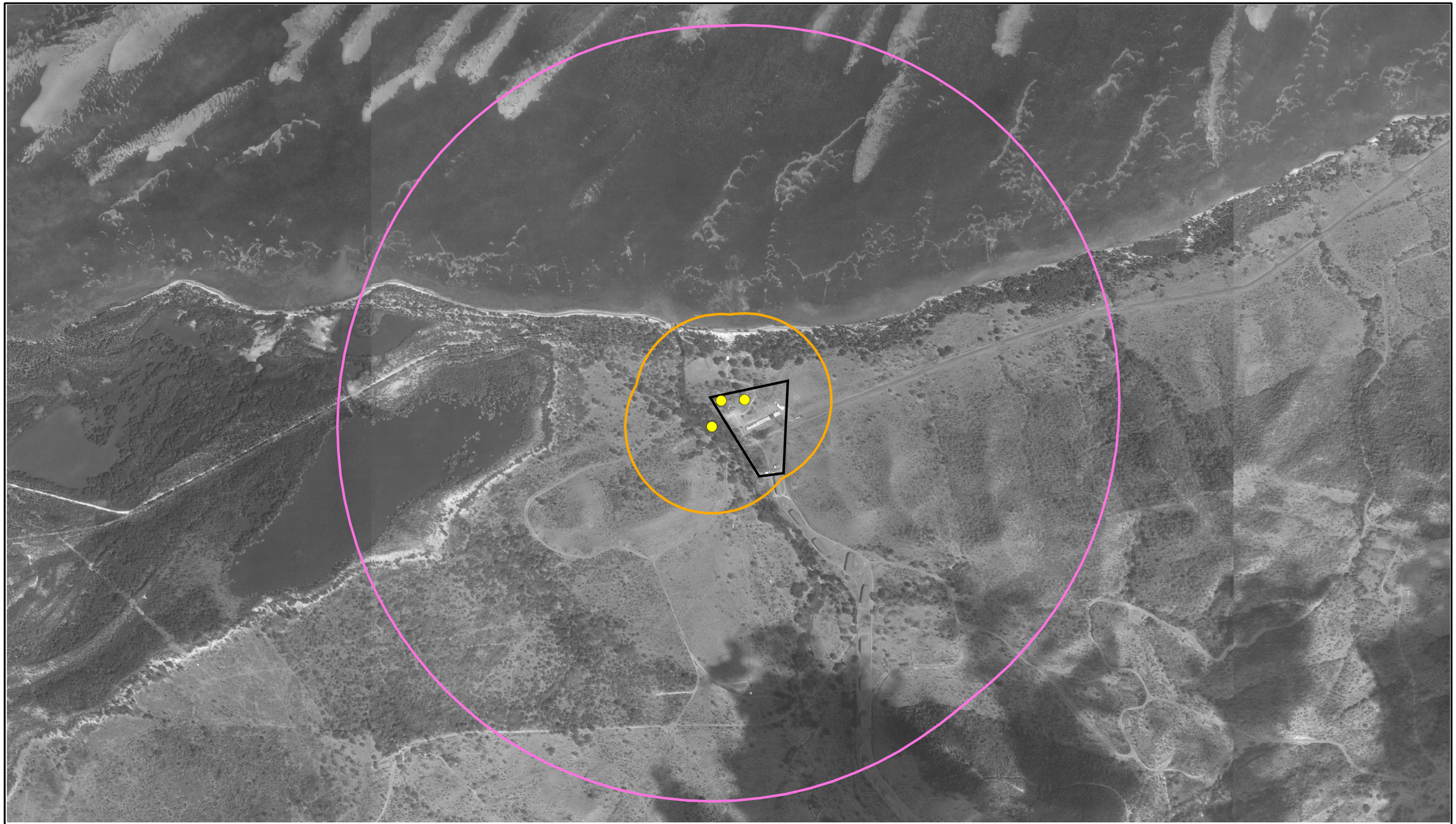
N
W E
S

Feet
0 500 1,000

Figure 2-8
SWMU 4 (Beach Areas) Exclusion Zones
Former NASD, West Vieques, Puerto Rico

CH2MHILL

File Path: \\ariadne\18gis\vieques2\figures\mxd\figure2-8_swmu4_beach_ez.mxd, Date: 8/2/05, User: munwin



Source: 1967 Aerial

Environmental AOC Boundary

4,000 ft EZ (Intentional Detonations)

925 ft EZ (Unintentional Detonations)

Approximate Location of MEC

Note: Buildings that appear in the aerial photo no longer exist.

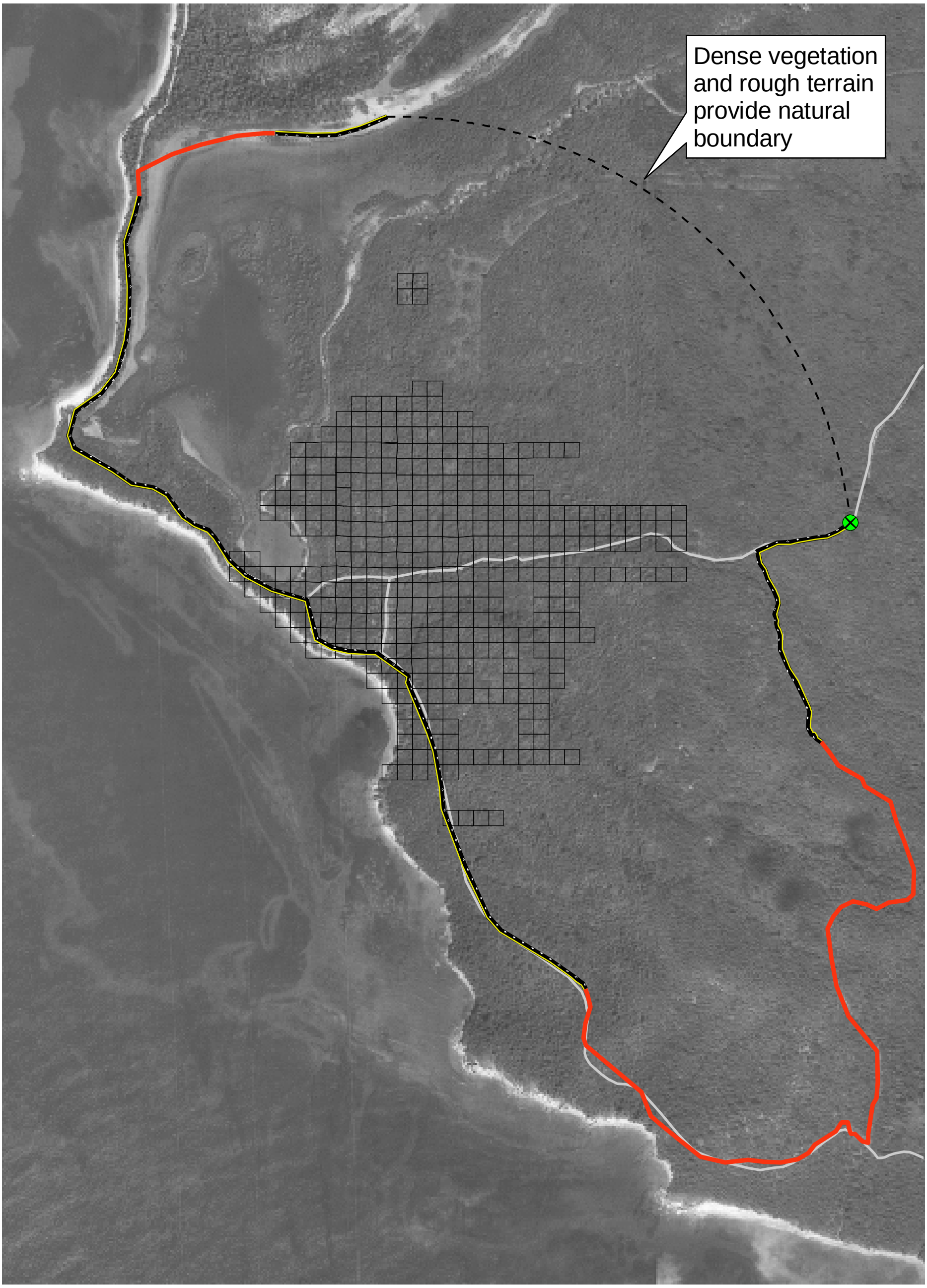
N W E S

0 1,000 2,000 Feet

Figure 2-9
AOC R Exclusion Zones
Former NASD, Vieques, Puerto Rico

CH2MHILL

File Path: \\ariadne\proj\18gis\vieques2\figures\mxd\figure2-9_aocR_excursionzones.mxd, Date: 8/2/05, User: munwin



Legend

- OB/OD BOUNDARY
- FENCE LINE INSIDE OB/OD AREA
- FENCE LINE OUTSIDE OB/OD AREA
- Gate

N
W E
S

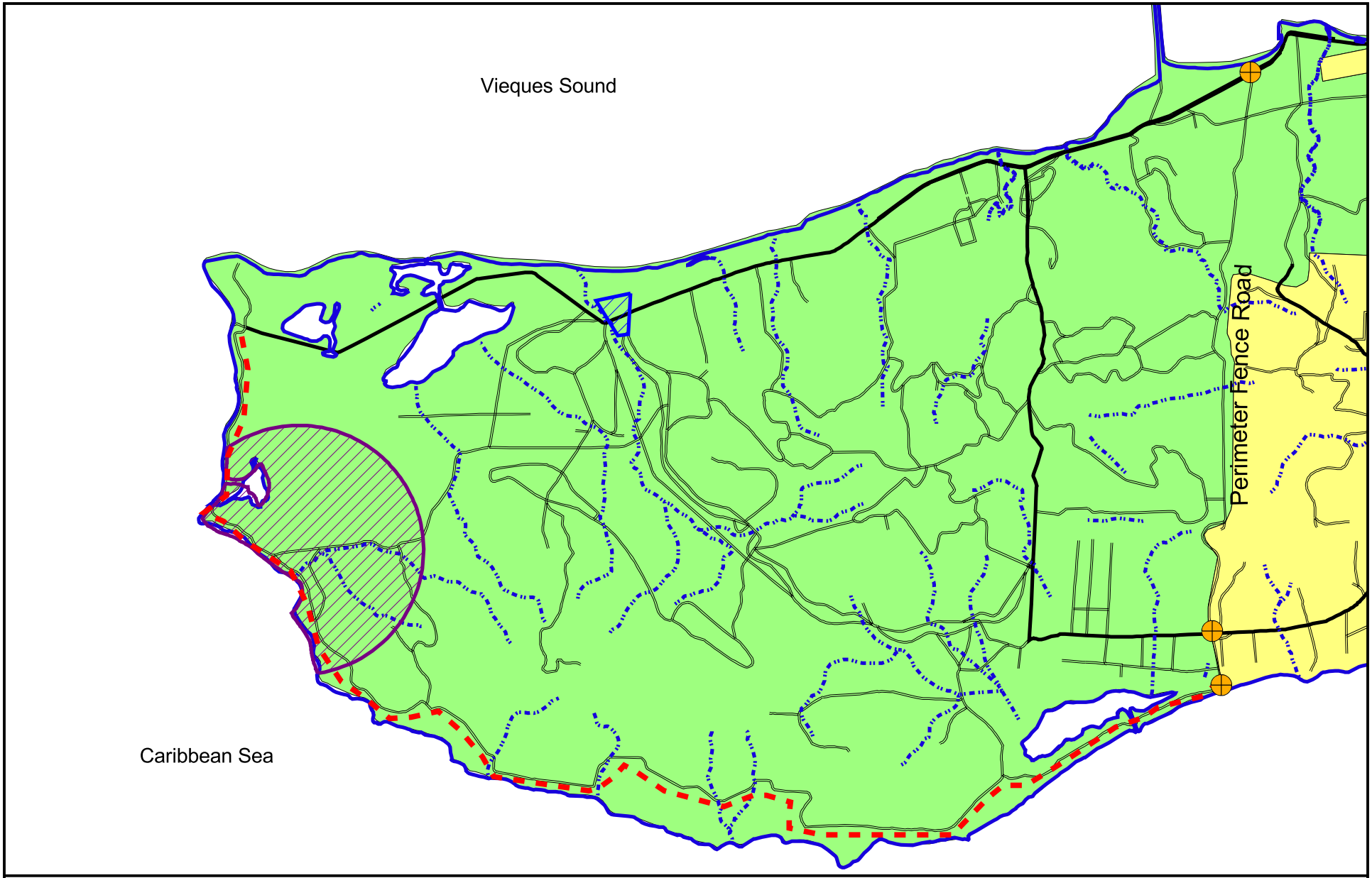
Feet
0 500 1,000

Figure 2-10
Security Fence
SWMU 4 MEC Remedial Investigation
Former NASD, West Vieques, Puerto Rico

CH2MHILL

File Path: \\Vbo20009180\USB drive\18gis\vieques\figures\mxd\figure2-10_securityfence.mxd, Date: 9/29/04, User: munwin

NOTE: Original figure created in color



Traffic Control Point
FOOT TRAIL
SWMU 4
AOC R
STREAMS
MAJOR ROADS
MINOR ROADS

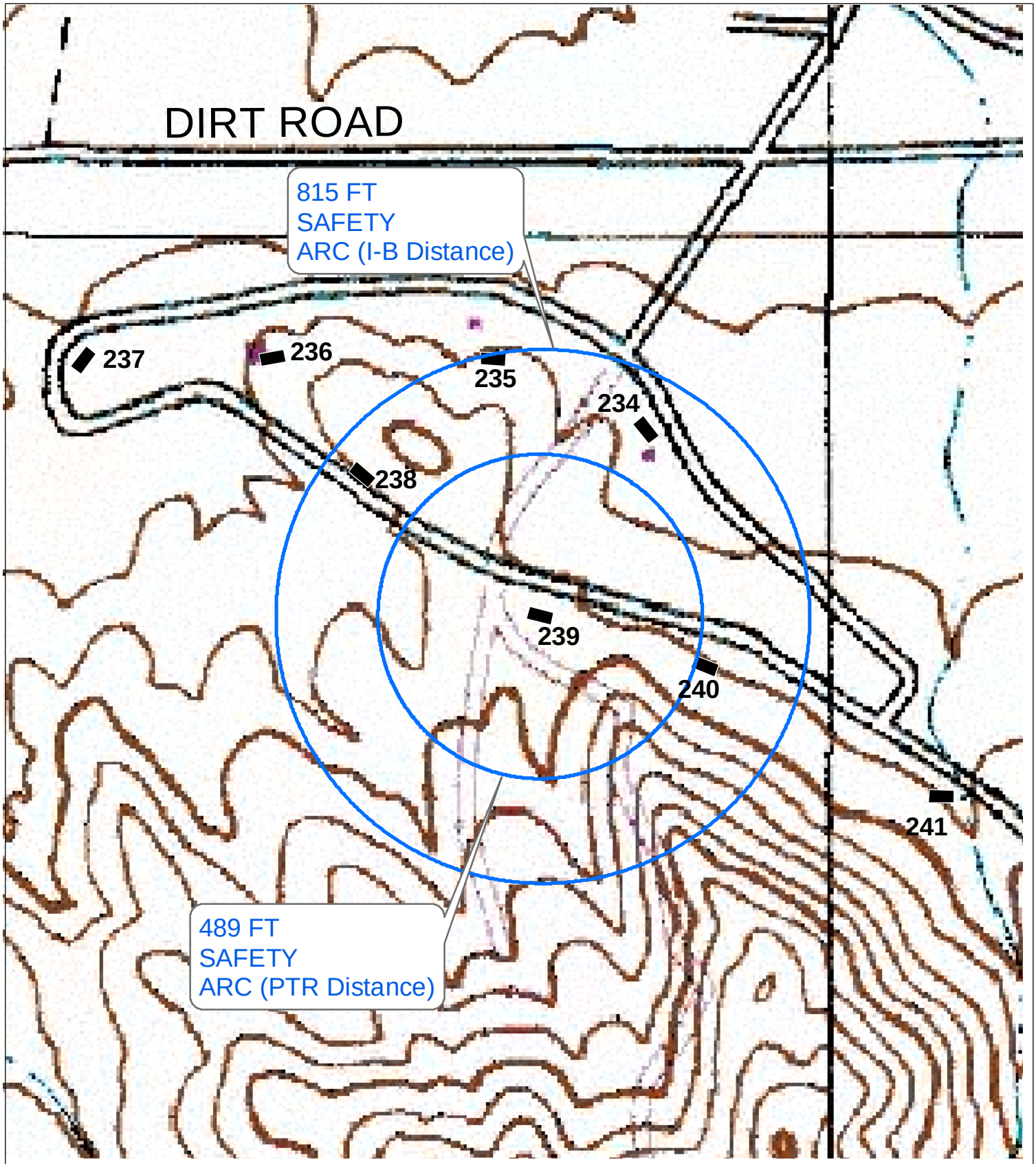
N

0 700 1400 Feet

Figure 2-11
Traffic Control Points for
AOC R Restricted Area
Former NASD, West Vieques, Puerto Rico

CH2M HILL

File Path: (usbdrive) g:\18gis\vieques\figures\vieques_doi_fws.apr, Date: 28JUL04, User: munwin



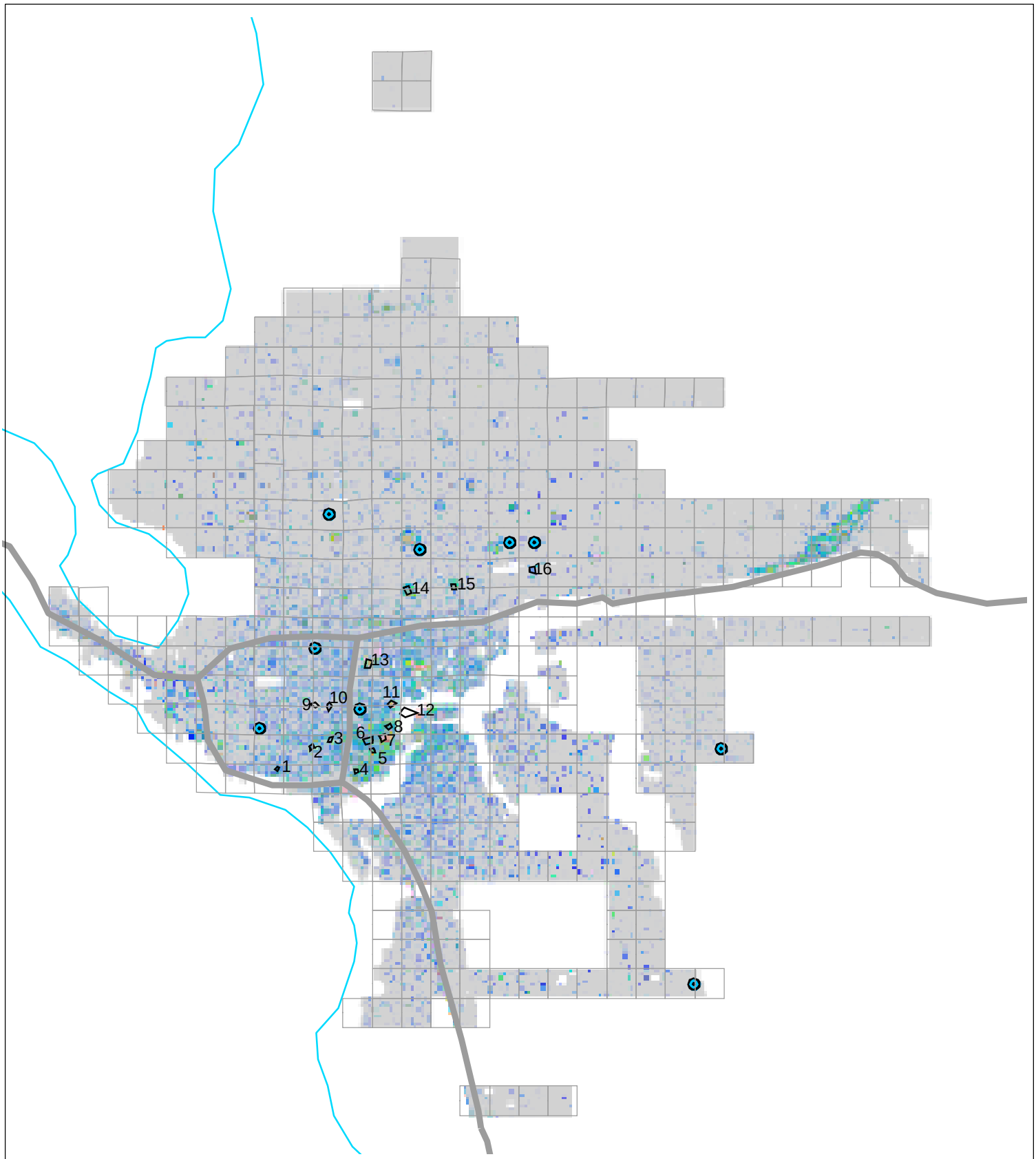
N

0 200 400 Feet

Figure 2-12
Building 239, Earth-Covered Magazine Safety Arcs
Former NASD, Vieques Island, Puerto Rico

CH2M HILL

File Path: \\Vbo20009180\USB drive\18gis\vieques\figures\mxd\figure2-12_safetyarcs.mxd, Date: 9/29/04, User: munwin



Legend

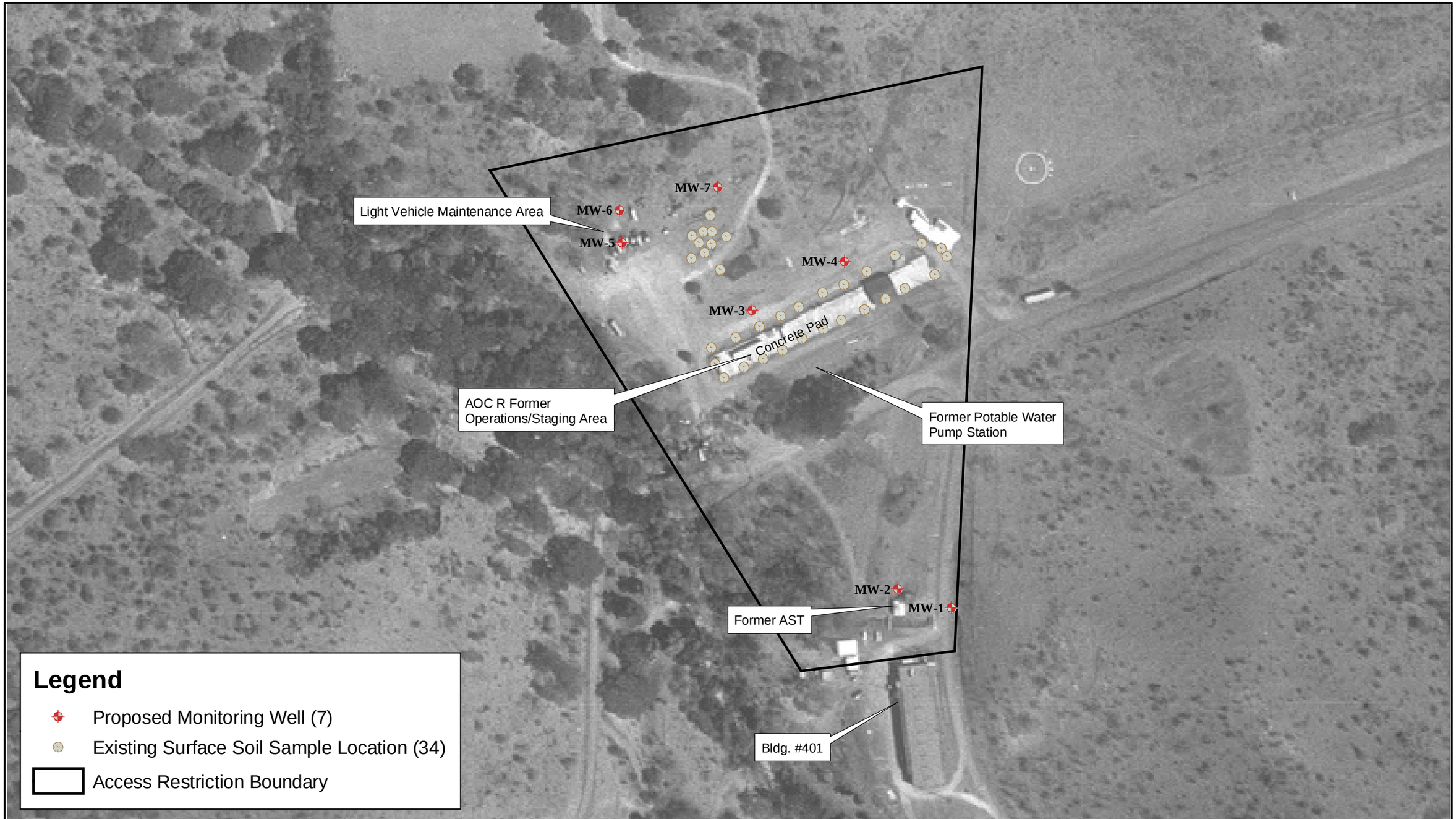
- Proposed Monitoring Wells
- OB/OD Pit
- Shoreline/Lagoon

200 0 200 Feet

Figure 2-13
Proposed Sampling Locations
in SWMU 4 Remedial Investigation
Former NASD, West Vieques, Puerto Rico

CH2M HILL

File Path: \\Vbo20009180\USB drive\18gis\vieques\figures\mxd\figure2-13_psamplocs.mxd, Date: 9/29/03, User: munwin



Source: 1967 Aerial

N
W E
S

0 150 300 Feet

Figure 2-14
Proposed Sampling Locations in AOC R Remedial Investigation
Former NASD, Vieques, Puerto Rico
CH2MHILL

File Path: \\vbo20009180\usb drive\18gis\vieques\figures\mxd\figure2-14_aocR_propsamplocs.mxd, Date: 9/29/04, User: munwin

SECTION 3

Amount/Type of MEC (MEC Overview and MGFD)

3.1 General

3.1.1 SWMU 4 MEC Overview

Records of the amount and type of MEC disposed of at the OB/OD pits are not available. Table 3-1 lists the type and amount of ordnance recovered during previous field studies of the SWMU 4 site.

To date, 90 percent of MEC items recovered at the SWMU 4 have been 20mm projectiles. Additionally, 98 percent of MEC items were found within 6 inches of the ground surface.

TABLE 3-1
MEC Items Encountered During Previous Studies of SWMU 4

Nomenclature	Quantity
High Explosive 20mm Projectile, M56	1,228
Cartridge Casing, 20mm	335
Assorted Blank Small Arms Cartridges (5.56mm & 7.62mm)	187
Armor Piercing 20mm Projectile	6
Incendiary 20mm Projectile	4
High Explosive 30mm Projectile	6
Photoflash Cartridge, M123A1	33
Photoflash Cartridge, M112	1
White Phosphorus Igniter, M23	1
White Phosphorus Canister	2
Illumination Hand Flare	3
Assorted Aircraft Cartridge Actuated Devices	5
Unknown Rocket Motor Catapult	1
Hydrostatic Fuze, MK 230	19
Mechanical Time Fuze, M25	3
Proximity Fuze (VT) M517	1
Bomb Fuze, Nose, Impact M904	2
Nose Fuze, Model Unknown	4
Fuze Booster, MK 300	1
Fuze Booster, Model Unknown	4
Rocket Warhead, MK II	6

TABLE 3-1
MEC Items Encountered During Previous Studies of SWMU 4

Nomenclature	Quantity
Rocket Motor, 5-inch	2
Rocket Motor, 2.75-inch	5
Projectile, 75mm, M48	3
Semi-armor Piercing Rocket Warhead, 5-inch	1
Rocket Warhead, 5-inch	1
High Explosive Projectile, 3-inch	2
Illumination Projectile, 3-inch	2
Signal Cartridge Projectile, 3-inch	1
Incendiary Bomb, AN-M52	1
Ballastite Propellant Increment (6.5 pounds)	2
Tail Boom & Primer for 81mm Mortar	15

3.1.2 AOC R MEC Overview

The MEC items listed in Table 3-2 were discovered during environmental sampling activities in August 2004. All external features of the MK-series LDGP bombs indicate the items are concrete-filled. However, during demilitarization procedures the items will be considered and treated as live, HE-filled bombs.

TABLE 3-2
MEC Items Encountered at AOC R in August 2004

Nomenclature	Quantity
MK 84, 2,000-pound, LDGP Bomb	2
MK 82, 500-pound, LDGP Bomb	2
MK 81, 250-pound LDGP Bomb	1
Inert Load-Training Version of MK 12 Bomb	1
LAU-3, 2.75-inch Rocket Launcher	4
2.75-inch Rocket Motor, 4-Tube Shipping and Storage Container	1
Ammunition Can	3

3.1.3 Munition with the Greatest Fragmentation Distance (MFGD)

Based on available data, the Munition with the Greatest Fragmentation Distance (MGFD) is the MK-63 5-inch Rocket Warhead at SWMU 4 and the MK 84 2000-pound LDGP bomb at AOC R. Table 3-3 presents the calculated fragment and overpressure distances for the MK-63 warhead and the MK 84 LDGP bomb.

During MR actions, if an item is encountered with a greater fragmentation distance, the Q-D arcs will be adjusted based on the item encountered and an amendment to the safety submission initiated.

3.1.4 Explosive Soils or Facilities

No explosive soils or explosive-contaminated buildings are expected to be encountered.

3.1.5 Other Description or Status of MRS Not Previously Covered

Much of the SMWU 4 and AOC R areas are densely vegetated. This could make all aspects of this project more difficult.

TABLE 3-3
Fragment and Calculated Overpressure Distances

MR Area	Munition[a]	Hazardous Fragment Distance[b] (ft)	Maximum Fragment Distance[c] (ft)	K50 Over Pressure Distance[d] (ft)
AOC R	MK 84 LDGP Bomb	925	3882	491
SWMU 4	MK 63 Rocket Warhead (5-inch)	506	2772	124

[a]For Q-D siting purposes, the MGFD and resulting EZ distances will be established for SWMU 4, based on the greatest of the fragmentation or overpressure distances for the types of MEC expected to be encountered. Fragment hazard distances will be recalculated based on the NEW, in the event different MEC are found.

[b]OP5, Volume 1, Tables 7-8 and 7.9, and DDESB TP-16, the distance at which fragments do not exceed a hazardous fragment density of one hazardous fragment per 600 ft^2, where a hazardous fragment is defined as a fragment having an impact energy of 58 ft-lb or greater.

[c]OP5, Volume 1, Tables 13.1 and 13.2, and DDESB TP-16, the maximum distance at which a fragment of the munition may travel based on munition design specifications and maximum trajectories.

[d]As calculated in accordance with OP5, Volume 1, Table 7.9 and DDESB TP-16, the distance at which blast overpressure exposure from Hazard Division 1.1 explosives is less than 0.90 psi—the lower end of the permissible exposure level for inhabited buildings and administrative and housing areas per Chapter 2, para. D.6 of DoD 6055.9-STD. Determined from the formula: $50 \times NEW^{1/3}$.

SECTION 4

Start Date

4.1 Schedule

Work on this project is projected to begin in October 2005. It is expected to take 6 to 8 weeks to complete.

SECTION 5

Frostline (Site Stability)

There is no frostline due to the tropical climate of the site. However, the island of Vieques is subject to potentially significant weather events such as tropical storms and hurricanes. These storms could cause accelerated surface erosion exposing or relocating near-surface MEC. Following these events, a visual inspection of cleared areas will be accomplished to determine if MEC has been exposed or relocated. Action will be taken based on the results of these inspections.

SECTION 6

Response Techniques

6.1 Introduction

The techniques used to detect, recover, and destroy MEC are summarized below. More detailed information may be found in the Site Specific Work Plans.

6.2 Surface Clearance of MEC

Prior to vegetation removal, environmental sampling, or digital geophysical mapping (DGM) the UXO subcontractor will carefully inspect the sites with the aid of an appropriate geophysical instrument (e.g., Schonstedt GA-52Cx magnetometer) for evidence of munitions on the ground surface.

The surface clearance will remove MEC (deemed safe to move); munitions and range-related debris; and other metallic objects that could interfere with vegetation removal, environmental sampling, or geophysical survey.

MEC items that are too hazardous to move or cannot be positively identified will be disposed of by the UXO subcontractor using blow-in-place (BIP) methods.

Since it is anticipated for the MR Action to be regulated under CERCLA, a RCRA permit will not be required. However, the treatment will satisfy the intent.

The UXO Technicians will provide a detailed description of the type and quantity of MEC encountered, including nomenclature, condition, and location found.

Any mechanized equipment to include support equipment such as lifting slings used to lift/ move MEC items that have not been positively identified as being inert must comply with the requirements of OP 5, Chapter 10 (Ordnance Handling Equipment).

6.3 Vegetation Removal

Vegetation will be removed from sites in SWMU 4 and AOC R to allow for site survey, environmental sampling, and DGM survey.

Vegetation removal methods may include vehicle-mounted mowers or hand-grubbing using chainsaws, axes, machetes, clippers etc.

The possibility of using “Controlled Burning” to remove vegetation is currently being evaluated with the Environmental Quality Board for the Commonwealth of Puerto Rico.

Prior to any mechanized vegetation removal, UXO personnel will conduct a magnetometer assisted visual surface sweep for MEC. If required, surface MEC will be disposed of or removed before vegetation removal begins. Once the UXO personnel have completed the visual surface sweep they will move ahead to areas awaiting visual surface sweeping to

insure a minimum separation distance of 500 ft. is maintained between the mechanized system and UXO personnel. If the operator sees anything in his path that warrants re-inspection the cutting will be momentarily halted and the UXO personnel will be brought back in to inspect the item of concern. MEC items identified during the initial surface sweep that cannot be safely removed will be conspicuously marked and the mechanized vegetation system operator thoroughly briefed before the commencement or continuation of brush clearing.

While a visual survey/sweep of the ground surface will always be conducted before any mechanized vegetation removal, the possibility exits for smaller munitions (75mm or less) to remain obscured at the ground surface due to the dense vegetation. Therefore, when utilizing vehicle mounted brush cutting equipment, the operator shall be adequately shielded to protect against an unintentional detonation of any items not discovered during the initial MEC surface sweep/survey.

It is anticipated that mechanized brush cutting will be accomplished using the TAZ II Caterpillar which has been armored with 0.75-in.-thick A572 steel plating and 2.65-in.-thick bullet resistant glass to protect the operator from the fragments and blast from an unintentional detention of a 75mm HE M48 projectile. Exhibits 6.3-1 through 6.3-4 provide detailed photographs of the TAZ II Caterpillar. Appendix A describes how this procedure and the associated engineering controls will address the concern of unintentional detonation during mechanized vegetation clearing.

EXHIBIT 6.3-1
TAZ II Caterpillar

EXHIBIT 6.3-2
TAZ II Caterpillar Armoring (Front View of Cab)

EXHIBIT 6.3-3
TAZ II Caterpillar Armoring (Side View of Cab)

EXHIBIT 6.3-4
TAZ II Caterpillar (Vegetation Cutting Head)

If vegetation is so dense that it prevents a thorough surface sweep, a systematic "Step-Down" method of brush clearance will be instituted to allow for a complete surface clearance/survey. All cutting will stop 6 inches above ground level. This will prevent disturbing subsurface MEC that may be entangled in plant roots if the roots are ripped from the soil.

Care will be taken to ensure cut trees and branches land in areas where MEC surface clearance has been performed. Cut trees and branches will not be dragged or carried through areas where surface MEC sweep has not been conducted.

After completion of vegetation clearance a comprehensive MEC identification and data collection will be completed.

6.4 Digital Geophysical Mapping (DGM)

To estimate the amount and extent of subsurface munitions at selected munitions response sites limited DGM may be conducted. This survey will use EM-61 electromagnetic geophysical equipment and Schonstedt GA-52Cx magnetometer (or industry equivalents). Selection of geophysical instrument will be based on field conditions such as soil types, accessibility, terrain, and vegetation density.

No Geophysical Prove-Out (GPO) is planned; however, geophysical test plots will be established to verify proper functioning of instruments in varied soil types.

The equipment will be carried over the sites by a two-person team along transects. Readings of subsurface metallic anomalies, indicative of buried munitions, will be collected continuously along each transect or recorded separately by GPS instruments.

Selected geophysical instruments are considered "passive" and do not require certification under the Navy's Hazards of Electromagnetic Radiation to Ordnance (HERO) program. RF transmitters, such as cellular phones and portable radios, will not be used in areas where EMR-susceptible MEC has been visually identified on the surface or in areas where disposal of susceptible MEC is documented.

Precise locations of the geophysical anomalies, using the latitude and longitude coordinate system, will be established using a global positioning system (GPS). Items identified by the survey team will be flagged for removal by follow-on UXO removal teams. The procedures for completing the geophysical survey are detailed in the Site Specific Work Plan.

These geophysical survey methods were selected based on best available technology for the scope of work and allocated funds. No limitations to survey methods or equipment are expected based on terrain or soil type. If problems are encountered in the field selected methods/equipment will be reevaluated.

6.5 Subsurface Removal of Munitions

No subsurface removal of MEC is planned for the MR Actions at SWMU 4 and AOC R. However, limited subsurface removal of MEC may become necessary to ensure field team safety. Subsurface anomalies that interfere with environmental sampling or site access by vehicles may require intrusive investigation.

During intrusive investigations in the SWMU-4 Site a 506ft exclusion zone (EZ) will be established around the excavation site. In the AOC-R site a 1,250ft EZ will be established. Only essential personnel will be allowed in the EZ during excavation. Heavy equipment may be used for initial digging; however, the final 12 inches of earth will be excavated using only hand-tools.

6.6 Treatment and Disposal of Munitions

Each MEC item discovered on the surface during MR Activities will be inspected by at least two qualified UXO personnel. MEC items which are too hazardous to move or cannot be positively identified will be disposed of using BIP procedures.

MEC items known or suspected to contain an explosive hazard will be treated/disposed of using standard demolition techniques. MEC items determined safe to move may be transported to a designated consolidation point within the MRS for disposal.

All MEC disposal operations will be performed under the direct supervision of an on-site Senior UXO Supervisor (SUXOS). An on-site UXO Site Safety Officer (UXOSO) will closely monitor operations, strictly enforce safety, ensure adherence to procedures, and verify the exclusion area is evacuated of non-essential personnel.

6.7 Quality Control & Quality Assurance Standards

This is an assessment and inspection of potential MR Sites. UXO Teams are expected to sweep and clear work sites of surface MEC and provide safety escort and MEC avoidance support to project personnel. Subsurface anomalies will be acquired, but not excavated unless they present a hazard to project personnel or interfere with environmental sampling. QC testing will be accomplished by the UXOQCS and site Geophysicist. No subsurface anomalies will be excavated. The following definable features of work and minimum standards will serve as the foundation of the QC process.

6.7.1 Site Survey and Mapping

All work sites must be accurately mapped. Grids and transects will be laid out with reference to control points/monuments.

6.7.2 Surface MEC Removal/Disposal

Surface MEC must be removed from site prior to initiating any other activity (e.g. vegetation removal and subsurface anomaly acquisition).

6.7.3 Vegetation Removal

All vegetation less than 4 inches in diameter must be cleared to within 6 inches of ground level before subsurface anomaly acquisition can be accomplished.

6.7.4 Subsurface Anomaly Acquisition/Data Collection

All MR sites must have complete coverage with handheld detectors or DGM instruments. Location of anomalies will be marked in the field (flagged) and recorded in a GPS database.

6.7.5 MPPEH Disposition

All munitions debris and range-related debris will be inspected, verified, and certified free of all explosive hazards before release to a qualified recycle facility.

CH2M HILL Munitions Response personnel will oversee subcontractor activities to ensure complete coverage of selected assessment/investigation sites.

6.8 Personnel Qualifications

All project personnel will meet the training and experience requirements outlined in the USACE Engineer Pamphlet 1110-1-18 and DDESB Technical Paper No. 18.

6.9 Certification and Processing of Material Potentially Presenting an Explosive Hazard (MPPEH)

This inspection, inert inspection, and certification of MPPEH shall be conducted in accordance with DoD 4160.21-M, Chapter 4, Paragraph B.3.

MPPEH recovered during the clearance process will be inspected by at least two qualified UXO Technicians. MPPEH will be segregated into items known or suspected to contain explosive hazard(s) and items known to contain no explosive hazard. Inert verification will be accomplished by a technically qualified DoD person.

MPPEH known or suspected to contain an explosive hazard will remain on the munitions response site awaiting further treatment/demilitarization. MPPEH known to be free of all explosive hazards will be demilitarized (if required) and transported to a secure area and sorted into Munitions Debris and Range-Related Debris.

MPPEH which require further treatment/demilitarization will undergo an additional inspection by at least two qualified UXO Technicians following treatment/demilitarization. This inspection will be verified by the SUXOS and UXOSO before the MPPEH is transported to the secure holding area.

Treatment of MPPEH may include exposing internal cavities/inert fillers with explosive charges to allow visual inspection. Demilitarization may include crushing, bending, or deforming.

Containers used to hold inert certified and verified material will be secured to prevent the addition of unauthorized material and will be labeled with the following signed statement:

"This certifies and verifies that the AEDA residue, Range Residue and/or Explosive Contaminated property listed has been 100 percent properly inspected and to the best of our knowledge and belief, are inert and/or free of explosives or other dangerous materials."

Prior to final disposition, the Project Manager and NAVFAC RPM will arrange for a US Navy employee to conduct a final certification of all MPPEH. This person must be designated in writing as trained and qualified to conduct this certification. This person will also sign all required documents required for disposition (e.g. DD Form 1348-1 with demilitarization/inert statement). This final certification should be conducted just prior to transfer to a qualified recycle facility.

During the entire process an accurate and unbroken chain of custody must be maintained for all MPPEH placed in the secure holding area. This will be accomplished through a well documented inspection/certification/verification process, key-issue logs (to the secure area) and up-to-date inventories of collected MPPEH.

6.10Soil and Groundwater Sampling

6.10.1 Surface Soil Sampling

Before surface soil sampling activities are initiated, the desired or suitable alternate locations will be cleared by a UXO Contractor utilizing a Schonstedt GA-52Cx or equivalent magnetometer.

Soil Sampling Techniques typically employed include stainless steel trowel sampling and stainless steel hand auger sampling, depending on the nature of the material to be sampled. A stainless steel trowel will be used to collect samples of loosely packed materials and a

stainless steel hand auger for densely packed materials. Surface soil samples will be obtained from a depth of 0 to 6 inches bgs.

6.10.2 Soil Borings

Before soil borings are initiated, the location will be inspected for MEC contamination by the UXO Contractor. Borings will be advanced using one of the following methods: a truck-mounted drill rig, an all-terrain vehicle (ATV) drill rig, using a split-spoon sampler or a hand auger.

A magnetometer with down-hole monitoring capabilities will be used for all borings greater than 4 feet in depth. The borehole will be started with a hand held portable auger. At not more than a 1-foot depth, the auger will be withdrawn and the magnetometer lowered into the borehole. This procedure will ensure smaller MEC items, undetectable from the surface, are identified. If no magnetic anomalies are found, the procedure will be repeated at 1-foot intervals to the maximum depth of the auger, but no less than 4 feet. If the proposed borehole site is free of magnetic anomalies, the drilling equipment may be brought on site and utilized. Borehole monitoring with the magnetometer shall continue at 1-foot intervals, until the sampling is completed.

If MEC contamination prevents access to the sampling site(s) by the drilling equipment, soil borings may be advanced using a hand auger or a tripod-mounted sampling device. The locations and total depth of hand-augured boreholes will be determined in the field based upon subsurface soil conditions and site-specific sampling objectives.

6.10.3 Monitoring Well Installation

Before monitoring wells are installed, the location will be inspected for MEC contamination by the UXO Contractor. The same procedures, utilizing a magnetometer with down-hole monitoring capabilities as for soil borings, will be employed by the UXO Contractor for monitoring well installation. Proposed monitoring well locations are shown in Figures 2-13 and 2-14. Borings will be advanced using one of the following methods: a truck-mounted drill rig, an all-terrain vehicle (ATV) drill rig, using a split-spoon sampler or a hand auger.

Monitoring well borings will usually be installed using hollow stem augers, unless field conditions require the use of a rotary or sonic method. The rotary method will utilize the addition of drilling water, not drilling mud. At locations where permanent wells are not required or well installation cannot be completed using a truck-mounted or ATV drilling rig, drive points may be installed. Drive points are 2-inch-diameter, stainless steel monitoring wells with short screens, and are installed by driving the well point into the ground. These will be installed in wet areas where the weight of a drilling rig cannot be supported, or because of steep slopes or dense forest. Drive points may also be installed in an open borehole.

SECTION 7

Quantity-Distance Overview

This section contains information on various activities and locations planned for the three sites where a minimum safe distance is required for MEC operations. These activities and locations are described below and are illustrated in the figures provided in Section 2, Description of ESS Related Site Maps.

7.1 MEC Areas

7.1.1 Restricted Area

For SWMU 4, the Restricted Area is defined as all land inside the fence and dense vegetation surrounding SWMU 4. Access to the Restricted Area is controlled by a single gate and warning signs posted at intervals on the fence.

At AOC R, the Restricted Area is defined as all land west of "Perimeter Fence Road." This is the north-south road that defines the eastern boundary of the Former NASD. Access to this Restricted Area will be controlled by three traffic-control points. One traffic-control point will be located at the intersection of Highway 200 and Perimeter Fence Road; the second traffic-control point will be at the intersection of Highway 201 and Perimeter Fence Road. The third traffic-control point will prevent pedestrians from accessing the foot-trail that runs along the southeast coast of the Vieques Wildlife Refuge.

Restricted Areas will only be established and enforced during activities directly related to Munitions Response. Although all facilities within the proposed restricted areas are uninhabited, the areas will be thoroughly checked before any Munitions Response activities commence. Once established, only project personnel and authorized, escorted visitors are allowed within the Restricted Area.

7.1.2 Exclusion Zones

An Exclusion Zone is a safety arc established around MR activities. EZs are established within the Restricted Area to further limit access to sites of MEC intrusive operations or planned detonations.

EZs are 3-dimensional. A Notice-to-Airmen will be coordinated through local FAA officials.

If EZ extends beyond the land boundaries of the Restricted Area, institutional and engineering controls will be established and enforced to ensure public safety. These controls include public safety announcements, roadblocks, evacuations, tamping of disposal shots, and relocating munitions prior to disposal.

If EZ extends into coastal waters, procedures will be implemented to prevent public exposure to the hazard. A Notice-to-Mariners will be coordinated through local U.S. Coast Guard officials. Project personnel will be posted at key vantage points on the beach to observe any encroachment into the exclusion zone. These sentries will be in radio/cell

phone communication with the work site supervisor. A small boat or personal watercraft will be available to relay a warning to boats entering the exclusion zone.

7.1.3 Unintentional Detonation EZ

Prior to the start of any MEC intrusive operations an EZ for unintentional detonation will be established. This EZ is based upon the hazardous fragment distance for the NEW of the MGFD.

EZ for Unintentional Detonation in SWMU 4

The unintentional detonation EZ during MEC intrusive operations is 506ft. Distance is based on the NEW for the MGFD (15lbs for the MK 63 rocket warhead) and derived from NAVSEA OP 5 Vol. 1, Table 7-9.

EZ for Unintentional Detonation in AOC R

The unintentional detonation EZ during MEC intrusive operations is 925ft. Distance is based on the NEW for the MGFD (945lbs for the MK 84 LDGP Bomb) and derived from NAVSEA OP 5 Vol. 1, Table 7-8.

7.1.4 Intentional Detonation EZ

Prior to intentional detonation of any MEC item(s) an EZ will be established. Intentional detonation EZ is based on the Maximum Case Fragment Throw Range. Rogue fragments may travel farther.

MEC items should be arranged in the safest possible configuration. Lugs and strong-backs should point downward. Base-plates and noses should point in the safest possible direction. Counter-charges should be placed to prevent shaped charge jets from forming during disposal. Counter-charges should be placed underneath MEC items containing white phosphorous (WP) to prevent unconsumed WP from becoming mixed with the soil.

The SUXOS and UXOSO will closely monitor the type and quantity of MEC items in consolidation shots to ensure maximum NEW is not exceeded.

Manned roadblocks will be established on all access roads leading to intentional detonation site. Personnel at these roadblocks will be in radio or cell phone communication with each other, the SUXOS, and the UXOSO.

EZ for Intentional Detonation in SWMU 4

The intentional detonation EZ in SWMU 4 is 2,772ft. This is based on the Maximum Case Fragment Throw Range of the MGFD (MK 63 5-inch rocket warhead). Intentional detonations in these MR Areas will not exceed 50lbs NEW (including demolition materials).

If MEC item(s) are significantly smaller than 5 inches in diameter, the EZ can be recalculated and reduced using NAVSEA OP 5 Vol. 1, Table 13-1 or 13-2.

EZ for Intentional Detonation in AOC R

The intentional detonation EZ in AOC R is 4,000ft. This is based on the Maximum Case Fragment Throw Range of the MGFD (Mk 84 LDGP Bomb = 3,880ft) and rounded up to

4,000ft. Intentional detonations in this MR Area will not exceed 1,000lbs NEW (including demolition materials).

7.1.5 Inhabited Building Distance (IBD)

IBD is the minimum permissible distance between and inhabited building and a potential explosion site (PES).

Based on the MGFD of a MK 63 Rocket Warhead, the IBD from the munitions response sites in SWMU 4 during this investigation is 506-feet. There are no inhabited buildings within these distances from SWMU 4.

For the MGFD of a MK 84 LDGP bomb at AOC R, the IBD is 1,250 feet. There are no inhabited buildings within the IBD of AOC R.

Procedures will be implemented to prevent public exposure to the hazards. A Notice-to-Mariners will be coordinated through local Coast Guard officials. Project personnel will be posted at key vantage points to observe any encroachment into the exclusion zone. These sentries will be in radio/cell phone communication with the work site supervisor. A small boat or personal watercraft may be employed to relay a warning to boats encroaching the area.

TABLE 7-1
Inhabited Building Distance (IBD) and Public Transportation Route Distance (PTRD)

Storage Facility/MR Area	MGFD	NEW (lbs)	Hazardous Fragment Distance (ft)	IBD (ft)	PTRD (ft)
ECM 239	N/A	150	N/A	815	489
SWMU 4	MK 63 Rkt WH	15	506	506	N/A
AOC R	MK 84 LDGP	945	925	1,250	N/A

7.1.6 Team Separation Distances

Team separation distances provide a safety zone between work sites.

Team Separation Distances in AOC R

Teams working within AOC R will maintain a minimum of 400ft separation distance. This is based on the K40 over pressure distance of 392 feet for the Mk 84 LDGP Bomb, the MGFD for this MR Area.

Team Separation Distance in SWMU 4

Teams working within SWMU 4 will maintain a minimum of 200ft separation distance. This is based on the K40 over pressure distance of 99 feet for the Mk 63 Rocket Warhead, the MGFD for this MR Area.

7.1.7 Preliminary Site Work

Preliminary site work such as elevation surveys, geophysical surveying and detecting anomalies does not require an Exclusion Zone for Q-D purposes.

7.2 Explosive Storage Magazines

A Type 2 portable explosives storage magazine, as described in Section 55.208 (a) (4) of ATF P 5400.7, ATF Explosives Law and Regulations has been sited at the entrance to Earth Covered Magazine (ECM) 239. Per NOSSA letter 8020 Ser N7112/30 of 11 Jan 02, this magazine was approved for the storage of demolition explosives for munitions response operations. The magazine has been sited for 150 pounds of Hazard Class/Division 1.1 Explosives. The IBD for the portable magazine is 815 feet and the PTRD is 489 feet.

Details on the storage of explosives for the SWMU 4 work site can be found in the *Final Magazine Siting Plan for SWMU 4, SWMU 6 and AOC J, Former NASD, Vieques Island Puerto Rice* (CH2M HILL, 26 November 2001).

7.3 Planned or Established Treatment Sites

7.3.1 Open Detonation in SWMU 4

Disposal by detonation will be conducted at the munitions response site where the MEC is found. MEC items from two or more sites may be consolidated if they are safe to move. No single disposal event will exceed 50 lbs. NEW (including demolition materials).

The exclusion zone for disposal operations will be 2,772-feet, based on MGFD of a MK 63 Rocket Warhead. The applicable Notice to Airmen and Notice to Mariners will be issued prior to all planned detonations.

The gate at the entrance to SWMU 4 will be manned during disposal operations. Personnel at the gate will be in radio or cell phone communication with the SUXOS and UXOSO.

7.3.2 Open Detonation in AOC R

Disposal by detonation will be conducted at the munitions response site where the MEC is found. MEC items from two or more sites may be consolidated if they are safe to move. No single disposal event will exceed 1000 lbs. NEW (including demolition materials).

The exclusion zone for disposal operations will be 4,000-feet, based on MGFD of a MK 84 LDGP Bomb. The applicable Notice to Airmen and Notice to Mariners will be issued prior to all planned detonations.

The traffic control points at the north and south ends of Perimeter Fence Road will be manned during disposal operations. Personnel at the traffic control points will be in radio or cell phone communication with the SUXOS and UXOSO.

7.4 Footprint Areas

There are three types of footprint areas: blow-in-place (BIP), MEC collection points within a search grid, and consolidated shots within a search grid. The following outlines the size of the exclusion zone that apply around these areas:

7.4.1 SWMU 4 Blow-in-Place

A 2,772-foot exclusion area (based on MGFD of MK 63 Rocket Warhead) will be established around all MEC which is too dangerous to move and requires BIP disposal. Prior to disposal, it may be necessary for UXO and Geophysical teams to continue working inside the exclusion zones in order to locate and identify other MEC requiring BIP disposal. This will allow for more efficient disposal operations.

7.4.2 AOC R Blow-in-Place

A 4,000-foot exclusion area (based on MGFD of MK 84 LDGP Bomb) will be established around all MEC which is too dangerous to move and requires BIP disposal. Prior to disposal, it may be necessary for UXO and Geophysical teams to continue working inside the exclusion zones in order to locate and identify other MEC requiring BIP disposal. This will allow for more efficient disposal operations.

7.4.3 SWMU 4 Collection Points

MEC recovered may be consolidated onsite if deemed safe to move. Collection point locations will be designated by the SUXOS and briefed during the morning safety meetings and updated as required. The NEW (including demolition materials), for collection points within SWMU 4 will not exceed 50 lbs. A 601-ft footprint area will be established around each collection point.

7.4.4 AOC R Collection Points

MEC recovered may be consolidated onsite if deemed safe to move. Collection point locations will be designated by the SUXOS and briefed during the morning safety meetings and updated as required. The NEW (including demolition materials), for collection points within AOC-R will not exceed 1000lbs. A 1,250-ft footprint area will be established around each collection point.

7.4.5 SWMU 4 In-Grid Consolidation Shots

MEC recovered onsite may be consolidated for in-grid consolidated disposal if deemed safe to move. These consolidated disposal shots will not exceed 50 lbs. NEW (including demolition materials). The SUXOS will determine where in-grid consolidation shots will take place and ensure the appropriate exclusion zone (2,772-feet) is established.

7.4.6 AOC R In-Grid Consolidation Shots

MEC recovered onsite may be consolidated for in-grid consolidated disposal if deemed safe to move. These consolidated disposal shots will not exceed 1000 lbs. NEW (including demolition materials). The SUXOS will determine where in-grid consolidation shots will take place and ensure the appropriate exclusion zone (4,000-feet) is established.

SECTION 8

Offsite Disposal

Recovered MEC will be destroyed onsite. No offsite disposal is anticipated.

SECTION 9

Environmental Considerations

Previous ecological studies of western Vieques have demonstrated that the past MEC response actions have not had a significant impact on the ecological receptors of the area. As a result, the MEC response actions are not anticipated to impact the surrounding ecological environment. However, if any natural or archeological resource may be impacted by these activities, the Environmental Resource Manager (DoI) will be contacted.

SECTION 10

Technical Support

Oversight for the MEC response actions will be provided, under the direction of the Naval Facilities Engineering Command, by a CH2MHILL UXO Safety Officer (UXOSO). Field investigations will be conducted by qualified MEC personnel including: Senior Unexploded Ordnance Supervisor, UXO Technician III and UXO Technician II field staff. MEC personnel will be drawn from CH2M HILL or qualified subcontractor(s). All UXO Technicians shall meet the training and experience requirements of DDESB Technical Paper 18.

Assistance for unknown MEC or suspected Chemical Warfare Materiel (CWM) will be requested through the US Army's 52d Ordnance Group at Fort Gillem, GA (404) 469-3333 or the US Navy's EOD Technical Support Center at (301) 744-4069 or 1-877-363-4636. Request(s) will be coordinated through the LANTDIV RPM and the Department of the Interior.

SECTION 11

Land Use Restrictions

Over the next few years, DOI will develop the Comprehensive Conservation Plan for the Vieques National Wildlife Refuge. This plan will outline the land use plan for managing the refuge. Until that process is complete, it is difficult to reach a final decision on cleanup. While all military activities have ceased at the Former NASD, the U.S. Navy retains responsibility for any MEC and/or environmental concerns that may exist there.

Any land use restrictions such as access control planned for the Former NASD are expected to be consistent with those established for state and federal wildlife refuges.

SECTION 12

Public Involvement

A Technical Review Committee (TRC) was formed with members of the community and regulators in 2001. Quarterly meetings are conducted at facilities on Vieques to discuss the progress of MEC response action and environmental restoration activities as well as community concerns.

Other public involvement activities will include:

- A Community Relations Plan, developed with EPA in 2001, will continue to be implemented during this project.
- The Navy prepared an MEC safety video to be presented at public meetings and within the public schools.
- MEC Safety Awareness fact sheets were prepared and distributed to the public.
- Public notices are published in the local newspaper when planned detonation activities occur.

SECTION 13

References

Magazine Siting Plan for Solid Waste Management Unit 4 (SWMU 4), SWMU 6, and Area of Concern J; Former Naval Ammunition Support Detachment; Vieques Island, Puerto Rico, 26 November 2001, Revision 1, 17 May 2002(Final); CH2M HILL.

Solid Waste Management Unit 4 Munitions and Explosives of Concern Remedial Investigation Report, Former Naval Ammunition Support Detachment, Vieques Island, Puerto Rico, March 2004 (Draft); CH2M HILL.

Remedial Investigation/Feasibility Study Work Plan for AOC I and AOC R at the Former U.S. Naval Ammunition Support Detachment (NASD) Vieques Island, Puerto Rico August 2004 (Final); CH2M HILL.

Munitions Response Site Identification and Notification Report, 9 August 2004; Electronically Transmitted from NAVFAC Atlantic to Naval Ordnance Safety & Security Activity.

Appendix A
Risk Evaluation – Mechanized Vegetation Removal Process

Risk Evaluation
Mechanized Vegetation Removal Process
Solid Waste Management Unit 4 (SWMU 4)
and Area of Concern "R" (AOC R)
Former Naval Ammunition Support Detachment (NASD)
Vieques, Puerto Rico

The following information is intended to both present the methods for minimizing the possibility of occurrence of an unintentional detonation of military munitions during mechanized vegetation removal as well as to present the probability and severity of the event should it occur. This risk evaluation will, in general, follow the guidance presented in OPNAVINST 3500.39A and MILSTD 882D and will be presented in two parts. The first section will present the explosive safety hazards associated with different site worker/explosive hazard interactions along with the suggested control(s) to minimize the hazard. The importance of this section is to convey to the reader all the procedures and engineering controls that will be used to maximize worker safety. The explosive exposure risk that remains following the employment of all the procedures and engineering controls will be evaluated in Section Two by 2 separate Risk Assessment Matrices. Both Risk Assessment Matrices consider the elements of hazard severity and mishap probability.

Section One

Hazard: Mechanized vegetation removal needs to occur in an area that is known to contain unexploded ordnance. The density of the vegetation is such that the operator of the vegetation removal machinery may not be able to see all explosive hazards in the path of the mechanized system.

Control: Qualified UXO Technicians will always proceed the mechanized vegetation removal system with a visual inspection of the vegetation and the ground surface. They will remain separated by the mechanized system at all times while cutting is being conducted by a minimum of 500 ft.

Hazard: The vegetation is so dense that the visual inspection team can not advance and can not effectively see the ground surface to assess the presences or absences of MEC.

Control: If vegetation is so dense that it prevents a thorough surface sweep, a systematic "Step-Down" method of vegetation removal will be instituted to allow for a complete surface clearance/survey. The mechanized system will begin by cutting the vegetation at the highest point (canopy). Since projected or dropped military munitions that fail to function are expected to come to rest at or below the ground surface the upper portions of the vegetation cover can be safely removed to allow the UXO Technicians an improved view of the path ahead. This type of phased removal will continue until the determination can be made that the path ahead is reasonable clear of all but the smaller military munitions such as a 20mm projectile at the ground surface.

Hazard: MEC is detected in the path of the mechanized removal equipment.

Control: MEC items identified during the initial or subsequent surface sweep that cannot be safely moved, will be conspicuously marked and the equipment operator thoroughly briefed before the commencement or continuation of brush clearing to insure the item is avoided.

Hazard: The cut vegetation, "slash", prohibits the UXO Technicians from being able to see the ground surface following the step-down method of removal.

Control: All cutting will stop 6 inches above the ground surface. This will prevent the cutting mechanism from coming in contact with MEC that is small enough to be obscured by the slash and will also prevent disturbing subsurface MEC that might be close to or contain in plant roots that would be ripped from the soil if the cutting were to advance to the ground surface.

Hazard: Stopping the cutting blades 6 inches above the ground may prevent unintentional contact with most small munitions but MEC as large as a 75mm projectile could be difficult to visually detect due to the cut vegetative matter yet may stick up more than 6 inches on uneven ground. Such a condition and/or contact between MEC and the heavy tracks of the machinery could result in an unintentional detonation.

Control: The TAZ II Caterpillar that will be used for mechanized vegetation removal has been armored to survive an unintentional detonation of a 75mm HE projectile.

Section Two

This section is intended to evaluate the risk that remains after operational procedures and engineering controls have been put in place to reduce the risk from exposure to an explosive hazard. The risk will be presented in the form of a Risk Assessment Code (RAC) in accordance with OPNAVINST 3500.39A and a Mishap Risk Assessment in accordance with MILSTD 882.D. Both of the assessments consider the elements of probability and severity of occurrence and both will be used to evaluate the risk from explosive exposure to the unintentional detonation of a military munition greater in size than a 75mm projectile.

The RAC will be completed in accordance with the guidance found in OPNAVINST 3500.39A. In that guidance Hazard Severity is defined as an assessment of the worst credible consequence which can occur as a result of a hazard. Hazard Severity categories are assigned as Roman numerals according to the following criteria:
(1) Category I - The hazard may cause death, loss of facility/asset or result in grave damage to national interests.
(2) Category II - The hazard may cause severe injury, illness, property damage, damage to national or service interests or degradation to efficient use of assets.
(3) Category III - The hazard may cause minor injury, illness, property damage, damage to national, service or command interests or degradation to efficient use of assets.
(4) Category IV - The hazard presents a minimal threat to personnel safety or health, property, national, service or command interests or efficient use of assets.

OPNAVINST 3500.39A defines Mishap Probability as the probability that a hazard will

result in a mishap or loss, based on an assessment of such factors as location exposure (cycles or hours of operation), affected populations, experience or previously established statistical information. Mishap probability will be assigned an English letter according to the following criteria:
(1) Sub-category A - Likely to occur immediately or within a short period of time. Expected to occur frequently to an individual item or person or continuously to a fleet, inventory or group.
(2) Sub-category B - Probably will occur in time. Expected to occur several times to an individual item or person or frequently to a fleet, inventory or group.
(3) Sub-category C - May occur in time. Can reasonably be expected to occur some time to an individual item or person or several times to a fleet, inventory or group.
(4) Sub-category D - Unlikely to occur.

OPNAVINST 3500.39A defines the Risk Assessment Code (RAC) as an expression of risk which combines the elements of hazard severity and mishap probability. Using the matrix shown below, the RAC is expressed as a single Arabic number that can be used to help determine hazard abatement priorities. The RAC Definitions are as follows: 1 – Critical, 2 – Serious, 3 – Moderate, 4 –Minor, and 5 – Negligible

		Mishap Probability			
		A	B	C	D
Hazard Severity	I	1	1	2	3
	II	1	2	3	4
	III	2	3	4	5
	IV	3	4	5	5

The Hazard Severity category considered appropriate for the mechanized vegetation removal activity at Former Naval Ammunition Support Detachment is Category I, which means the hazard of an unintentional detonation of a MEC item larger than a 75mm projectile may cause death. The Mishap Probability Sub-category considered appropriate for the mechanized vegetation removal activity, considering all the operational procedures and engineering controls, is Sub-category D, meaning that the unintentional detonation of an MEC item larger than a 75mm projectile is considered unlikely to occur. Considering these selections the Operational Risk Management process will result in a RAC of 3, meaning that the risk is determined to be moderate.

A result of moderate risk from the Operational Risk Management process is not considered unusual nor excessive for the nature of Munitions Response work. Virtually all intrusive activities rely on adherence to procedure and method and the utilization of engineering control to insure that, regardless of the Hazard Severity, the event being assessed is unlikely to occur.

The Mishap Risk Assessment, as defined by MILSTD 882D, basically employs the same elements as does the RAC to assess the risk of occurrence. However, there are two differences between the RAC and the Mishap Risk Assessment. One being that the Mishap Risk Assessment identifies 5 levels of Mishap Probability where the RAC uses only 4 and

secondly the Mishap Risk Assessment identifies the recommended level of management from which the using organization/program team should seek Mishap Risk Acceptance.

In the guidance document MILSTD 882D Mishap severity categories are defined to provide a qualitative measure of the most reasonable credible mishap resulting from personnel error, environmental conditions, design inadequacies, procedural deficiencies, or system, subsystem, or component failure or malfunction. The suggested mishap severity categories presented in MILSTD 882D are as follows:

Category I - Catastrophic - Could result in death, permanent total disability, loss exceeding $1M, or irreversible severe environmental damage that violates law or regulation.
Category II - Critical - Could result in permanent partial disability, injuries or occupational illness that may result in hospitalization of at least three personnel, loss exceeding $200K but less than $1M, or reversible environmental damage causing a violation of law or regulation.
Category III - Marginal - Could result in injury or occupational illness resulting in one or more lost work days(s), loss exceeding $10K but less than $200K, or mitigatible environmental damage without violation of law or regulation where restoration activities can be accomplished.
Category IV - Negligible - Could result in injury or illness not resulting in a lost work day, loss exceeding $2K but less than $10K, or minimal environmental damage not violating law or regulation.

In the guidance document MILSTD 882D Mishap probability is defined as the probability that a mishap will occur during the planned life expectancy of the system. It can be described in terms of potential occurrences per unit of time, events, population, items, or activity. Assigning a quantitative mishap probability to a potential design or procedural hazard is generally not possible early in the design process. At that stage, a qualitative mishap probability may be derived from research, analysis, and evaluation of historical safety data from similar systems. Supporting rationale for assigning a mishap probability is documented in hazard analysis reports. Mishap probability levels suggested in MILSTD 882D are as follows:

Level A - Frequent - Likely to occur often in the life of an item, with a probability of occurrence greater than 10^{-1} in that life.
Level B – Probable - Will occur several times in the life of an item, with a probability of occurrence less than 10^{-1} but greater than 10^{-2} in that life.
Level C – Occasional - Likely to occur some time in the life of an item, with probability of occurrence less than 10^{-2} but greater than 10^{-3} in that life.
Level D – Remote -Unlikely but possible to occur in the life of an item, with a probability of occurrence less than 10^{-3} but greater than 10^{-6} in that life.
Level E - Improbable - So unlikely, it can be assumed occurrence may not be experienced, with a probability of occurrence less than 10^{-6} in that life.

The Mishap Risk Assessment matrix will include the elements of mishap severity and mishap probability. As presented in MILSTD 882 D this matrix allows one to assign a mishap risk assessment value to a hazard based on these elements. This value can then be

used to rank different hazards as to their associated mishap risks and ultimately to a risk acceptance level. An example of a mishap risk assessment matrix is shown below.

SEVERITY PROBABILITY	Catastrophic	Critical	Marginal	Negligible
Frequent	1	3	7	13
Probable	2	5	9	16
Occasional	4	6	11	18
Remote	8	10	14	19
Improbable	12	15	17	20

The Mishap risk assessment values from the above table can be used in grouping individual hazards into mishap risk categories. The categories can then be used to recommend specific actions, in the Mishap Risk Acceptance Level column, such as mandatory reporting of certain hazards to management for formal acceptance of the risk. The Mishap Risk Acceptance Levels, in the table below, have be altered from the example provided in the guidance document to include management position specific to this project. The following table includes a listing of mishap risk categories and the associated assessment values and acceptance levels.

Mishap Risk Assessment Value	Mishap Risk Category
1 - 5	High
6 - 9	Serious
10 - 17	Medium
18 - 20	Low

Similar to the hazard severity category of the RAC evaluation the Mishap Risk Assessment Mishap Severity category considered appropriate for the mechanized vegetation removal activity at Former Naval Ammunition Support Detachment is Category I - Catastrophic, which means the hazard of an unintentional detonation of a MEC item larger than a 75mm projectile may cause death. The Mishap Probability level considered appropriate for the mechanized vegetation removal activity, considering all the operational procedures and engineering controls, is Level D, meaning that the unintentional detonation of an MEC item larger than a 75mm projectile is considered remote or unlikely but possible. Considering these selections the Mishap Risk Assessment value in accordance with MILSTD 882 D is eight (8). This Mishap Risk Assessment value results is a slightly more significant Risk Category than did the RAC evaluation. The Mishap Risk Assessment value of 8 equates to a risk category of Serious.

Draft

Explosives Safety Submission Ammendment

Solid Waste Management Units 6 and 7 (SWMUs 6 and 7) and Area of Concern "J" (AOC J)

Former Naval Ammunition Support Detachment (NASD) Vieques, Puerto Rico

CTO Task Order 002
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2-1 Regional Location Map

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2-3 Munitions Response Area SWMU 6

2-4 Munitions Response Area SWMU 7

2-5 Munitions Respone Area AOC J

2-6 DOI FWS Preliminary Land Use Plan

2-7 SWMU 6 Exclusion Zones

2-8 SWMU 7 Exclusion Zones

2-9 AOC J Exclusion Zones

2-10 Traffic Control Points for SWMU 6, SWMU 7, and AOC J Restricted Areas

2-11 Building 239, Earth-Covered Magazine Safety Arcs

2-12 Proposed Sampling Locations in SWMU 6 Remedial Investigation

2-13 Proposed Sampling Locations in SWMU 7 Remedial Investigation

2-14 Proposed Sampling Locations in AOC J Remedial Investigation

Acronyms and Abbreviations

AOC	Area of Concern
ATV	all-terrain vehicle
bgs	below ground surface
BIP	blow-in-place
CCP	Comprehensive Conservation Plan
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLEAN	Comprehensive Long-Term Environmental Action Navy
CRP	Community Relations Plan
CWM	Chemical Warfare Material
DDESB	Department of Defense Explosives Safety Board
DGM	Digital Geophysical Mapping
DoD	Department of Defense
DOI	Department of Interior
ECM	Earth Covered Magazine
EIS	Environmental Impact Statement
EOD	Explosive Ordnance Disposal
EQB	Environmental Quality Board
ERM	Environmental Resource Manager
ESS	Explosive Safety Submission
EZ	Exclusion Zone
ft	feet
FWS	U.S. Fish and Wildlife Service
GIS	Geographic Information System
GPO	Geophysical Prove-out
GPS	Global Positioning System
HERO	Hazards of Electromagnetic Radiation to Ordnance
IBD	Inhabited Building Distance
LANTDIV	Atlantic Division
MEC	Munitions and Explosives of Concern
MFGD	Munition with the Greatest Fragmentation Distance
MPPEH	Munitions that Present a Potential Explosive Hazard
MR	Munitions Response
NASD	Naval Ammunition Support Detachment
NAVFAC	Naval Facilities Engineering Command
NEW	Net Explosive Weight

OP	Observation Post
ORS	Ordnance Related Scrap
PA	Preliminary Assessment
PTRD	Public Transportation Route Distance
QC	Quality Control
RCRA	Resource Conservation and Recovery Act
SUXOS	Senior Unexploded Ordnance Supervisor
SWMU	Solid Waste Management Unit
TRC	Technical Review Committee
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
UXO/OE	Unexploded Ordnance/Ordnance and Explosives
UXOQCS	Unexploded Ordnance Quality Control Supervisor
UXOSO	Unexploded Ordnance Safety Officer
VNWR	Vieques National Wildlife Refuge
WP	white phosphorous

SECTION 1

Project Summary

1.1 Introduction

This Explosives Safety Submission (ESS) supplements work plans associated with the collection of environmental samples in the vicinity of Solid Waste Management Units (SWMUs) 6 and 7 and Area of Concern (AOC) J in the Former Naval Ammunition Support Detachment (NASD). Approximately 14 Munitions and Explosives of Concern (MEC) items were discovered near SWMU 6, approximately 12 MEC items near SWMU 7, and eleven items near AOC J. No live MEC items have been discovered at the three sites. The disposal/demilitarization of these items, and any similar items found during the site inspection is also covered under this ESS.

The ESS was prepared by CH2M HILL for the Naval Facilities Engineering Command (NAVFAC) Atlantic Division (LANTDIV) under Navy Contract N62470-02-D-3052, Navy Comprehensive Long-Term Environmental Action Navy (CLEAN), District III, Contract Task Order 002.

This ESS has been prepared in accordance with the requirements presented in Department of Defense (DoD) 6055.9-std; DoD ammunition and explosives safety standards, Chapter 12; NAVSEA OP 5 Volume 1, the guidance presented in Department of Defense Explosive Safety Board (DDESB) Memorandum of 27 February 1998, and the NOSSA Guidelines for Preparing an Explosives Safety Submission, (NOSSA Instruction 8020.15, 8 March 2004).

1.2 Conclusions from Previous Studies and Reports

SWMU 6 covers an area of approximately 1 acre on the Former NASD. The site was used during the 1960s and 1970s as a disposal area for solid and generic waste from past Navy operations within the NASD. U.S. Navy personnel disposed of items including cans of lubricants and oil, solvents, paints, and rubble.

An Expanded Preliminary Assessment/Site Investigation (PA/SI) was conducted by CH2M HILL in 2000. No live MEC items were encountered during that investigation, however, inactive MEC have been identified. A detailed inventory of MEC items recovered during the investigation is included in Table 3-1 of this document.

SWMU 7 covers an area of approximately 1 acre on the Former NASD. From the early 1960's to the late 1970's, a steep ditch at the site was used for disposal of solid waste materials. Discarded materials disposed on SWMU 7 include old tires, sheet metal, empty containers such as drums, cans, and bottles, old batteries, and construction rubble.

During the Expanded PA/SI conducted by CH2M HILL in 2000, the MEC avoidance survey noted the presence of ordinance related scrap (ORS) items in the ditch but no unexploded ordinance/ordinance and explosives (UXO/OE) items were found. A detailed inventory of MEC items recovered is included in Table 3-2 of this document.

AOC J encompasses an area of approximately 1.2 acres within the Former NASD and was used from 1965 to 1973 as a solid waste dumpsite for construction staging activities. After 1973, some of the unidentified solid waste disposed of at the site was removed from the site and placed in a municipal landfill off base. No records were kept indicating the location of the landfill. The types of waste taken from the site are unknown.

During the Expanded PA/SI conducted in 2000, a MEC survey team identified several scrap metal pieces associated with ordnance and explosive items; however, no live munitions items were found. A detailed inventory of MEC items recovered is included in Table 3-3 of this document.

1.3 Suspected Type and Amount of Ordnance Contamination

MEC items discovered at SWMU 6 appear to be concrete-filled practice bombs. Munitions related accessories, such as empty bomb dispensers, and empty shell casings, have also been found within SWMU 6. A more detailed description of the types of ordnance that may be encountered is provided in Section 3 of this ESS.

MEC items discovered at SWMU 7 appear to be Mk 56 Drill Mines. Munitions related scrap, such as rocket launchers, and ammunition cans, have also been found within SWMU 7. A more detailed description of the types of ordnance that may be encountered is provided in Section 3 of this ESS.

MEC related scrap items discovered at AOC J include cartridge canisters and bases, and flash tubes. A more detailed description of the types of ordnance that may be encountered is provided in Section 3 of this ESS.

1.4 Planned Future Use of Property

The US Fish and Wildlife Service (FWS) is in the process of preparing the Comprehensive Conservation Plan (CCP)/Environmental Impact Statement (EIS) for the Vieques National Wildlife Refuge (VNWR). This document will provide long-term guidance for land management and use.

The Navy will maintain control of SWMU 7 and AOC J until all requirements of environmental characterization and cleanup are satisfied. However, SWMU 7 and AOC J will ultimately be returned to the Municipality of Vieques for development.

1.5 Remediation Goals

Remediation goals will be established following publication of the final version of the FWS land use planand beased on the land-use plans of the Municipality of Vieques. For this investigation environmental samples will be taken within the MR Sites to confirm the success of debris removal.

1.6 Type of Munitions Response Action

The Puerto Rico Environmental Quality Board (EQB) provides oversight for work on the Former NASD. Although the site has not been officially placed under the domain of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) or Resource Conservation and Recovery Act (RCRA), the EQB has generally followed CERCLA guidelines for projects conducted near the Former NASD Area (west Vieques).

The munitions response action at SWMUs 6 and 7 and AOC J is primarily MEC avoidance to facilitate the safe completion of debris removal and collection of environmental samples. Future actions may include surface MEC removal, digital geophysical mapping (DGM), and subsurface MEC removal.

SECTION 2

Maps

2.1 Regional Map

Figure 2-1: Regional Location Map

2.2 Activity Maps

Figure 2-2: Munition Response Areas

Figure 2-3: Munitions Response Area SWMU 6

Figure 2-4: Munitions Response Area SWMU 7

Figure 2-5: Munitions Response Area AOC J

Figure 2-6: DOI FWS Preliminary Land Use Plan

Figure 2-7: SWMU 6 Exclusion Zones

Figure 2-8: SWMU 7 Exclusion Zones

Figure 2-9: AOC J Exclusion Zones

Figure 2-10: Traffic Control Points for SWMU 6, SWMU 7 and AOC J Restricted Areas

2.3 Q-D Map

Figure 2-11: Building 239, Earth Covered Magazine Explosive Safety Arcs

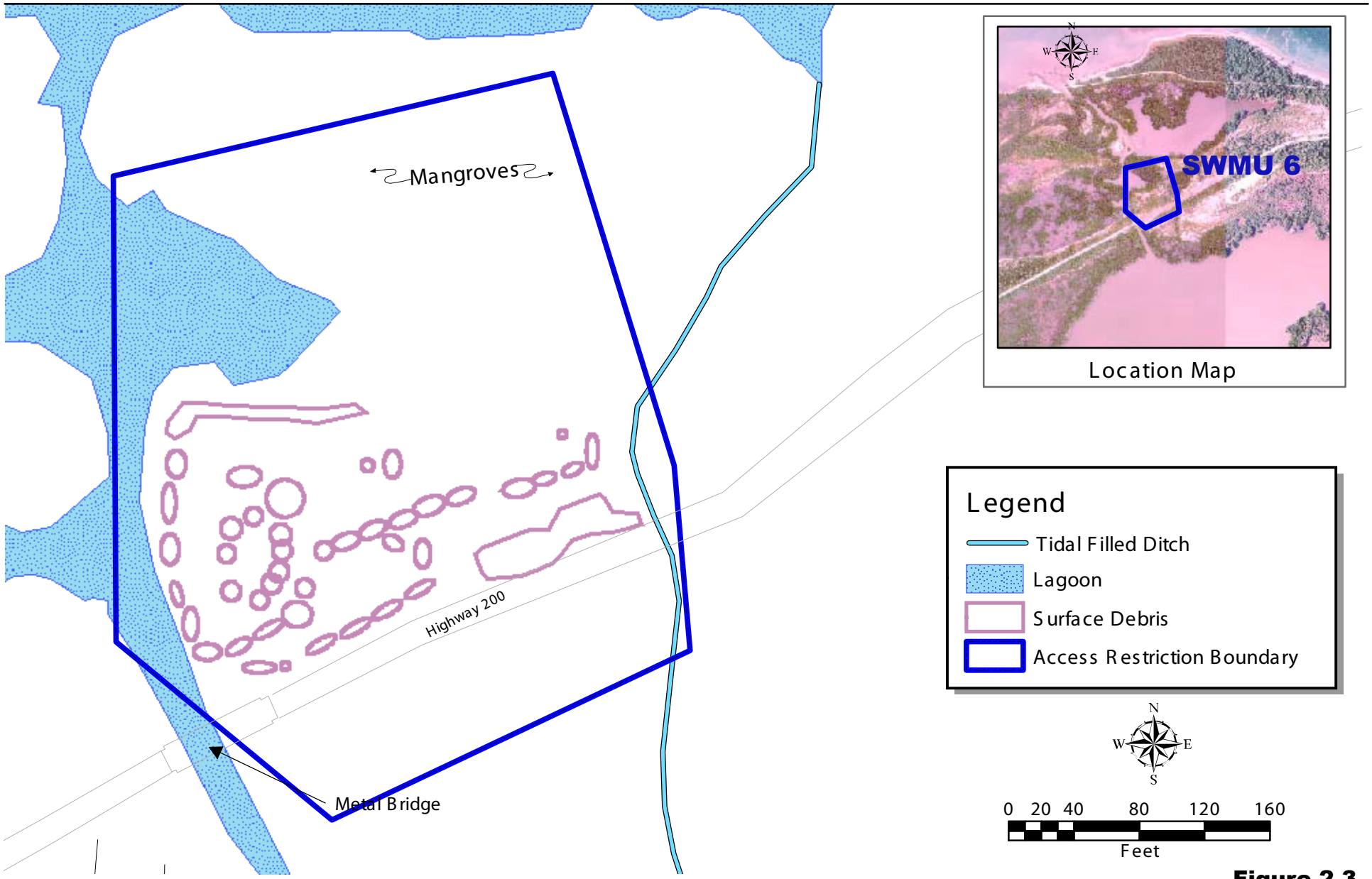
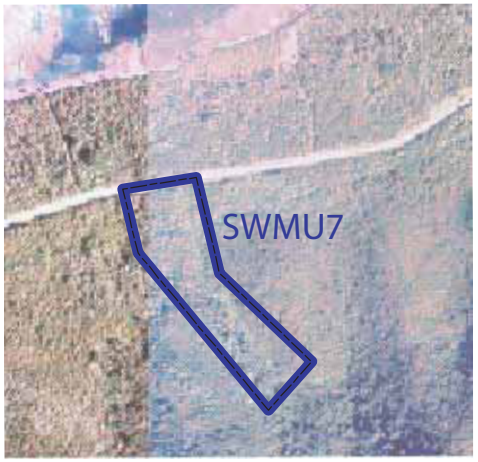


Figure 2-3

Munitions Response Area SWMU 6

Former NASD, West Vieques, Puerto Rico

CH2MHILL



Location Map

Legend

- Access Restriction Boundary
- Interpreted Waste Boundary
- Visual Waste Boundary
- Top of Ditch
- Ditch

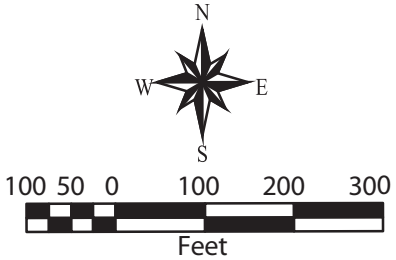


Figure 2-4

Munitions Response Area SWMU 7

Former NASD, West Vieques, Puerto Rico

CH2MHILL

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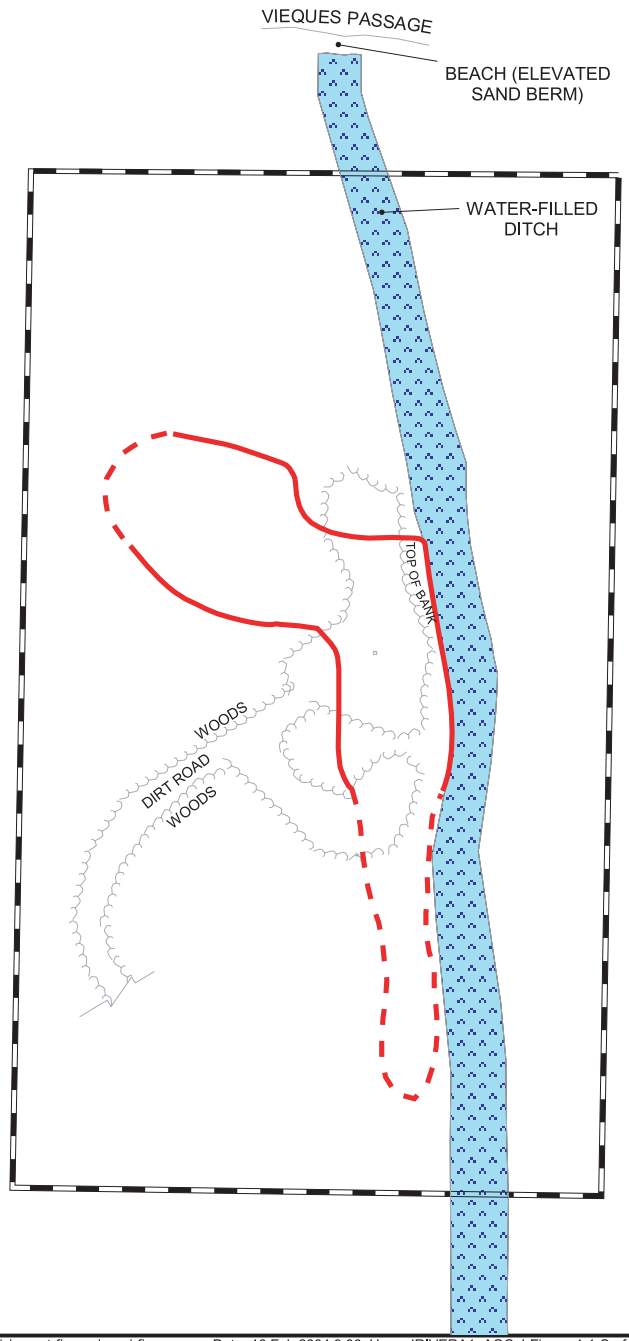
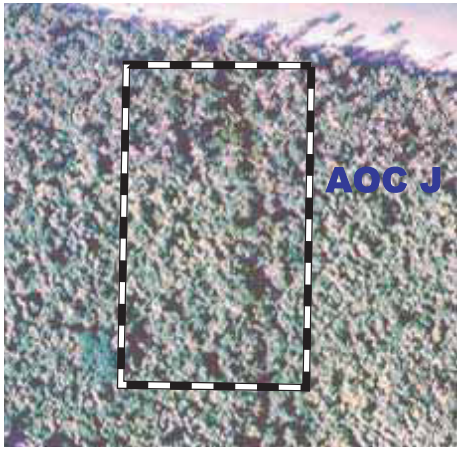




Location Map

Legend

- Access Restriction Boundary
- Interpreted Waste Boundary - Geophysics
- Visual Waste Boundary

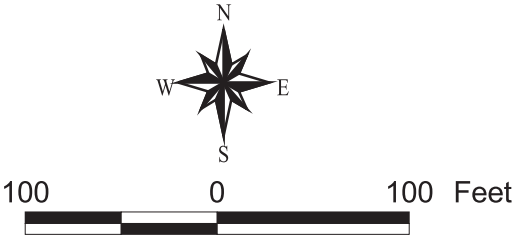
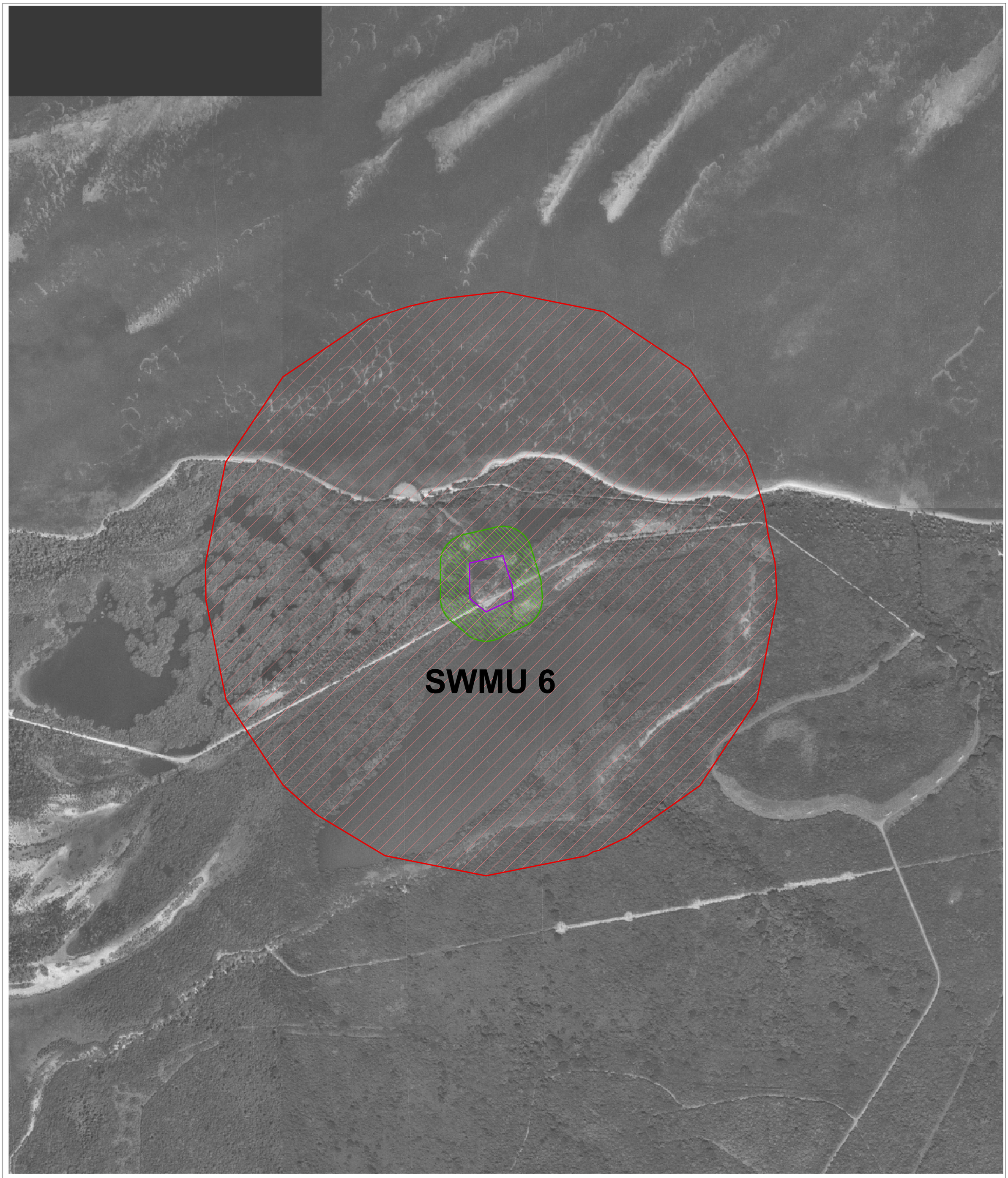


Figure 2-5
Munitions Response Area AOC J
Former NASD, West Vieques, Puerto Rico

CH2MHILL

File Path: t:\environmental\navy clean ii_vieques_ pr\former nasd\gis\report-figures\aoc j-figures.apr, Date: 16 Feb 2004 9:06, User: JRIVERA1, AOC J-Figure-4-1-Surface Soil



Legend

- SWMU Boundary
- 236' Unintentional Detonation
- 2,133' Intentional Detonation

N
W E
S

0 400 800 Feet

Figure 2-7
Exclusion Zones
SWMU 6
Former NASD, West Vieques, Puerto Rico

CH2MHILL

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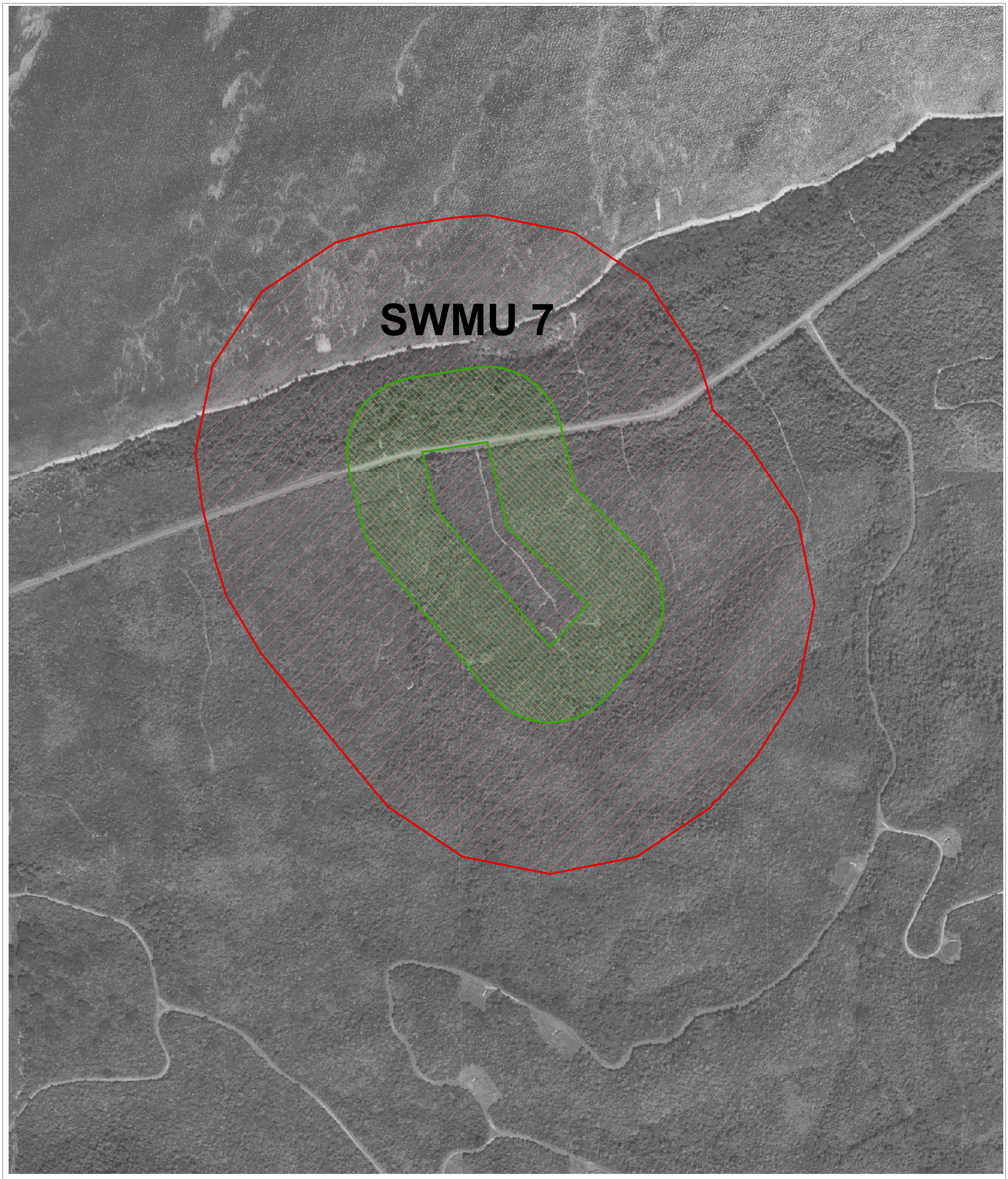

Legend

- SWMU Boundary
- 456' Unintentional Detonation
- 1,375' Intentional Detonation

N
W E
S

0 300 600 Feet

Figure 2-8
Exclusion Zones
SWMU 7
Former NASD, West Vieques, Puerto Rico

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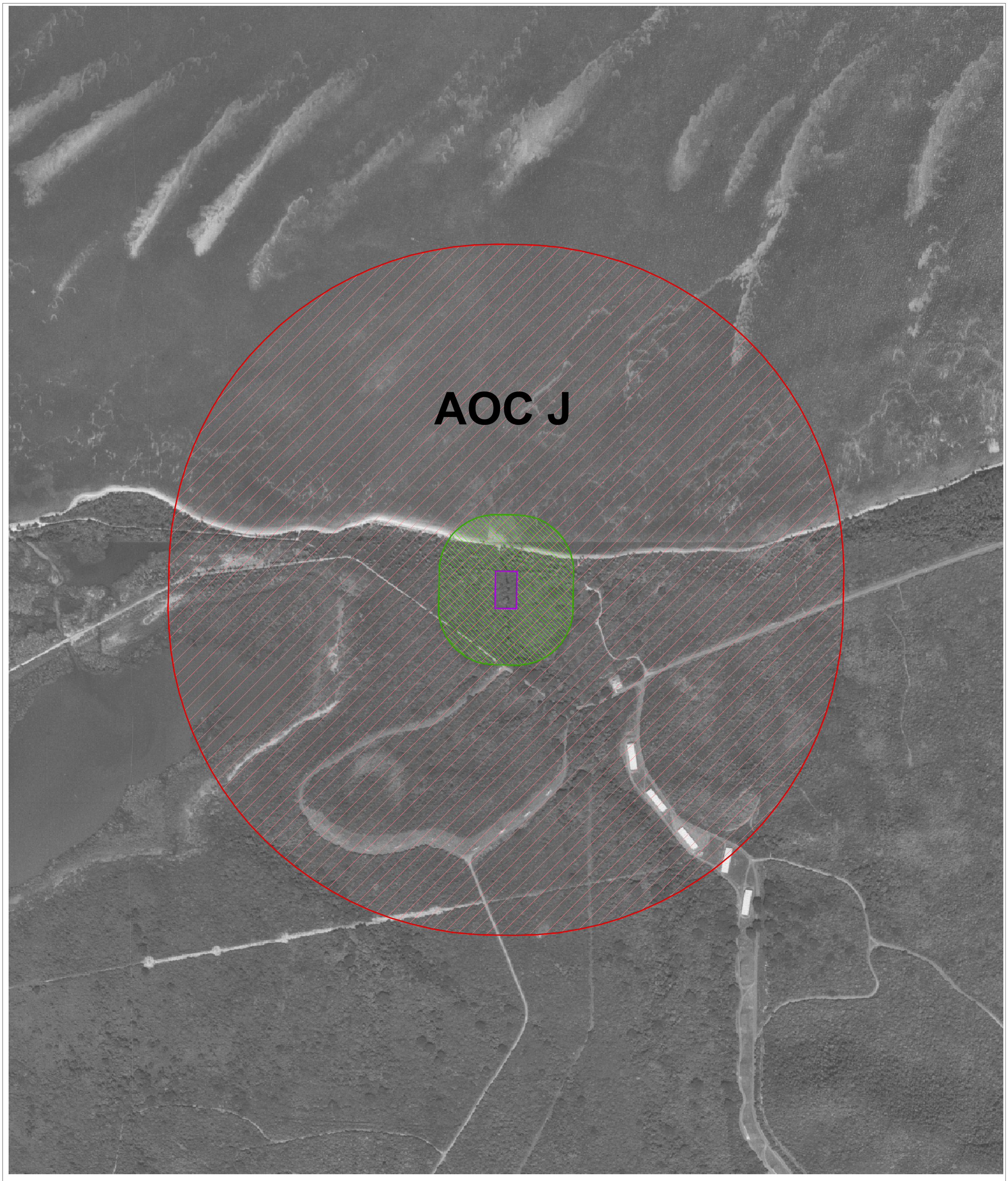

Legend

- AOC Boundary
- 456' Unintentional Detonation
- 2,641' Intentional Detonation

N
W E
S

0 400 800 Feet

Figure 2-9
Exclusion Zones
AOC J
Former NASD, West Vieques, Puerto Rico

CH2MHILL

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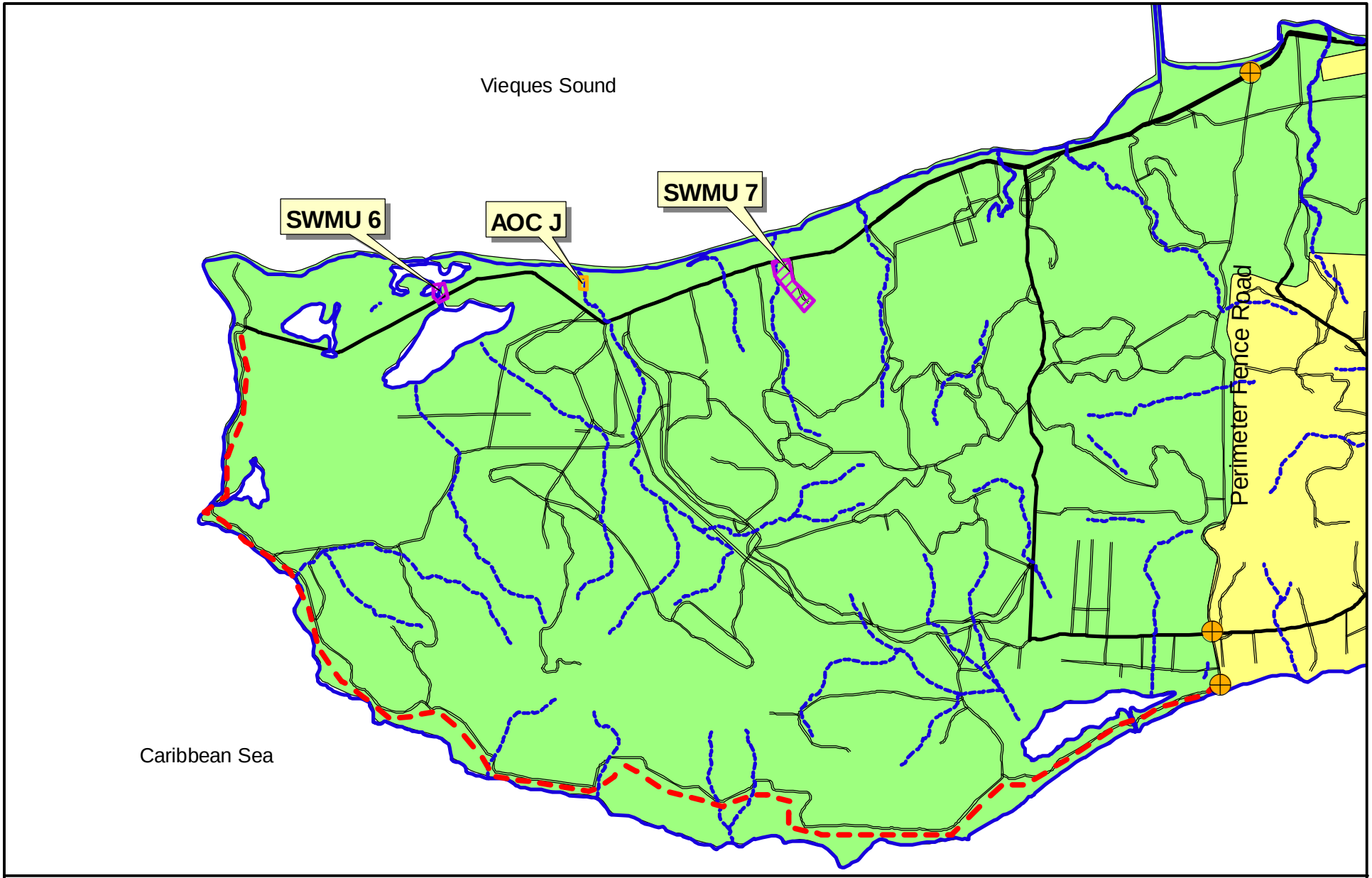

Traffic Control Point
FOOT TRAIL
SWMUs 6 and 7
AOC J
STREAMS
MAJOR ROADS
MINOR ROADS

N

0 700 1400 Feet

Figure 2-10
Traffic Control Points for
SWMU 6, SMWU 7, and AOC J Restricted Area
Former NASD, West Vieques, Puerto Rico

CH2M HILL

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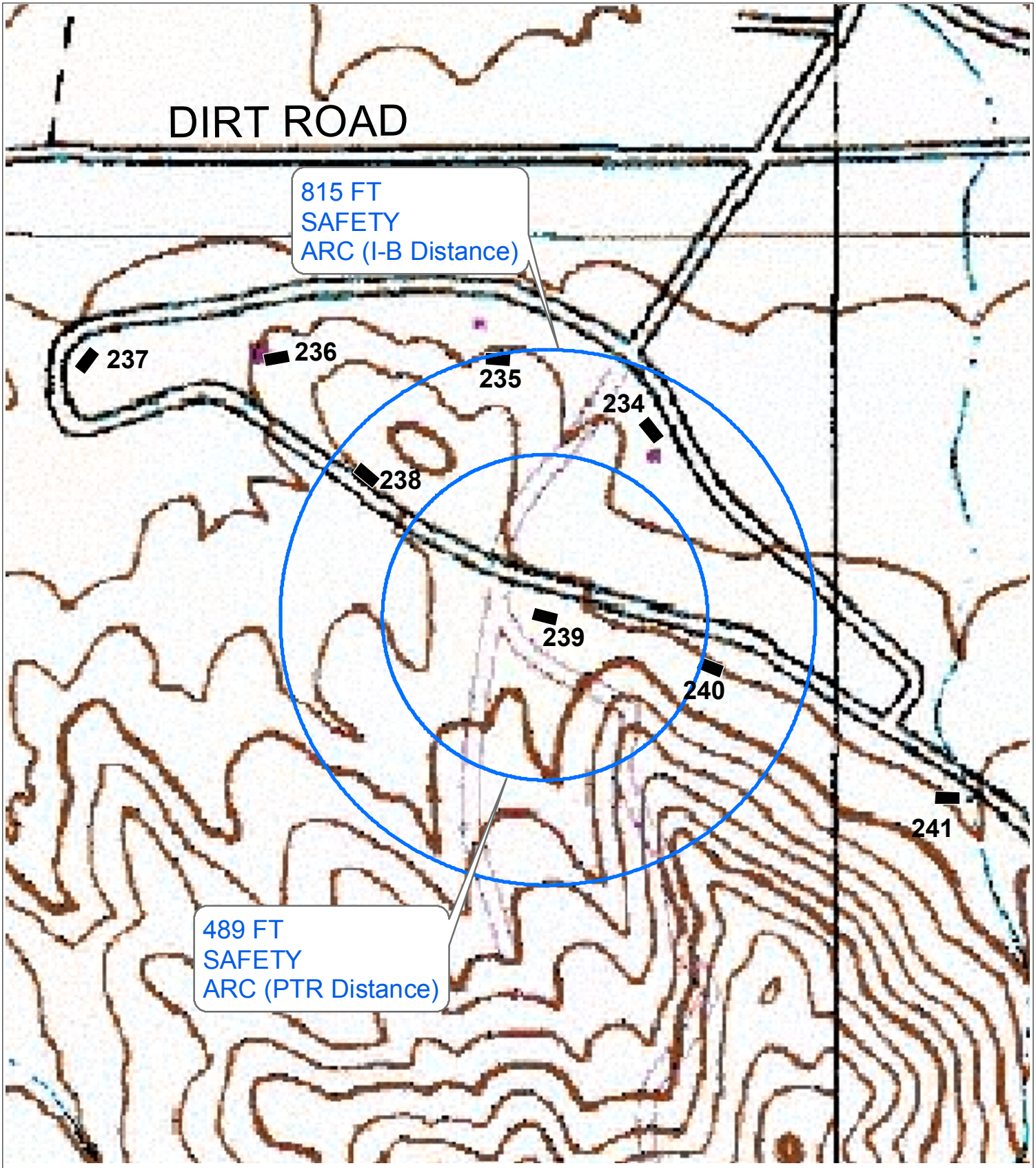

Deactivated Earth-Covered Magazines (unoccupied)

N

0 200 400 Feet

Figure 2-11
Building 239, Earth-Covered Magazine Safety Arcs
Former NASD, Vieques Island, Puerto Rico

CH2MHILL

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SECTION 3

Amount/Type of MEC (MEC Overview and MGFD)

2.1 General

2.1.1 SWMU 6 MEC Overview

Records of the amount and type of MEC disposed of at SWMU 6 are not available. Table 3-1 lists the type and amount of ordnance encountered during previous field studies of the SWMU 6 site.

TABLE 3-1
MEC Items Encountered During Previous Studies of SWMU 6

Nomenclature	Quantity
Mk 15 Practice Bomb, 100-pound (Water or Wet Sand Filled) Empty	12 (Approximately)
SUU-7/A Bomb Dispenser	2

2.1.2 SWMU 7 MEC Overview

Records of the amount and type of MEC disposed of at SWMU 7 are not available. Table 3-2 lists the type and amount of ordnance encountered during previous field studies of the SWMU 7 site. All external features indicate that ordnance contains no explosive components.

TABLE 3-2
MEC Items Encountered During Previous Studies at SWMU 7

Nomenclature	Quantity
Mk 56 Drill Mine	2
LAU-3 Rocket Launcher (2.75-inch)	10 (Approximately)
Ammunition Cans	Assorted

2.1.3 AOC J MEC Overview

Records of the amount and type of MEC disposed of at AOC J are not available. Table 3-3 lists the type and amount of ordnance encountered during previous field studies of AOC J. All external features indicate that ordnance contains no explosive components.

TABLE 3-3
MEC Items Encountered During Previous Studies at AOC J

Nomenclature	Quantity
106 mm Cartridge Case	2
106 mm Cartridge Base	1
Flash Tube	1
Cartridge Canister	6

2.1.4 Munition with the Greatest Fragmentation Distance (MFGD)

Based on available data, the MGFD is the BLU-3/B Bomblet at SWMU 6, the M229 rocket warhead at SWMU 7, and the M346 106mm Projectile at AOC J. Table 3-4 presents the calculated fragment and overpressure distances for these items.

During MR actions, if an item is encountered with a greater fragmentation distance, the Q-D arcs will be adjusted based on the item encountered and an amendment to the safety submission initiated.

TABLE 3-4
Fragment and Calculated Overpressure Distances

MR Area	Munition[a]	Hazardous Fragment Distance[b] (ft)	Maximum Fragment Distance[c] (ft)	K50 Over Pressure Distance[d] (ft)
SWMU 6	BLU-3/B Bomblet	236	2133	39
SWMU 7	MK 24 Rocket Warhead (5-inch)	456	1375	94
AOC J	M346 106 mm Projectile	456	2641	100

[a]For Q-D siting purposes, the MGFD and resulting EZ distances will be established for SWMU 4, based on the greatest of the fragmentation or overpressure distances for the types of MEC expected to be encountered. Fragment hazard distances will be recalculated based on the NEW, in the event different MEC are found.

[b]OP5, Volume 1, Tables 7-8 and 7.9, and DDESB TP-16, the distance at which fragments do not exceed a hazardous fragment density of one hazardous fragment per 600 ft^2, where a hazardous fragment is defined as a fragment having an impact energy of 58 ft-lb or greater.

[c]OP5, Volume 1, Tables 13.1 and 13.2, and DDESB TP-16, the maximum distance at which a fragment of the munition may travel based on munition design specifications and maximum trajectories.

[d]As calculated in accordance with OP5, Volume 1, Table 7.9 and DDESB TP-16, the distance at which blast overpressure exposure from Hazard Division 1.1 explosives is less than 0.90 psi—the lower end of the permissible exposure level for inhabited buildings and administrative and housing areas per Chapter 2, para. D.6 of DoD 6055.9-STD. Determined from the formula: $50 \times NEW^{1/3}$.

2.1.5 Explosive Soils or Facilities

No explosive soils or explosive-contaminated buildings are expected to be encountered.

2.1.6 Other Description or Status of MRS Not Previously Covered

Much of the SMWU 6 and 7 and AOC J areas are densely vegetated. This could make all aspects of this project more difficult.

SECTION 4

Start Date

3.1 Schedule

Work on this project is projected has not been scheduled, however is anticipated to begin in early 2006.

SECTION 5

Frostline (Site Stability)

There is no frostline due to the tropical climate of the site. However, the island of Vieques is subject to potentially significant weather events such as tropical storms and hurricanes. These storms could cause accelerated surface erosion exposing or relocating near-surface MEC. Following these events, a visual inspection of cleared areas will be accomplished to determine if MEC has been exposed or relocated. Action will be taken based on the results of these inspections.

SECTION 6

Response Techniques

5.1 Introduction

The techniques used to detect, recover, and destroy MEC are summarized below. More detailed information may be found in the Site Specific Work Plans.

5.2 Surface Clearance of MEC

Prior to vegetation removal, environmental sampling, or DGM the UXO subcontractor will carefully inspect the sites with the aid of an appropriate geophysical instrument (e.g., Schonstedt GA-52Cx magnetometer) for evidence of munitions on the ground surface.

The surface clearance will remove MEC (deemed safe to move); munitions and range-related debris; and other metallic objects that could interfere with vegetation removal, environmental sampling, or geophysical survey.

MEC items that are too hazardous to move or cannot be positively identified will be disposed of by the UXO subcontractor using blow-in-place (BIP) methods.

Since it is anticipated for the MR Action to be regulated under CERCLA, a RCRA permit will not be required. However, the treatment will satisfy the intent.

The UXO Technicians will provide a detailed description of the type and quantity of MEC encountered, including nomenclature, condition, and location found.

Any mechanized equipment to include support equipment such as lifting slings used to lift/move MEC items that have not been positively identified as being inert must comply with the requirements of OP 5, Chapter 10 (Ordnance Handling Equipment).

5.3 Vegetation Removal

Vegetation will be removed from sites in SWMUs 6 and 7 and AOC J to allow for site survey, environmental sampling, and DGM survey.

Vegetation removal methods may include vehicle-mounted mowers or hand-grubbing using chainsaws, axes, machetes, clippers etc.

The possibility of using "Controlled-Burning" to remove vegetation is currently being evaluated with the EQB for the Commonwealth of Puerto Rico.

Prior to any mechanized vegetation removal, UXO personnel will conduct a magnetometer assisted visual sweep for MEC. If required, surface MEC will be disposed of or removed before vegetation removal begins. Once the UXO personnel have completed the visual sweep they will move ahead to areas awaiting visual surface sweeping to insure a minimum separation distance of 500 ft. is maintained between the mechanized system and UXO

personnel. If the operator sees anything in his path that warrants re-inspection the cutting will be momentarily halted and the UXO personnel will be brought back in to inspect the item of concern. MEC items identified during the initial surface sweep that cannot be safely removed will be conspicuously marked and the mechanized vegetation system operator thoroughly briefed before the commencement or continuation of brush clearing.

While a visual survey/sweep of the ground surface will always be conducted before any mechanized vegetation removal, the possibility exits for smaller munitions (75mm or less) to remain obscured at the ground surface due to the dense vegetation. Therefore, when utilizing vehicle mounted brush cutting equipment, the operator shall be adequately shielded to protect against an unintentional detonation of any items not discovered during the initial MEC surface sweep/survey.

It is anticipated that mechanized brush cutting will be accomplished using the TAZ II Caterpillar which has been armored with 0.75-in.-thick A572 steel plating and 2.65-in.-thick bullet resistant glass to protect the operator from the fragments and blast from an unintentional detention of a 75mm HE M48 projectile. Exhibits 6.3-1 through 6.3-4 provide detailed photographs of the TAZ II Caterpillar. Appendix A describes how this procedure and the associated engineering controls will address the concern of unintentional detonation during mechanized vegetation clearing.

EXHIBIT 6.3-1
TAZ II Caterpillar

EXHIBIT 6.3-2

TAZ II Caterpillar Armoring (Front View of Cab)

EXHIBIT 6.3-3
TAZ II Caterpillar Armoring (Side View of Cab)

EXHIBIT 6.3-4
TAZ II Caterpillar (Vegetation Cutting Head)

If vegetation is so dense that it prevents a thorough surface sweep, a systematic "Step-Down" method of brush clearance will be instituted to allow for a complete surface clearance/survey. All cutting will stop 6 inches above ground level. This will prevent disturbing subsurface MEC that may be entangled in plant roots if the roots are ripped from the soil.

Care will be taken to ensure cut trees and branches land in areas where MEC surface clearance has been performed. Cut trees and branches will not be dragged or carried through areas where surface MEC sweep has not been conducted.

After completion of vegetation clearance a comprehensive MEC identification and data collection will be completed.

5.4 Digital Geophysical Mapping (DGM)

To estimate the amount and extent of subsurface munitions at selected munitions response sites limited DGM may be conducted. This survey will use EM-61 electromagnetic geophysical equipment and Schonstedt GA-52Cx magnetometer (or industry equivalents). Selection of geophysical instrument will be based on field conditions such as soil types, accessibility, terrain, and vegetation density.

No Geophysical Prove-Out (GPO) is planned, however, geophysical test plots will be established to verify proper functioning of instruments in varied soil types.

The equipment will be carried over the sites by a two-person team along transects. Readings of subsurface metallic anomalies, indicative of buried munitions, will be collected continuously along each transect or recorded separately by global positioning system (GPS) instruments.

Selected geophysical instruments are considered "passive" and do not require certification under the Navy's Hazards of Electromagnetic Radiation to Ordnance (HERO) program. RF transmitters, such as cellular phones and portable radios, will not be used in areas where EMR-susceptible MEC has been visually identified on the surface or in areas where disposal of susceptible MEC is documented.

Precise locations of the geophysical anomalies, using the latitude and longitude coordinate system, will be established using a GPS. Items identified by the survey team will be flagged for removal by follow-on UXO removal teams. The procedures for completing the geophysical survey are detailed in the Site Specific Work Plans.

These geophysical survey methods were selected based on best available technology for the scope of work and allocated funds. No limitations to survey methods or equipment are expected based on terrain or soil type. If problems are encountered in the field selected methods/equipment will be reevaluated.

5.5 Subsurface Removal of Munitions

No subsurface removal of MEC is planned for the MR Actions at SWMUs 6 and 7 and AOC J. However, limited subsurface removal of MEC may become necessary to ensure field team safety. Subsurface anomalies that interfere with environmental sampling or site access by vehicles may require intrusive investigation.

During intrusive investigations in the SWMU 6 Site a 236 feet (ft) exclusion zone (EZ) will be established around the excavation site. In the SWMU 7 and AOC J Sites a 456ft EZ will be established. Only essential personnel will be allowed in the EZ during excavation. Heavy equipment may be used for initial digging, however, the final 12 inches of earth will be excavated using only hand-tools.

5.6 Treatment and Disposal of Munitions

Each MEC item discovered on the surface during MR Activities will be inspected by at least two qualified UXO personnel. MEC items which are too hazardous to move or cannot be positively identified will be disposed of using BIP procedures.

MEC items known or suspected to contain an explosive hazard will be treated/disposed of using standard demolition techniques. MEC items determined safe to move may be transported to a designated consolidation point within the MR Site for disposal.

All MEC disposal operations will be performed under the direct supervision of an on-site Senior UXO Supervisor (SUXOS). An on-site UXO Site Safety Officer (UXOSO) will closely monitor operations, strictly enforce safety, ensure adherence to procedures, and verify the exclusion area is evacuated of non-essential personnel.

5.7 Quality Control & Quality Assurance Standards

This is an assessment and inspection of potential MR Sites. UXO Teams are expected to sweep and clear work sites of surface MEC and provide safety escort and MEC avoidance

support to project personnel. Subsurface anomalies will be recorded, but not excavated unless they present a hazard to project personnel or interfere with environmental sampling. Quality Control (QC) testing will be accomplished by the UXO Quality Control Supervisor (UXOQCS) and site Geophysicist. No subsurface anomalies will be excavated. The following definable features of work and minimum standards will serve as the foundation of the QC process.

5.7.1 Site Survey and Mapping

All work sites must be accurately mapped. Grids and transects will be laid out with reference to control points/monuments.

5.7.2 Surface MEC Removal/Disposal

Surface MEC must be removed from site prior to initiating any other activity (e.g. vegetation removal and subsurface anomaly acquisition).

5.7.3 Vegetation Removal

All vegetation less than 4 inches in diameter must be cleared to within 6 inches of ground level before subsurface anomaly acquisition can be accomplished.

5.7.4 Subsurface Anomaly Acquisition/Data Collection

All MR sites must have complete coverage with handheld detectors or DGM instruments. Location of anomalies will be marked in the field (flagged) and recorded in a Global Information System (GIS) database.

5.7.5 Material Potentially Presenting an Explosive Hazard (MPPEH) Disposition

All munitions debris and range-related debris will be inspected, verified, and certified free of all explosive hazards before release to a qualified recycle facility.

CH2M HILL MR Personnel will oversee subcontractor activities to ensure complete coverage of selected assessment/investigation sites.

5.8 Personnel Qualifications

All project personnel will meet the training and experience requirements outlined in the United States Army Corps of Engineers (USACE) Engineer Pamphlet 1110-1-18 and DDESB Technical Paper No. 18.

5.9 Certification and Processing of MPPEH

This inspection, inert inspection, and certification of MPPEH shall be conducted in accordance with DoD 4160.21-M, Chapter 4, Paragraph B.3.

MPPEH recovered during the clearance process will be inspected by at least two qualified UXO Technicians. MPPEH will be segregated into items known or suspected to contain

explosive hazard(s) and items known to contain no explosive hazard. Inert verification will be accomplished by a technically qualified DoD person.

MPPEH known or suspected to contain an explosive hazard will remain on the munitions response site awaiting further treatment/demilitarization. MPPEH known to be free of all explosive hazards will be demilitarized (if required) and transported to a secure area and sorted into Munitions Debris and Range-Related Debris.

MPPEH which require further treatment/demilitarization will undergo an additional inspection by at least two qualified UXO Technicians following treatment/demilitarization. This inspection will be verified by the SUXOS and UXOSO before the MPPEH is transported to the secure holding area.

Treatment of MPPEH may include exposing internal cavities/inert fillers with explosive charges to allow visual inspection. Demilitarization may include crushing, bending, or deforming.

Containers used to hold inert certified and verified material will be secured to prevent the addition of unauthorized material and will be labeled with the following signed statement:

"This certifies and verifies that the AEDA residue, Range Residue and/or Explosive Contaminated property listed has been 100 percent properly inspected and to the best of our knowledge and belief, are inert and/or free of explosives or other dangerous materials."

Prior to final disposition, the Project Manager and NAVFAC RPM will arrange for a U.S. Navy employee to conduct a final certification of all MPPEH. This person must be designated in writing as trained and qualified to conduct this certification. This person will also sign all required documents required for disposition (e.g. DD Form 1348-1 with demilitarization/inert statement). This final certification should be conducted just prior to transfer to a qualified recycle facility.

During the entire process an accurate and unbroken chain of custody must be maintained for all MPPEH placed in the secure holding area. This will be accomplished through a well documented inspection/certification/verification process, key-issue logs (to the secure area) and up-to-date inventories of collected MPPEH.

5.10 Soil and Groundwater Sampling

5.10.1 Surface Soil Sampling

Before surface soil sampling activities are initiated, the desired or suitable alternate locations will be cleared by a UXO Contractor utilizing a Schonstedt GA-52Cx or equivalent magnetometer.

Soil Sampling Techniques typically employed include stainless steel trowel sampling and stainless steel hand auger sampling, depending on the nature of the material to be sampled. A stainless steel trowel will be used to collect samples of loosely packed materials and a stainless steel hand auger for densely packed materials. Surface soil samples will be obtained from a depth of 0 to 6 inches below ground surface (bgs).

5.10.2 Soil Borings

Before soil borings are initiated, the location will be inspected for MEC contamination by the UXO Contractor. Borings will be advanced using one of the following methods: a truck-mounted drill rig, an all-terrain vehicle (ATV) drill rig, using a split-spoon sampler or a hand auger.

A magnetometer with down-hole monitoring capabilities will be used for all borings greater than 4-ft in depth. The borehole will be started with a hand held portable auger. At not more than a 1-foot depth, the auger will be withdrawn and the magnetometer lowered into the borehole. This procedure will ensure smaller MEC items, undetectable from the surface, are identified. If no magnetic anomalies are found, the procedure will be repeated at 1-foot intervals to the maximum depth of the auger, but no less than 4-ft. If the proposed borehole site is free of magnetic anomalies, the drilling equipment may be brought on site and utilized. Borehole monitoring with the magnetometer shall continue at 1-foot intervals, until the sampling is completed.

If MEC contamination prevents access to the sampling site(s) by the drilling equipment, soil borings may be advanced using a hand auger or a tripod-mounted sampling device. The locations and total depth of hand-augured boreholes will be determined in the field based upon subsurface soil conditions and site-specific sampling objectives.

SECTION 7

Quantity-Distance Overview

This section contains information on various activities and locations planned for the three sites where a minimum safe distance is required for MEC operations. These activities and locations are described below and are illustrated in the figures provided in Section 2, Description of ESS Related Site Maps.

6.1 MEC Areas

6.1.1 Restricted Area

At SWMUs 6 and 7 and AOC J, the Restricted Area is defined as all land west of "Perimeter Fence Road." This is the north-south road that defines the eastern boundary of the Former NASD. Access to this Restricted Area will be controlled by three traffic-control points. One traffic-control point will be located at the intersection of Highway 200 and Perimeter Fence Road; the second traffic-control point will be at the intersection of Highway 201 and Perimeter Fence Road. The third traffic-control point will prevent pedestrians from accessing the foot-trail that runs along the southeast coast of the VNWR.

Restricted Areas will only be established and enforced during activities directly related to Munitions Response. Although all facilities within the proposed restricted areas are uninhabited, the areas will be thoroughly checked before any MR Activities commence. Once established, only project personnel and authorized, escorted visitors are allowed within the Restricted Area.

6.1.2 Exclusion Zones

An Exclusion Zone is a safety arc established around MR activities. EZs are established within the Restricted Area to further limit access to sites with MEC intrusive operations or planned detonations.

EZs are 3-dimensional. A Notice-to-Airmen will be coordinated through local Federal Aviation Administration (FAA) officials.

If an EZ extends beyond the land boundaries of the Restricted Area, institutional and engineering controls will be established and enforced to ensure public safety. These controls include public safety announcements, roadblocks, evacuation, tamping of disposal shots, and relocating munitions prior to disposal.

If an EZ extends into coastal waters, procedures will be implemented to prevent public exposure to the hazard. A Notice-to-Mariners will be coordinated through local Coast Guard officials. Project personnel will be posted at key vantage points on the beach to observe any encroachment into the exclusion zone. These sentries will be in radio/cell

phone communication with the work site supervisor. A small boat or personal watercraft will be available to relay a warning to boats entering the exclusion zone.

6.1.3 Unintentional Detonation EZ

Prior to the start of any MEC intrusive operations an EZ for unintentional detonation will be established. This EZ is based upon the hazardous fragment distance for the Net Explosive Weight (NEW) of the MGFD.

6.1.3.1 EZ for Unintentional Detonation in SWMU 6

The unintentional detonation EZ in SWMU 6 during MEC intrusive operations is 236-ft. Distance is based on NEW for the MGFD (0.35lb for the BLU-3/B Bomblet) and derived from NAVSEA OP 5 Vol. 1, Table 7-9.

6.1.3.2 EZ for Unintentional Detonation in SWMU 7

The unintentional detonation EZ in SWMU 7 during MEC intrusive operations is 456-ft. Distance is based on NEW for the MGFD (4.8lbs for the M229 Rocket Warhead [2.75-inch]) and derived from NAVSEA OP 5 Vol. 1, Table 7-9.

6.1.3.3 EZ for Unintentional Detonation in AOC J

The unintentional detonation EZ in AOC J during MEC intrusive operations is 456-ft. Distance is based on the NEW for the MGFD (8lbs for the M346 106mm Projectile) and derived from NAVSEA OP 5 Vol. 1, Table 7-9.

6.1.4 Intentional Detonation EZ

Prior to intentional detonation of any MEC item(s) an EZ will be established. Intentional detonation EZ is based on the Maximum Case Fragment Throw Range. Rogue fragments may travel farther.

MEC items should be arranged in the safest possible configuration. Lugs and strong-backs should point downward. Base-plates and noses should point in the safest possible direction. Counter-charges should be placed to prevent shaped charge jets from forming during disposal. Counter-charges should be placed underneath MEC items containing white phosphorous (WP) to prevent unconsumed WP from becoming mixed with the soil.

The SUXOS and UXOSO will closely monitor the type and quantity of MEC items in consolidation shots to ensure maximum NEW is not exceeded.

Manned roadblocks will be established on all access roads leading to intentional detonation sites. Personnel at these roadblocks will be in radio or cell phone communication with each other, the SUXOS, and the UXOSO.

6.1.4.1 EZ for Intentional Detonation in SWMU 6

The intentional detonation EZ in SWMU 6 is 2,133-ft. This is based on the Maximum Case Fragment Throw Range of the MGFD (BLU-3/B Bomblet). Intentional detonations in these MR Areas will not exceed 50lbs NEW.

If MEC item(s) are significantly smaller than 5 inches in diameter, the EZ can be recalculated and reduced using NAVSEA OP 5 Vol. 1, Table 13-1 or 13-2.

6.1.4.2 EZ for Intentional Detonation in SWMU 7

The intentional detonation EZ in SWMU 7 is 1,375-ft. This is based on the Maximum Case Fragment Throw Range of the MGFD (M229 Rocket Warhead). Intentional detonations in this MR Area will not exceed 50lbs NEW.

If MEC item(s) are significantly smaller than 5 inches in diameter, the EZ can be recalculated and reduced using NAVSEA OP 5 Vol. 1, Table 13-1 or 13-2.

6.1.4.3 EZ for Intentional Detonation in AOC J

The intentional detonation EZ in AOC J is 2,641-ft. This is based on the Maximum Case Fragment Throw Range of the MGFD (M346 106mm Projectile). Intentional detonations in AOC J will not exceed 25lbs NEW (including demolition materials).

If MEC item(s) are significantly smaller than 4.2 inches in diameter, the EZ can be recalculated and reduced using NAVSEA OP 5 Vol. 1, Table 13-1 or 13-2.

6.1.5 Inhabited Building (IB) and Public Transportation Route (PTR) Distances

The Inhabited Building Distance (IBD) is the minimum permissible distance between an inhabited building and a potential explosion site. The Public Transportation Route Distance (PRTD) is calculated at 60 percent of the IBD.

Based on the MGFD of a BLU-3/B Bomblet, the IBD from the munitions response sites in SWMU 6 during this investigation is 236-ft. The PRTD is calculated at 60 percent of the IBD, in this case, 142-ft. There are no inhabited buildings within the IBD of SWMU 6, however, Highway 200 does fall within the PRTD.

For the MGFD of a M229 Rocket Warhead at SWMU 7, the IBD is 456-ft and the PRTD is 274-ft. There are no inhabited buildings within the IBD of SWMU 7, however, Highway 200 does fall within the PRTD.

For the MGFD of a M346 106mm Projectile, the IBD is 456-ft and the PRTD is 274-ft. There are no inhabited buldings within the IBD of AOC Jand no public transportation routes within the PRTD.

If PTR Distance from SWMUs 6, 7, or AOC J extend into coastal waters, procedures will be implemented to prevent public exposure to the hazard. A Notice-to-Mariners will be coordinated through local Coast Guard officials. Project personnel will be posted at key vantage points on the beach to observe any encroachment into the PTRD. These sentries will be in radio/cell phone communication with the work site supervisor. A small boat or personal watercraft may be employed to relay a warning to boats encroaching within the PTRD.

TABLE 7-1
Inhabited Building Distance (IBD) and Public Transportation Route Distance (PTRD)

Storage Facility/MR Area	MGFD	NEW (lbs)	Hazardous Fragment Distance (ft)	IBD (ft)	PTRD (ft)
SWMU 6	BLU-3/B Bomblet	0.35	236	236	142
SWMU 7	M229 Rocket Warhead	4.8	456	456	274
AOC J	M346 106mm Projectile	8.0	456	456	274

6.1.6 Team Separation Distances

Team separation distances provide a safety zone between work sites.

6.1.6.1 Team Separation Distances in SWMU 6

Teams working within SWMU 6 will remain a minimum of 50-ft separation distance. This is based on the K50 over pressure distance of 39-ft for the BLU-3/B Bomblet, the MGFD for this MR Area.

6.1.6.2 Team Separation Distances in SWMU 7

Teams working within SWMU 7 will remain a minimum of 100-ft separation distance. This is based on the K50 over pressure distance of 94-ft for the MK 24 Rocket Warhead, the MGFD for this MR Area.

6.1.6.3 Team Separation Distances in AOC J

Teams working within AOC J will remain a minimum of 100-ft separation distance. This is based on the K50 over pressure distance of 100-ft for the M346 106mm Projectile, the MGFD for this MR Area.

6.1.7 Preliminary Site Work

Preliminary site work such as elevation surveys, geophysical surveying and detecting anomalies does not require an Exclusion Zone for Q-D purposes.

6.2 Explosive Storage Magazines

A type 2 portable explosives storage magazine, as described in Section 55.208 (a) (4) of ATF P 5400.7, ATF Explosives Law and Regulations has been sited at the entrance to Earth Covered Magazine (ECM) 239. Per NOSSA letter 8020 Ser N7112/30 of 11 Jan 02, this magazine was approved for the storage of demolition explosives for munitions response operations. The magazine has been sited for 150 pounds of Hazard Class/Division 1.1 Explosives. The IBD for the portable magazine is 815-ft and the PTRD is 489-ft.

Details on the storage of explosives for the SWMU 6 and AOC J work sites can be found in the *Final Magazine Siting Plan for SWMU 4, SWMU 6and AIC J, Former NASD, Vieques Island Puerto Rico (CH2M HILL, 26 November 2001).*

6.3 Planned or Established Treatment Sites

6.3.1 Open Detonation in SWMU 6

Disposal by detonation will be conducted at the munitions response site where the MEC is found. MEC items from two or more sites may be consolidated if they are safe to move. No single disposal event will exceed 50 lbs. NEW H/C 1.1.

The exclusion zone for disposal operations will be 3,000-ft, based on MGFD of all sites (M346 106mm Projectile) and rounded up for an added measure of safety. The applicable Notice to Airmen and Notice to Mariners will be issued prior to all planned detonations.

Traffic control will be established by restricting entry via Highway 200. Personnel monitoring the traffic control points will be in radio or cell phone communication with the SUXOS and UXOSO.

6.3.2 Open Detonation in SWMU 7

Disposal by detonation will be conducted at the munitions response site where the MEC is found. MEC items from two or more sites may be consolidated if they are safe to move. No single disposal event will exceed 50 lbs. NEW H/C 1.1.

The exclusion zone for disposal operations will be 3,000-ft, based on MGFD of all sites (M346 106mm Projectile) and rounded up for an added measure of safety. The applicable Notice to Airmen and Notice to Mariners will be issued prior to all planned detonations.

Traffic control will be established by restricting entry via Highway 200. Personnel monitoring the traffic control points will be in radio or cell phone communication with the SUXOS and UXOSO.

6.3.3 Open Detonation in AOC J

Disposal by detonation will be conducted at the munitions response site where the MEC is found. MEC items from two or more sites may be consolidated if they are safe to move. No single disposal event will exceed 25 lbs. NEW (including demolition material).

The exclusion zone for disposal operations will be 3,000-ft, based on MGFD of all sites (M346 106mm Projectile) and rounded up for an added measure of safety. The applicable Notice to Airmen and Notice to Mariners will be issued prior to all planned detonations.

Traffic control will be established by restricting entry via Highway 200. Personnel monitoring the traffic control points will be in radio or cell phone communication with the SUXOS and UXOSO.

6.4 Footprint Areas

There are three types of footprint areas: BIP, MEC collection points within a search grid, and consolidated shots within a search grid. The following outlines the size of the exclusion zone that apply around these areas:

6.4.1 SWMU 6 Blow-in-Place

A 2,133-ft exclusion area (based on MGFD of BLU-3/B Bomblet) will be established around all MEC which is too dangerous to move and requires BIP disposal. Prior to disposal, it may be necessary for UXO and Geophysical teams to continue working inside the exclusion zones in order to locate and identify other MEC requiring BIP disposal. This will allow for more efficient disposal operations.

6.4.2 SWMU 7 Blow-in-Place

A 1,375-ft exclusion area (based on MGFD of M229 Rocket Warhead) will be established around all MEC which is too dangerous to move and requires BIP disposal. Prior to disposal, it may be necessary for UXO and Geophysical teams to continue working inside the exclusion zones in order to locate and identify other MEC requiring BIP disposal. This will allow for more efficient disposal operations.

6.4.3 AOC J Blow-in-Place

A 2,641-ft exclusion area (based on MGFD of a M346 106 mm Projectile) will be established around all MEC which is too dangerous to move and requires BIP disposal. Prior to disposal, it may be necessary for UXO and Geophysical teams to continue working inside the exclusion zones in order to locate and identify other MEC requiring BIP disposal. This will allow for more efficient disposal operations.

6.4.4 SWMU 6 Collection Points

MEC recovered may be consolidated onsite if deemed safe to move. Collection point locations will be designated by the SUXOS and briefed during the morning safety meetings and updated as required. The NEW (including demolition materials) for collection points within SWMU 6 will not exceed 50 lbs. A 601-ft footprint area will be established around each collection point.

6.4.5 SWMU 7 Collection Points

MEC recovered may be consolidated onsite if deemed safe to move. Collection point locations will be designated by the SUXOS and briefed during the morning safety meetings and updated as required. The NEW (including demolition materials) for collection points within SWMU 7 will not exceed 50 lbs. A 601-ft footprint area will be established around each collection point.

6.4.6 AOC J Collection Points

MEC recovered may be consolidated onsite if deemed safe to move. Collection point locations will be designated by the SUXOS and briefed during the morning safety meetings and updated as required. The NEW (including demolition materials) for collection points

within AOC J will not exceed 25 lbs. A 546-ft footprint area will be established around each collection point.

6.4.7 SWMU 6 In-Grid Consolidation Shots

MEC recovered onsite may be consolidated for in-grid consolidated disposal if deemed safe to move. These consolidated disposal shots will not exceed 50 lbs. NEW (including demolition materials). The SUXOS will determine where in-grid consolidation shots will take place and ensure the appropriate exclusion zone (2,133-ft) is established.

6.4.8 SWMU 7 In-Grid Consolidation Shots

MEC recovered onsite may be consolidated for in-grid consolidated disposal if deemed safe to move. These consolidated disposal shots will not exceed 50 lbs. NEW. The SUXOS will determine where in-grid consolidation shots will take place and ensure the appropriate exclusion zone (1,375-ft) is established.

6.4.9 AOC J In-Grid Consolidation Shots

MEC recovered onsite may be consolidated for in-grid consolidated disposal if deemed safe to move. These consolidated disposal shots will not exceed 25 lbs. NEW. The SUXOS will determine where in-grid consolidation shots will take place and ensure the appropriate exclusion zone (2,641-ft) is established.

SECTION 8

Offsite Disposal

Recovered MEC will be destroyed onsite. No offsite disposal is anticipated.

SECTION 9

Environmental Considerations

Previous ecological studies of western Vieques have demonstrated that the past MEC response actions have not had a significant impact on the ecological receptors of the area. As a result, the MEC response actions are not anticipated to impact the surrounding ecological environment. However, if any natural or archeological resource may be impacted by these activities, the Environmental Resource Manager (ERM) will be contacted.

SECTION 10

Technical Support

Oversight for the MEC response actions will be provided, under the direction of NAVFAC, by a CH2M HILL UXO Safety Officer (UXOSO). Field investigations will be conducted by qualified MEC personnel including: Senior Unexploded Ordnance Supervisor, UXO Technician III and UXO Technician II field staff. MEC personnel will be drawn from CH2M HILL or qualified subcontractor(s). All UXO Technicians shall meet the training and experience requirements of DDESB Technical Paper 18.

Assistance for unknown MEC or suspected Chemical Warfare Material (CWM) will be requested through the US Army's 52d Ordnance Group at Fort Gillem, GA (404) 469-3333 or the US Navy's Explosive Ordnance Disposal (EOD) Technical Support Center at (301) 744-4069 OR 1-877-363-4636. Request(s) will be coordinated through the LANTDIV RPM and the DOI.

SECTION 11

Land Use Restrictions

Over the next few years, the DOI will develop the CCP for the VNWR. This plan will outline the land use plan for managing the refuge. Until that process is complete, it is difficult to reach a final decision on cleanup. While all military activities have ceased at the Former NASD, the U.S. Navy retains responsibility for any MEC and/or environmental concerns that may exist there.

Any land use restrictions such as access control planned for the Former NASD are expected to be consistent with those established for state and federal wildlife refuges.

SECTION 12

Public Involvement

A Technical Review Committee (TRC) was formed with members of the community and regulators in 2001. Quarterly meetings are conducted at facilities on Vieques to discuss the progress of MEC response action and environmental restoration activities as well as community concerns.

Other public involvement activities will include:

- A Community Relations Plan (CRP), developed with the U.S. Environmental Protection Agency (USEPA) in 2001, will continue to be implemented during this project.
- The Navy prepared an MEC safety video to be presented at public meetings and within the public schools.
- MEC Safety Awareness fact sheets were prepared and distributed to the public.
- Public notices are published in the local newspaper when planned detonation activities occur.

SECTION 13

References

Magazine Siting Plan for Solid Waste Management Unit 4 (SWMU 4), SWMU 6, and Area of Concern J; Former Naval Ammunition Support Detachment; Vieques Island, Puerto Rico, 26 November 2001, Revision 1, 17 May 2002(Final); CH2M HILL.

Expanded Preliminary Assessment/Site Investigation, Phase II, Seven Sites; Former Naval Ammunition Support Detachment; Vieques Island, Puerto Rico, 28 August 2002 (Final); CH2M HILL.

Remedial Investigation/Feasibility Study Work Plan for Solid Waste Management Unit (SWMU) 6, SWMU 7, Area of Concern (AOC) h, and AOC J; Former Naval Ammunition Support Detachment;Vieques Islan, Puerto Rico, July 2003 (Final); CH2M HILL.

Munitions Response Site Identification and Notification Report, 9 August 2004; Electronically Transmitted from NAVFAC Atlantic to Naval Ordnance Safety & Security Activity.

DEPARTMENT OF THE NAVY
NAVAL ORDNANCE SAFETY & SECURITY ACTIVITY
FARRAGUT HALL BLDG D-323
23 STRAUSS AVENUE
INDIAN HEAD MD 20640-5555

8020
Ser N539/148
26 Jan 06

From: Commanding Officer, Naval Ordnance Safety and Security Activity
To: Commanding Officer, Naval Facilities Engineering Command, Atlantic

Subj: EXPLOSIVES SAFETY SUBMISSION (ESS) WAIVER REQUEST FOR MUNITIONS AND EXPLOSIVES OF CONCERN (MEC) AVOIDANCE AT SOLID WASTE MANAGEMENT UNIT (SWMU) 6 AT THE FORMER NAVAL AMMUNITION SUPPORT DETACHMENT (NASD) VIEQUES, PUERTO RICO

Ref: (a) NAVFAC ATLANTIC ltr of 18 Jan 06 (w/encl)
(b) NOSSAINST 8020.15, Military Munitions response Program Oversight dtd 8 Mar 04
(c) NAVSEA OP 5, Revision 7

1. The Naval Ordnance Safety and Security Activity (NOSSA) reviewed the reference (a) request to waive the Explosives Safety Submission (ESS) for MEC avoidance at SWMU 6 at the former NASD Vieques, Puerto Rico. Based on the information presented in reference (a), and on the ESS criteria in references (b) and (c), NOSSA waives the requirement.

2. NOSSA understands that your operational risk/hazard assessment of the proposed Site Inspection concluded the probability of encountering Munitions and Explosives of Concern (MEC) is negligible. NOSSA further understands that you will have an Unexploded Ordnance (UXO) contractor on an immediate standby status as a conservative measure, although not required for a negligible risk/hazard.

3. Personnel are to avoid intentional physical contact with any MEC. Use anomaly avoidance techniques (as defined in reference (b)) at all times, including when you install grid corner stakes and collect soil and sediment samples. If you should discover MEC, however, take appropriate action and report the find to NOSSA using Enclosure (1) of reference (b).

Subj: EXPLOSIVES SAFETY SUBMISSION (ESS) WAIVER REQUEST FOR MUNITIONS AND EXPLOSIVES OF CONCERN (MEC) AVOIDANCE AT SOLID WASTE MANAGEMENT UNIT (SWMU) 6 AT THE FORMER NAVAL AMMUNITION SUPPORT DETACHMENT (NASD) VIEQUES, PUERTO RICO

4. The NOSSA point of contact for this ESS waiver is Mr. Douglas Murray, who can be contacted at DSN 354-4450 or commercial at 301-744-4450.

Pamela G. Clements

PAMELA G. CLEMENTS
By direction

Copy to:
CNO (N411; N45C)
COMNAVFACENGCOM (EN)
NOSSA ESSOLANT (N5L)

DEPARTMENT OF THE NAVY
NAVAL ORDNANCE SAFETY & SECURITY ACTIVITY
FARRAGUT HALL BLDG D-323
23 STRAUSS AVENUE
INDIAN HEAD MD 20640-5555

8020
Ser N539/149
26 Jan 06

From: Commanding Officer, Naval Ordnance Safety and Security Activity
To: Commanding Officer, Naval Facilities Engineering Command, Atlantic

Subj: EXPLOSIVES SAFETY SUBMISSION (ESS) WAIVER REQUEST FOR MUNITIONS AND EXPLOSIVES OF CONCERN (MEC) AVOIDANCE AT AREA OF CONCERN (AOC) "J" AT THE FORMER NAVAL AMMUNITION SUPPORT DETACHMENT (NASD) VIEQUES, PUERTO RICO

Ref: (a) NAVFAC ATLANTIC ltr of 18 Jan 06 (w/encl)
(b) NOSSAINST 8020.15, Military Munitions response Program Oversight dtd 8 Mar 04
(c) NAVSEA OP 5, Revision 7

1. The Naval Ordnance Safety and Security Activity (NOSSA) reviewed the reference (a) request to waive the Explosives Safety Submission (ESS) for MEC avoidance at AOC "J" at the former NASD Vieques, Puerto Rico. Based on the information presented in reference (a), and on the ESS criteria in references (b) and (c), NOSSA waives the requirement.

2. NOSSA understands that your operational risk/hazard assessment of the proposed Site Inspection concluded the probability of encountering Munitions and Explosives of Concern (MEC) is negligible. NOSSA further understands that you will have an Unexploded Ordnance (UXO) contractor on an immediate standby status as a conservative measure, although not required for a negligible risk/hazard.

3. Personnel are to avoid intentional physical contact with any MEC. Use anomaly avoidance techniques (as defined in reference (b)) at all times, including when you install grid corner stakes and collect soil and sediment samples. If you should discover MEC, however, take appropriate action and report the find to NOSSA using Enclosure (1) of reference (b).

Subj: EXPLOSIVES SAFETY SUBMISSION (ESS) WAIVER REQUEST FOR MUNITIONS AND EXPLOSIVES OF CONCERN (MEC) AVOIDANCE AT SOLID WASTE MANAGEMENT UNIT (SWMU) 6 AT THE FORMER NAVAL AMMUNITION SUPPORT DETACHMENT (NASD) VIEQUES, PUERTO RICO

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26 Jan 06

From: Commanding Officer, Naval Ordnance Safety and Security Activity
To: Commanding Officer, Naval Facilities Engineering Command, Atlantic

Subj: EXPLOSIVES SAFETY SUBMISSION (ESS) WAIVER REQUEST FOR MUNITIONS AND EXPLOSIVES OF CONCERN (MEC) AVOIDANCE AT SOLID WASTE MANAGEMENT UNIT (SWMU) 7 AT THE FORMER NAVAL AMMUNITION SUPPORT DETACHMENT (NASD) VIEQUES, PUERTO RICO

Ref: (a) NAVFAC Atlantic ltr of 18 Jan 06 (w/encl)
(b) NOSSAINST 8020.15, Military Munitions response Program Oversight dtd 8 Mar 04
(c) NAVSEA OP 5, Revision 7

1. The Naval Ordnance Safety and Security Activity (NOSSA) reviewed the reference (a) request to waive the Explosives Safety Submission (ESS) for MEC avoidance at SWMU 7 at the former NASD Vieques, Puerto Rico. Based on the information presented in reference (a), and on the ESS criteria in references (b) and (c), NOSSA waives the requirement.

2. NOSSA understands that your operational risk/hazard assessment of the proposed Site Inspection concluded the probability of encountering Munitions and Explosives of Concern (MEC) is negligible. NOSSA further understands that you will have an Unexploded Ordnance (UXO) contractor on an immediate standby status as a conservative measure, although not required for a negligible risk/hazard.

3. Personnel are to avoid intentional physical contact with any MEC. Use anomaly avoidance techniques (as defined in reference (b)) at all times, including when you install grid corner stakes and collect soil and sediment samples. If you should discover MEC, however, take appropriate action and report the find to NOSSA using Enclosure (1) of reference (b).

Subj: EXPLOSIVES SAFETY SUBMISSION (ESS) WAIVER REQUEST FOR MUNITIONS AND EXPLOSIVES OF CONCERN (MEC) AVOIDANCE AT SOLID WASTE MANAGEMENT UNIT (SWMU) 7 AT THE FORMER NAVAL AMMUNITION SUPPORT DETACHMENT (NASD) VIEQUES, PUERTO RICO

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HABITAT CHARACTERIZATION AT AREAS OF CONCERN (AOC) AOC-R, PI-4, PI-7, PAOC-J, K, and S FOR THE FORMER VIEQUES NAVAL TRAINING RANGE (VNTR) AND NAVAL AMMUNITION SUPPORT DETACHMENT (NASD), VIEQUES ISLAND, PUERTO RICO

FINAL REPORT

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ACRONYMS AND ABBREVIATIONS

°	degree
%	Percent
ac	Acres
AOC	Areas of Concern
EMA	Eastern Maneuver Area
F	Farenheit
Ft	Feet/Foot
LIA	Live Impact Area
mi	Miles
NASD	Naval Ammunition Support Detachment
NSRR	Naval Site Roosevelt Roads
PI	Photo Identified
U.S.	United States
USFWS	United States Fish and Wildlife Service
VNTR	Vieques Naval Training Range

1.0 INTRODUCTION

Biological field assessments were conducted at Areas of Concern (AOC), Potential AOC, and photo identified (PI) as part of a Comprehensive Long-Term Environmental Action Navy (CLEAN) III Contract, Contract Task Order 007 and 39 Vieques Island, Puerto Rico. The purpose of this evaluation was to identify habitat or potential habitat that currently is or could be used by the Least Tern, Roseate Tern, or any other listed endangered, threatened, critical or sensitive avian species, within the specified areas of concern. Additionally, a survey of the flora was conducted to determine if any State or Federally listed plant species were present

2.0 SITE LOCATION

Puerto Rico is located on the Antillean Island Arc separating the Caribbean Sea from the Atlantic Ocean. The island of Vieques is located approximately 6 miles (mi) southeast of Puerto Rico (**Figure 1**). It is approximately 21 mi long (east to west) and 4.5 mi wide (north to south), encompassing a total of 32,986 acres (ac). There are two towns on the island, Esperanza on the south side and Isabel Segunda on the north side.

The Navy administered approximately 22,437 ac on the west and east ends of the island. The former Naval Ammunition Support Detachment (NASD) was located on the western side of Vieques and encompasses 8,265 ac. The Eastern Maneuver Area (EMA) was composed of approximately 11,000 acres. This report covers AOC-R (former construction staging area) located within the former NASD and five sites (PI-4, PI-7, PAOC-J, K, and S) on eastern Vieques in the former Vieques Naval Training Range (VNTR) (**Figure 2**).

3.0 METHODS

To support this evaluation a field survey was conducted at each site in order to determine the presence of plant and animal species, and to determine whether preferred habitat was present for any federally endangered or threatened plant and/or animal species.

To support these assessments, a habitat characterization was conducted at each of the six sites. The objectives of the habitat characterization are listed below:

1. Identify terrestrial (upland), wetland (palustrine and estuarine), and marine habitat units, including subunits within and contiguous to each site;
2. Identify the dominant and subdominant plant species within each site habitat unit and contiguous habitat units and, where applicable, identify any vegetative gradients;
3. Identify wildlife species residing within or utilizing each site's habitat unit and contiguous habitat units, as well as species relative abundance;
4. Identify any critical habitat for federally designated threatened and endangered species within and contiguous to each site; and
5. Determine the known occurrences of federally designated threatened or endangered species, as well as the identification of habitat that may potentially support federally designated threatened and endangered species within and contiguous to each site.

Prior to field reconnaissance, vegetation communities were characterized into broad community types based on color signatures from true-color aerial photographs (November 4, 1999: scale, 1 inch equal 500 feet [ft]). Before conducting the fieldwork, a literature search was conducted for each federally protected species. During the September 19-20, 2005 surveys, 3 biologists (Mr. Carlos Laboy – animals and plants, Dr. Alberto E. Areces-Mallea – plants, and Dr. Dan Wilkinson – plants and animals) walked throughout each site and identified any federally protected species seen and evaluated the site for the presence or absence of preferred habitat for the species. Community types were verified in the field by walking the perimeter and the interior of the sites. Most plant species were identified in the field; however, some specimens were collected for identification using taxonomic references (Acevedo-Rodriguez 1996; Liogier 1982, 1985, 1988, 1994, 1995, 1997; Little and Wadsworth 1964; Little et al. 1964).

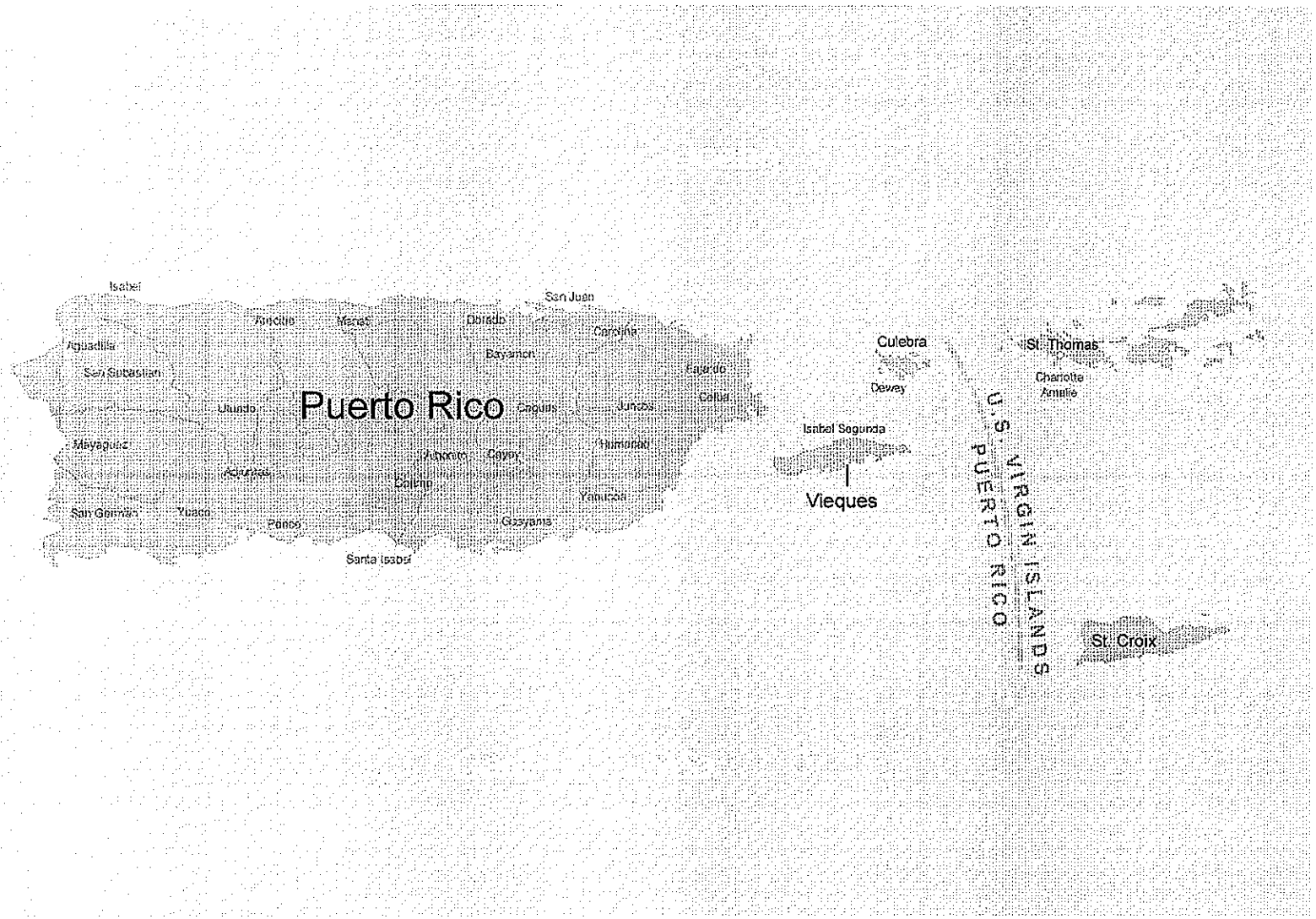

Figure 1. General Location of Vieques Island, Puerto Rico.

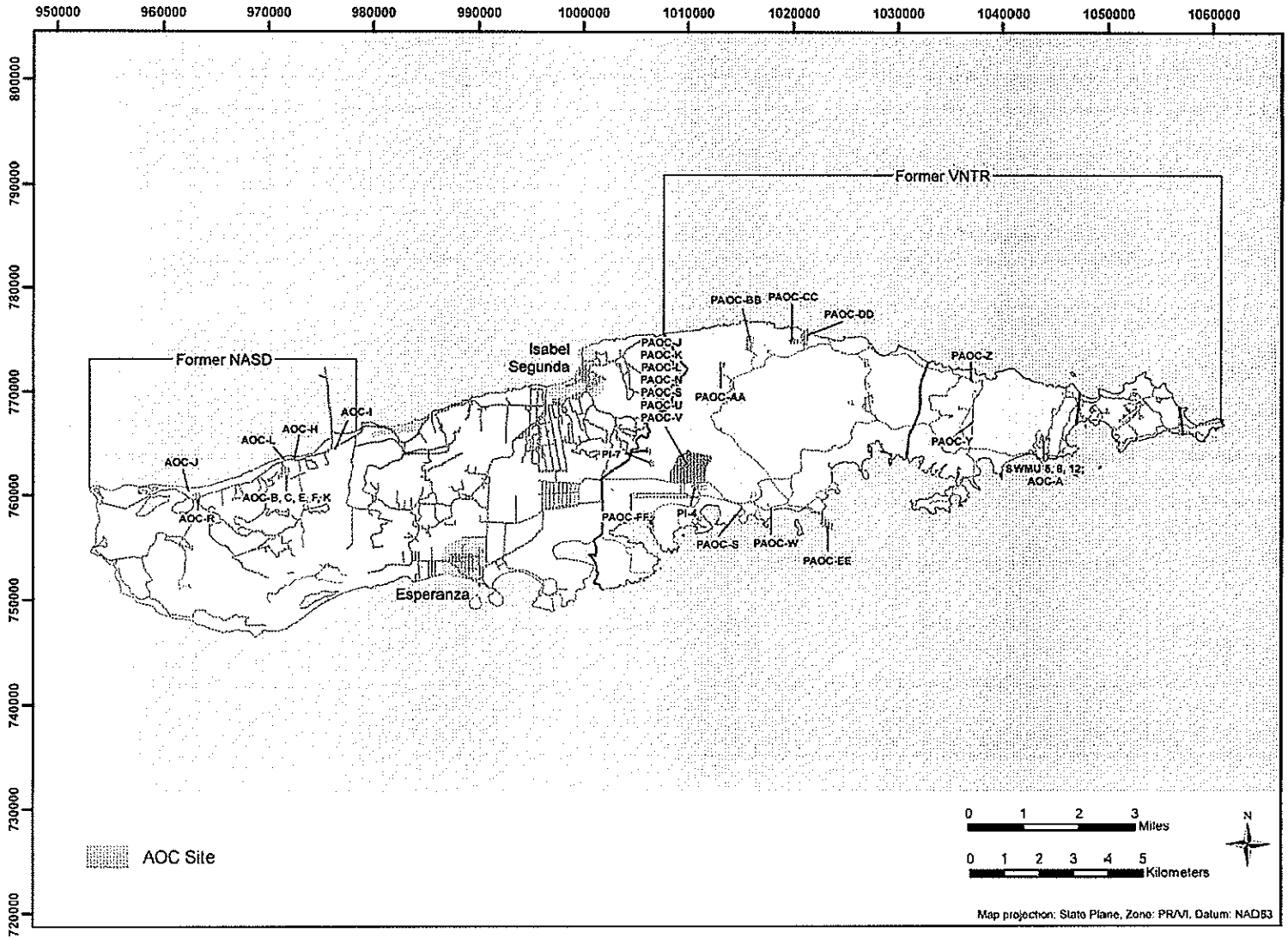

Figure 2. Location of the Areas of Concern (AOC) within the Former Naval Ammunition Support Detachment (NASD) and Former Vieques Naval Training Range (VNTR).

Wildlife species such as birds, mammals, and lizards that were found residing within or utilizing each site's habitat were identified during the field surveys. Wildlife habitat was also identified. A wildlife biologist characterized the habitats and determined the types of wildlife that could potentially inhabit the plant communities or sites. Any wildlife species observed, were identified in the field with the aid of 8 x 40 binoculars and reference guides (Raffaele 1989; Raffaele et al. 1998).

Several terrestrial and marine species that occur on or around Vieques are listed as protected by the United States (U.S.) Fish and Wildlife Service (USFWS). Sixteen federally listed species are known to occur or have the potential to occur within the former NASD and former VNTR (**Table 1**).

Table 1
Federally Listed Species Occurring or Potentially Occurring at the Former NASD and former VNTR

***Scientific Name* (Common Name)**	**Federal Status**
Plants	
Chaemacrista glandulosa var. *mirabilis* (Herb)	Endangered
Stahlia monosperma (Cobana negra)	Threatened
Calyptranthes thomasiana (Tree)	Endangered
Eugenia woodburyana (Evergreen tree)	Endangered
Goetzea elegans (Beautiful goetzea)	Endangered
Reptiles and Amphibians	
Chelonia mydas (Green sea turtle)	Threatened
Dermochelys coriacea (Leatherback sea turtle)	Endangered
Caretta caretta (Loggerhead sea turtle)	Threatened
Eretmochelys imbricata (Hawksbill sea turtle)	Endangered
Birds	
Falco peregrinus tundrius (Arctic peregrine falcon)	Threatened
Pelecanus occidentalis occidentalis (Brown pelican)	Endangered
Sterna dougalli dougalli (Roseate tern)	Endangered
Mammals	
Physeter macrocephalus (Sperm whale)	Endangered
Balaenoptera physalus (Fin whale)	Endangered
Megaptera novaeangliae (Humpback whale)	Endangered
Trichechas manatus (West Indian manatee)	Endangered

Source: Geo-Marine, Inc. 2001

In addition to identification of wildlife in the field, existing literature sources were used to identify any additional species that may have occurred on the sites but were not observed. Most of the wildlife that occurs in the area are bird and lizard species.

This report includes figures of each site showing the habitat units and subunits, landscaped areas, roadways, and structures where applicable. For each site, a list of plant species observed within the habitat unit is given, as well as a list of observed wildlife species. The location of known federally designated threatened and endangered species is given. Where applicable, a descriptive comparison of wildlife utilization between the site habitat and reference site habitat is given with a generalized food web applicable to each habitat unit.

4.0 RESULTS AND DISCUSSION

4.1 NATURAL HABITAT DESCRIPTION

Mean annual precipitation on Vieques is 45 inches, with the wet season extending from May to November and the dry season from December to April. Relative humidity in Vieques averages about 75%. Mean annual temperature is about 79.8°F with a record low of 60°F and a high of 98°F. The western part of the island is wetter than the eastern portion. Vegetation on the western part, where a portion of this survey took place, is mesic in nature.

Vieques lies within the subtropical dry forest life zone, covering about 66% of the island and the subtropical moist forest life zone at the higher elevations (Ewel and Whitmore 1973). Nine vegetation types have been identified on Vieques: (1) sandy beach; (2) beach scrub; (3) dry evergreen beach woodland; (4) mixed dry evergreen and deciduous woodland of the rocky coastal slopes; (5) ucar forest; (6) seasonal forests of high moisture areas such as ravines and drainage areas; (7) moist deciduous formation of the inner hills and slopes; (8) thorn scrub; and (9) mangroves. The vast majority of Navy land at Vieques is undeveloped and supports a variety of habitat types with thorn scrub communities the most common and interspersed with forested ravines and other upland forest types. Mangrove swamps, lagoons, coconut flats, and salt/sand flats occur along coastal areas.

The original dominant vegetation where the surveys took place was subtropical dry forest with subunits of thorn scrub, sandy beach, and mixed forests along the coast referred to as "coastal forest." Small subunits of mixed mangrove and seasonal forests were present near lagoons. Today all that remains are areas of highly modified disturbed vegetation at most of the sites that included former buildings, foundations, roadways, parking areas, trails, grassy areas, and quarries.

4.2 PROTECTED SPECIES

Cobana negra (*Stahlia monosperma*), a federally threatened tree, has been found between the boundary of black mangrove (*Avicennia germinans*) communities, salt flats, and the upland communities in the NASD and in the EMA. This species is also known to occur in coastal forests of southeastern Puerto Rico (Little and Wadsworth 1964). Based on recent surveys conducted within the NASD by Geo-Marine, Inc., there were only two locations where cobana negra is found (Wilkinson and Cubina 2000); neither of the two locations are located within the sites evaluated as part of this report. The preferred habitat for cobana negra was not thought to be present at the PI/PAOC sites discussed in this report.

The threatened Arctic peregrine falcon (*Falco peregrinus tundrius*) has been observed at former Naval Site Roosevelt Roads (NSRR) (GMI 1998). This species utilizes open grassland areas for potential feeding areas. The brown pelican (*Pelecanus occidentalis occidentalis*) and the roseate tern (*Sterna dougalli dougallii*) are not likely to utilize this terrestrial site but may be seen nearby due to the proximity of nearby marine habitat.

4.3 AOC-R (FORMER CONSTRUCTION STAGING AREA)

4.3.1 Site Description

AOC-R, the Former Construction Staging Area, located north and south of Highway 200 near a pump station was approximately 12 ac in size (**Figure 3**). There were two dirt trails leading to the site; one ran along the western edge of the site and one ran diagonally along the eastern edge of the site. Several habitats and man-made structure components were present. The southern boundary of the site was adjacent to a grassy mowed area along Highway 200. A pump station was inset about midway along the road. Behind the grassy area was a dense, scrubby, brushy area. A large concrete pad was located north of this scrub and extended the full width of the site. The pad was completely vegetated in the 1999 aerial photographs but in January 2001 during a previous survey the pad was sparsely covered with vines and weeds. Currently the pad is densely covered with vegetation. The vegetation surrounding the concrete pad had been cleared in the past. The western dirt path led past the concrete pad to a northern area that was separated from the pad by dense thorn scrub and coastal forest. The site is by secondary plant communities consisting of dense growth of vines, shubs, and herbaceous plants.

4.3.2 Vegetation Community Description

Although the concrete pad area had been cleared, a large number of weeds and vines were observed growing on top of the concrete pad and its surroundings The concrete pad was dominated by woody vegetation and vines. The original vegetation community at AOC-R from the road to near the coast is practically nonexistent. The area is covered by secondary communities consisting of an open mixture of

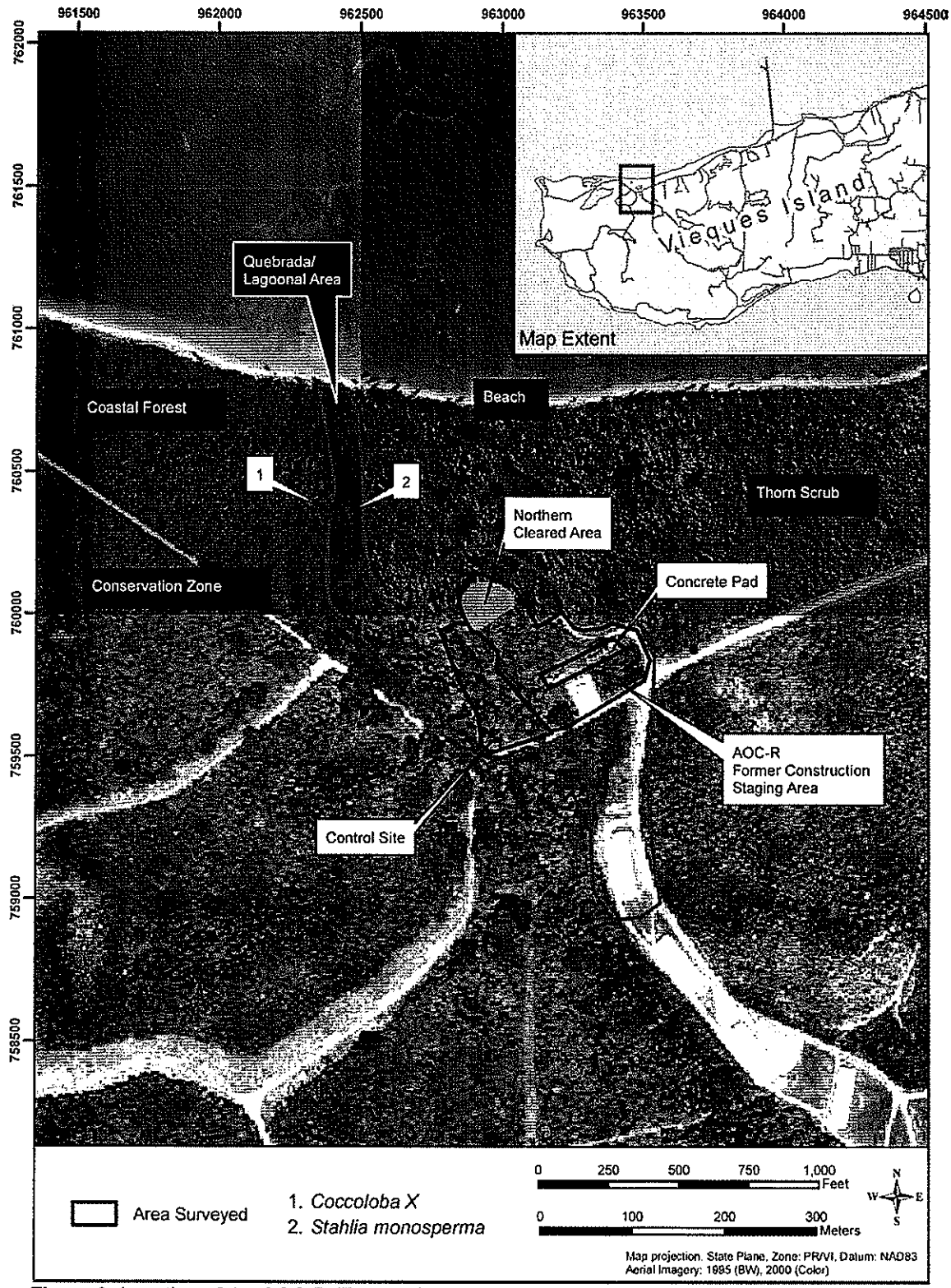

Figure 3. Location of the AOC-R (Former Construction Staging Area) and the Ephemeral Stream in the Former NASD.

native and invasive trees with an impressive overgrowth of vines and opportunistic shrubs and herbaceous plants. There are also open patches, grasses patches, densely covered with weedy plants. Much less disturbed and thus, much more interesting is the creek or ephemeral stream that borders the site on the west side. There are several native species that occur along this riparian area. On the east bank there is one large individual of a hybrid *Coccoloba* species (N 18 07 05.6 and W 65 33 04.4) that has been recorded recently on the east end of the island in the former Live Impact Area (LIA). On the west bank near the inland edge of the mangrove community we discovered two individuals of *Stahlia monosperma* (N18 07 05.7 and W 65 33 05.1) There were 140 plant species recored in this area, primarily weedy herbaceous invasive species (**Table 2**).

Table 2
List of All Plants Recorded in the AOC-R and AOC-J (in part) of the Former NASD, Vieques, 19 September 2005 (140 Species Total)

Family	Species	Common Name(s)
MIMOSACEAE	*Acacia farnesiana* (L.) Willd.	Aroma; Casha
MIMOSACEAE	*Acacia retusa* (Jacq.) Howard	Acacia zarza
MIMOSACEAE	*Acacia tortuosa* (L.) Willd.	Casia; Twisted acacia
FABACEAE	*Andira inermis* (W. Wr.) DC.	Moca; Angelin tree
POLYGONACEAE	*Antigonon leptopus* Hook & Arn.	Bellísima; Love chain
CAESALPINACEAE	*Bauhinia monandra* Kurz	Mariposa; Butterfly Bauhinia
ASTERACEAE	*Bidens alba* (L.) DC. var. (CH. Schulz) Melchert	Margarita
NYCTAGINACEAE	*Boerhavia diffusa* L.	Mata pavo; Hog-weed
POACEAE	*Bothriochloa pertusa* (L.) A. Camus	Huracán; Hurricane grass
BORAGINACEAE	*Bourreria succulenta* Jacq.	Palo de vaca
POACEAE	*Brachiaria fasciculata* (Sw.) S. T. Blake	----
POACEAE	*Brachiaria* sp.	----
COMBRETACEAE	*Bucida buceras* L.	Úcar
BURSERACEAE	*Bursera simaruba* (L.) Sarg.	Almácigo
BRASSICACEAE	*Cakile lanceolata* (Willd.) O. E. Schulz	Mostacilla; Sea-rocket
COMMELINACEAE	*Callisia repens* (Jacq.) L.	Cohitre enano
FABACEAE	*Canavalia rosea* (Sw.) DC.	Canavalia; Bay-bean
CAPPARACEAE	*Capparis hastata* Jacq.	Burro; Broad-leaved caper
SOLANACEAE	*Capsicum annuum* L. var. *glabriusculum* (Dunal) H. & P.	Ají picante; Bird pepper
CARICACEAE	*Carica papaya* L.	Lechosa, Papaya; Papaw
FLACOURTIACEAE	*Casearia guianensis* (Aubl.) Urban	Cafeíllo; Wild coffee
MORACEAE	*Cecropia schreberiana* Miq.	Yagrumo hembra; Trumpet tree
ULMACEAE	*Celtis trinervia* Lam.	Almez, Ruiseñor
POACEAE	*Cenchrus incertus* M. A. Curtis	Abrojo de dunas; Sand bur grass
FABACEAE	*Centrosema pubescens* Benth.	Flor de conchitas; Butterfly pea
FABACEAE	*Centrosema virginianum* (L.) Benth.	Conchita de Virginia
EUPHORBIACEAE	*Chamaesyce hirta* (L.) Millsp.	Lechecillo
EUPHORBIACEAE	*Chamaesyce messembryanthemifolia* (Jacq.) Dugand	Lecherito de costa
POACEAE	*Chloris petraea* Sw.	Yerba de dedo; Finger grass
POACEAE	*Chloris radiata* (L.) Sw.	Grama de costa; Plush-grass
MENISPERMACEAE	*Cissampelos pareira* L.	Oreja de ratón
VITACEAE	*Cissus verticillata* (L.) Nicolson & Jarvis	Bejuco de Caro; Pinakoop
VERBENACEAE	*Citharexylum fruticosum* L.	Palo de guitarra; Old woman bitter

Table 2 (Continued)
List of All Plants Recorded in the AOC-R and AOC-J (in part) of the former NASD, Vieques, 19 September 2005 (140 Species Total)

Family	Species	Common Name(s)
RUTACEAE	*Citrus aurantium* L.	Naranja, Naranja agria; Sour orange
POLYGONACEAE	*Coccoloba uvifera* (L.) L.	Uva de playa; Sea grape
POLYGONACEAE	*Coccoloba venosa* L.	Calambreña; Chiguery grape
POLYGONACEAE	*Coccoloba* sp. (hybrid?)	---
ARECACEAE	*Coccothrinax alta* (O. F. Cook) Becc.	Palma plateada; Silver palm
ARECACEAE	*Cocos nucifera* L.	Cocotero; Coconut palm
COMMELINACEAE	*Commelina diffusa* Burm. f.	Cohitre; Blue day flower
BORAGINACEAE	*Cordia collococca* L.	Capá rojo, Ateje; Red manjack
BORAGINACEAE	*Cordia dentata* Poir.	Capá blanco; White manjack
BORAGINACEAE	*Cordia polycephala* (Lam.) I. M. Johnst.	Basora prieta; Black sage
EUPHORBIACEAE	*Croton astroites* Dryand.	Maná
ASCLEPIDACEAE	*Cryptostegia grandiflora* R. Brown	Bejuco de goma; Rubber vine
CYPERACEAE	*Cyperus alternifolius* L.	Paragüita; Umbrella sage
CYPERACEAE	*Cyperus rotundus* L.	Coquí; Nut grass
FABACEAE	*Dalbergia ecastaphyllum* (L.) Taub.	Maraimaray
EUPHORBIACEAE	*Dalechampia scandens* L.	---
CAESALPINACEAE	*Delonix regia* (Bojer in Hook.) Raf.	Flamboyán; Flamboyant
POACEAE	*Digitaria insularis* (L) Mez	Zorra; Silky grass
DIOSCOREACEAE	*Dioscorea alata* L.	Ñame de agua; White yam
BIGNONIACEAE	*Distictis lactiflora* (Vahl) DC.	Liana fragante
ERYTHROXYLACEAE	*Erythroxylum brevipes* DC.	Brisselet
MYRTACEAE	*Eugenia biflora* (L.) DC.	Hoja menuda; Black rod-wood
EUPHORBIACEAE	*Euphorbia heterophylla* L.	Acerca amor
MORACEAE	*Ficus citrifolia* P. Miller	Jagüeillo; Boislaglu
FABACEAE	*Galactia striata* (Jacq.) Urban	---
RHAMNACEAE	*Gouania lupuloides* (L.) Urban	Bejuco de indio; Soap stick
NYCTAGINACEAE	*Guapira fragans* (Dum.-Cours.) Little	Corcho; Black mampoo
STERCULIACEAE	*Guazuma ulmifolia* Lam.	Guácima; Bastard cedar
AMARANTHACEAE	*Hymenocallis caribaea* (L. emend Gawl.) Herb.	Lirio blanco; Spider lily
LAMIACEAE	*Hyptis capitata* Jacq.	Botoncillo negro; Wild hops
MIMOSACEAE	*Inga laurina* (Sw.) Willd.	Guamá; Pomshock
CONVOLVULACEAE	*Ipomoea alba* L.	Bejuco de vaca; Moon-vine
CONVOLVULACEAE	*Ipomoea hederifolia* L.	Cambustera
CONVOLVULACEAE	*Ipomoea nil* (L.) Roth.	Bejuco de gloria
CONVOLVULACEAE	*Ipomoea tiliacea* (Willd.) Choisy in DC.	Bejuco blanco; Goat foot
CONVOLVULACEAE	*Ipomoea violacea* L.	Bejuco de luna; COSAT moon-vine
OLEACEAE	*Jasminum fluminense* Vell.	Jasmín oloroso
EUPHORBIACEAE	*Jatropha gossypiifolia* L.	Higuereta cimarrona
RHAMNACEAE	*Krugiodendron ferreum* (Vahl) Urban	Bariaco; Black iron-wood
COMBRETACEAE	*Laguncularia racemosa* (L.) Gaertn.	Mangle blanco; Mangrove
POACEAE	*Lasiacis ligulata* Hitch. & Chase	Millo trepador
MIMOSACEAE	*Leucaena leucocephala* (Lam.) De Wit	Acacia pálida; Wild tamarind

Table 2 (Continued)
List of All Plants Recorded in the AOC-R and AOC-J (in part) of the former NASD, Vieques, 19 September 2005 (140 Species Total)

Family	Species	Common Name(s)
VERBENACEAE	*Lippia nodiflora (L.) Michx.*	Cidrón, Yerba de sapo; Cape-wood
FABACEAE	*Lonchocarpus heptaphyllus* (Poir.) A. DC.	Forteventura, Palo hediondo
ONAGRACEAE	*Ludwigia octovalvis* (Jacq.) Raven	Cangá; Primrose willow
BIGNONIACEAE	*Macfadyena unguis-cati* (L.) A. Gentry	Bejuco de gato; Cat´s claw
FABACEAE	*Macroptilium lathyroides* (L.) Urban	Habichuela parada; Wild bush bean
MALVACEAE	*Malachra alceifolia* Jacq.	Malva de caballo; Bastard okra
ANACARDIACEAE	*Mangifera indica* L.	Mango; Mango tree
SAPINDACEAE	*Melicoccus bijugatus* Jacq.	Quenepa; Ginep
STERCULIACEAE	*Melochia nodiflora* Sw.	Bretónica prieta
STERCULIACEAE	*Melochia tomentosa* L.	Bretónica afelpada
CONVOLVULACEAE	*Merremia quinquefolia* (L) Hall. f.	Batatilla blanca
MIMOSACEAE	*Mimosa pudica* L.	Morivivi; Sensitive plant
CUCURBITACEAE	*Momordica charantia* L.	Cundeamor; Wild balsam apple
NYCTAGINACEAE	*Neea buxifolia* (Hook.f.) Heimerl	Nea, Nia; Neea
ORCHIDIACEAE	*Oeceoclades maculata* (Lindley) Lindley	Lengua africana
POACEAE	*Panicum laxum* Sw.	Malojillo de monte
POACEAE	*Panicum maximum* Jacq.	Yerba de guinea; Guinea grass
POACEAE	*Paspalidium geminatum* (Forssk.) Stapf.	Yerba de agua; Water grass
POACEAE	*Paspalum distichum* L.	—
POACEAE	*Paspalum millegrana* Schrad.	Cortadora, Paja brava
POACEAE	*Paspalum virgatum* L.	Cortadero, Matojo blanco
PASSIFLORACEAE	*Passiflora foetida* L.	Tagua-Tagua
PASSIFLORACEAE	*Passiflora suberosa* L.	Flor de pasion
SAPINDACEAE	*Paullinia pinnata* L.	Bejuco de paloma
PHYTOLACACEAE	*Petiveria alliacea* L.	Anamú; Garlic weed
EUPHORBIACEAE	*Phyllanthus niruri* L.	Quinino; Gale of the wind
PIPERACEAE	*Piper aduncum* L.	Higuillo de hoja menuda
PIPERACEAE	*Piper amalago* L.	Higuillo de limón; Shot-shot
NYCTAGINACEAE	*Pisonia aculeata* L.	Uña de gato; Prickly mampoo
MIMOSACEAE	*Prosopis juliflora* (Sw.) DC.	Mesquite
RUBIACEAE	*Psychotria nervosa* Sw.	Bálsamo, Palo moro
RUBIACEAE	*Randia aculeata* L.	Tintillo; Ink-berry
APOCYNACEAE	*Rauvolfia viridis* Willd. ex Roem. & Schultes	Cachimbo blanco; Bitter bush
FABACEAE	*Rhynchosia reticulata* (Sw.) DC.	Frijolillo de monte
PHYTOLACCACEAE	*Rivina humilis* L.	Carmín; Cat´s blood
ACANTHACEAE	*Ruellia tuberosa* L.	Saltaperico; Many-roots
MIMOSACEAE	*Samanea saman* (Willd.) Merrill	Samán; Rain tree
LILIACEAE	*Sansevieria hyacinthoides* (L.) Druce	Lengua de vaca; Sweet Sansevieria
SAPINDACEAE	*Serjania polyphylla* (L.) Radlk.	Bejuco de canastas; Basket wood
FABACEAE	*Sesbania sericea* (Willd.) Link	Papagayo
AIZOACEAE	*Sesuvium portulacastrum* (L.) L.	Verdolaga rosada; Sea purslane

Table 2 (Continued)
List of All Plants Recorded in the AOC-R and AOC-J (in part) of the former NASD, Vieques, 19 September 2005 (140 Species Total)

Family	Species	Common Name(s)
POACEAE	*Setaria geniculata* (Lam.) Beauv.	Arrocillo; Yellow fostail
MALVACEAE	*Sida rhombifolia L.*	Escoba colorada
POACEAE	*Spartina patens (Ait.) Muhl.*	Yerba de sal; Salt grass
RUBIACEAE	*Spermacoce sp.*	—
LOGANIACEAE	*Spigelia anthelmia* L.	Lombricera; Waterweed
ANACARDIACEAE	*Spondias mombin* L.	Jobo; Hogplum
POACEAE	*Sporobolus jacquemontii* Kunth	Pelo de burro
CAESALPINACEAE	*Stahlia monosperma* (Tul.) Urban	Cóbana negra, Polisandro
MALPIGHIACEAE	*Stigmaphyllon emarginatum* (Cav.) A. Juss.	Bejuco de San Pedro
CAESALPINACEAE	*Tamarindus indica* L.	Tamarindo; Tamarind
MYRTACEAE	*Syzygium malaccensis* (L.) Merr. & Perry	Manzana malaya; Malay apple
COMBRETACEAE	*Terminalia catappa* L.	Almendra; Indian almond
THELYPTERIDACEAE	*Thelypteris tetragona* (Sw.) Small	Helecho
MALVACEAE	*Thespesia populnea* (L.) Soland ex Correa	Emajagüilla; Cork tree
BORAGINACEAE	*Tournefortia hirsutissima* L.	Nigua
EUPHORBIACEAE	*Tragia volubilis* L.	Pringamoza; Stinging vine
ULMACEAE	*Trema micranthum* (L.) Blume.	Guacimilla, Palo de cabra
MELIACEAE	*Trichilia hirta* L.	Retamo; Broomstick
PHYTOLACACEAE	*Trichostigma octandrum* (L.) H. Walt.	Bejuco de nasa
TILIACEAE	*Triumpheta semitriloba* Jacq.	Cadillo de perro
FABACEAE	*Vigna luteola* (Jacq.) Benth.	Frijol silvestre
RUTACEAE	*Zanthoxylum martinicense* (Lam.) DC.	Ayúa, Cenizo; Prickly ash
RUTACEAE	*Zanthoxylum monophyllum* (Lam.) P. Wilson	Carrubio, Espino rubial; Yellow prickle
RHAMNACEAE	*Ziziphus mauritiana* Lam.	Aprín; Indian jujube

Note: Following the conservation of the name *Leguminosae* for the family (*sensu latiore*) in the third edition of the International Code of Botanical Nomenclature, many authors treat the three families of *Order Fabales* as one single family (*Leguminosae*) with three subfamilies. However, we distinguish three different families (*Fabaceae*, *Caesalpinaceae* and *Mimosaceae*) as does A. Cronquist in *The Evolution and Classification of Flowering Plants* (1988). This treatment is strongly supported by molecular data and no doubt will prevail in the future.

4.3.3 Wildlife Description

Several wildlife species were observed utilizing the vegetated areas, concrete pad, edges of the cleared areas, and woody vegetation. The sides of the concrete pad provided shade, foraging areas, and hiding places for the common, garden and spotted anoles, which were abundant. A mongoose was seen crossing a trail in a cleared area. Wildlife observed at AOC-R, and adjacent habitat, presented in **Table 3**.

Habitat for birds included thorn scrub, coastal forest, and the adjacent riparian forest (vegetation along the stream). Many birds were seen along the edges of the cleared area around the concrete pad, along the trails, and were using the vegetation associated with the concrete pad for perches. A total of 16 species of birds were seen at the AOC-R site during this survey,and an additional 6 species during a previous survey. An additional six species were found along the riparian site. The most common birds at the site were gray kingbirds, bananaquits, and greater Antillean grackles. Bird species included those that would typically be found in coastal forest and thorn scrub habitat. A Brown pelican was observed flying along the coast north of the site.

Table 3
Wildlife Observed at AOC-R and in the Adjacent Quabrada

Common Name	Scientific Name	AOC-R	Adjacent Habitat
Mammals			
Mongoose	*Herpestes auropunctatus*	X	
Reptiles and Amphibians			
Anole (Common Lizard)	*Anolis cristatellus*	X	X
Grass Anole (Garden Lizard)	*Anolis pulchellus*	X	X
Anole (Spotted Lizard)	*Anolis stratulus*	X	X
Birds			
Green Mango	*Anthracothorax viridis*	X	
Great Egret	*Ardea alba*		X
Great Blue Heron	*Ardea herodias*		X
Green Heron	*Butorides virescens*	X	
Mangrove Cuckoo	*Coccyzus minor*		X
Bananaquit	*Coereba flaveola*	X	X
Scaly-naped pidgeon	*Columba squamosa*		x
Common ground Dove	*Columbina passerina*	X	X
Smooth-billed Ani	*Crotophaga ani*	X	X
Adelaide's Warbler	*Dendroica adelaidae*	X	X
Caribbean Elaenia	*Elaenia martinica*		X
Magnificent Frigatebird	*Fregata magnificens*		X
Puerto Rico Woodpecker	*Melanerpes portoricensis*	X	X
Puerto Rican Flycatcher	*Myiarchus antillarum*		X
Yellow-crowned Night-Heron	*Nyctanassa violacea*	X	
Antillean-crested Hummingbird	*Orthorhyncus cristatus*	X	
Osprey	*Pandion haliaetus*		X
Cave swallow	*Pterochelidon fulva*		X
Greater Antillean Grackle	*Quiscalus niger*	X	X
Puerto Rican Lizard Cuckoo	*Saurothera vieilloti*		X
Black-faced Grassquit	*Tiaris bicolor*	X	
Yellow-faced Grassquit	*Tiaris olivacea*		X
Loggerhead Kingbird	*Tyrannus caudifasciatus*	X	X
Gray Kingbird	*Tyrannus dominicensis*	X	X
Pin-tailed Whydah	*Vidua macroura*	X	
White-winged dove	*Zenaida asiatica*	X	X
Zenaida Dove	*Zenaida aurita*	X	X

4.3.4 Protected Species

The site was located within thorn scrub and mixed coastal forest habitat. There were no federally protected species or preferred habitats observed at this upland site. However, along the adjacent stream bank on the west side there were two individuals of *Stahlia monosperma*. On the west bank near the inland edge of the mangrove community we discovered two individuals of *Stahlia monosperma* (N18 07 05.7 and W 65 33 05.1) This is a new site for this species, althought there are two previously recorded sites along the north shore in the former NASD. On the east bank there is one large individual of a hybrid *Coccoloba* species (N 18 07 05.6 and W 65 33 04.4) that has been recorded recently on the east end of the island in the former LIA, although this plant is of scientific interest it has no protected status.

4.4 PAOC-S (FORMER POL PIPELINE)

4.4.1 Site Description

This site parallels existing roads from near the shoreline to where it exits near the east end of the airstrip (**Figure 4**). The terrain was modified to create a level site for placement of the pipeline. We could not determine where the original pipeline was located since it has been taken out of service. Much of the old pipe is scattered on other side of what appears to be the original pipeline route.

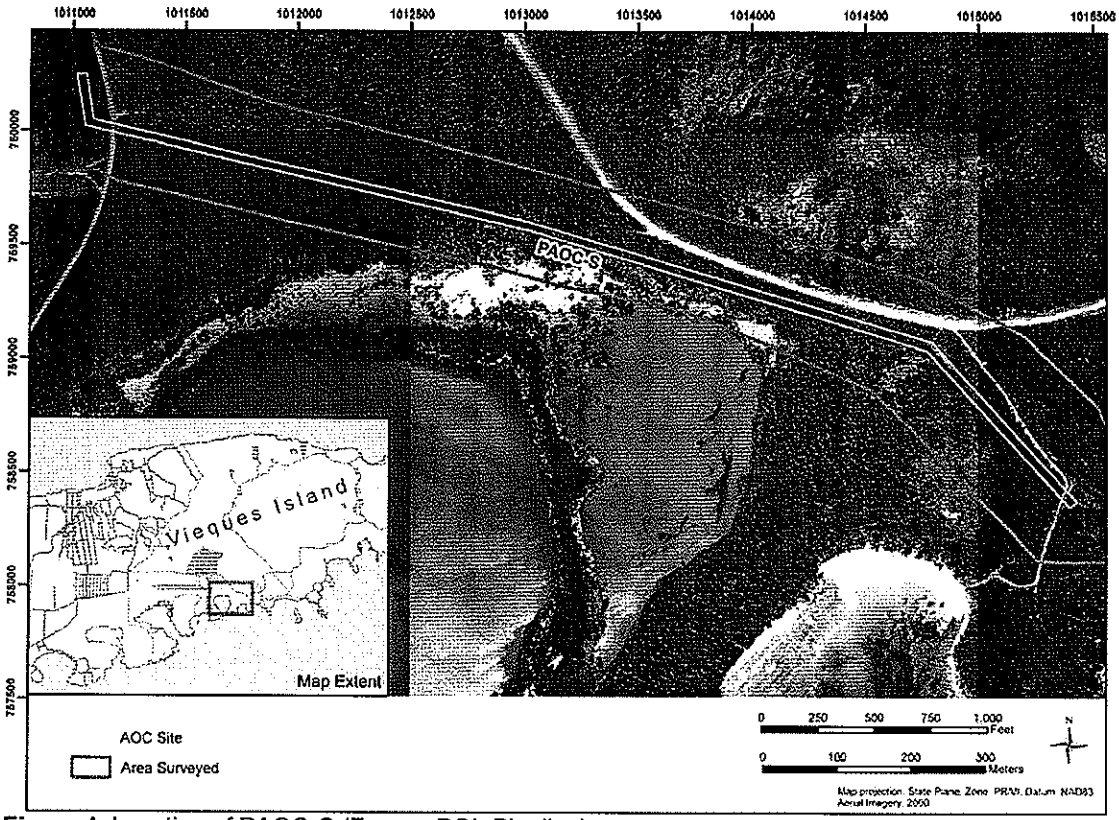

Figure 4. Location of PAOC-S (Former POL Pipeline).

4.4.2 Vegetation Community Description

The original plant community along the road was obviously cleared years ago when the pipeline was constructed. Currently, there are rocky mounds, leveled ground, built up areas, as well as other evidences of substrate disturbance (e.g., by bulldozer activity). The vegetation is basically a thorn scrub dominated by opportunistic acacias and *Prosopis*. There are many weedy invaders and at places there is a nearly continuous herbaceous layer. The pipeline borders a black mangrove forest (mangrove fringe of Bahia Tapon) for much of its length; however, previous wetlands examinations in this area have noted the pipeline and reported that no *Stahlia* trees were present in the available habitat at the edge of the tidal community. There were 116 plant species recorded along the pipeline route (**Table 4**). Most of which were invasive species that are characteristic of young secondary forest found on Vieques.

4.4.3 Wildlife Description

Several common wildlife species were observed utilizing the vegetated areas, pipes, edges of the cleared areas, and soil piles, and vegetation. Wildlife observed at PAOC-S, and adjacent habitat, is presented in **Table 5**.

Table 4
List of All Plants Recorded in the PAOC-S (Along the Pipeline) Area in the Former VNTR, 19 September 2005 (116 Species Total)

Family	Species	Common Name(s)
MALVACEAE	*Abutilon umbellatum* (L.) Sweet	Malvavisco cimarrón
MIMOSACEAE	*Acacia retusa* (Jacq.) Howard	Acacia zarza
MIMOSACEAE	*Acacia tortuosa* (L.) Willd.	Casia; Twisted acacia
AMARANTHACEAE	*Achyrantes aspera* L.	Rabo de gato; Man-better man
EUPHORBIACEAE	*Adelia ricinella* L.	Escambrón
RUTACEAE	*Amyris elemifera* L.	Cuabilla
AVICENNIACEAE	*Avicennia germinans* (L.) L.	Mangle negro; Black mangrove
BATIDACEAE	*Batis maritima* L.	Barilla; Salt wort
NYCTAGINACEAE	*Boerhavia coccinea* Mill.	Mata de pavo
POACEAE	*Bothriochloa pertusa* (L.) A. Camus	Huracán; Hurricane grass
BORAGINACEAE	*Bourreria succulenta* Jacq.	Palo de vaca
POACEAE	*Bouteloua americana* (L.f.) Scribn.	Yerba mesquite
POACEAE	*Brachiaria adspersa* (Trin.) Parodi	Millito; Brown top millet
POACEAE	*Brachiaria* sp.	----
BURSERACEAE	*Bursera simaruba* (L.) Sarg.	Almácigo
CAPPARACEAE	*Capparis flexuosa* (L.) L.	Palinguán; Caper tree
CAPPARACEAE	*Capparis indica* (L) Fawc. & Rendle	Sapo prieto; Linguam
SCROPHULARIACEAE	*Capraria biflora* L.	Té del país; Goat weed
SOLANACEAE	*Capsicum annuum* L. var. *glabriusculum* (Dunal) H. & P.	Ají picante; Bird pepper
SAPINDACEAE	*Cardiospermum microcarpum* HBK	Farolito; Ballon vine
FLACOURTIACEAE	*Casearia guianensis* (Aubl.) Urban	Cafeíllo; Wild coffee
POACEAE	*Cenchrus echinatus* L.	Abrojo; Bur grass
FABACEAE	*Centrosema pubescens* Benth.	Flor de conchitas; Butterfly pea
FABACEAE	*Centrosema virginianum* (L.) Benth.	Conchita de Virginia
CAESALPINACEAE	*Chamaecrista nictitans* (L.) Moench ssp. *Nictitans*	Moriviví bobo
EUPHORBIACEAE	*Chamaesyce hirta* (L.) Millsp.	Lechecillo
EUPHORBIACEAE	*Chamaesyce hyssopifolia* (L.) Small	Lechera
POACEAE	*Chloris inflata* Link	Horquetilla morada; Blue grass
POACEAE	*Chloris radiata* (L.) Sw.	Grama de costa; Plush-grass
VITACEAE	*Cissus trifoliata* L.	Caro costero
VITACEAE	*Cissus verticillata* (L.) Nicolson & Jarvis	Bejuco de Caro; Pinakoop
VERBENACEAE	*Citharexylum fruticosum* L.	Palo de guitarra; Old woman bitter
VERBENACEAE	*Clerodendron aculeatum* (L.) Schlecht.	Botón de oro; Crab prickly
FABACEAE	*Clitoria ternatea* L.	Bejuco de conchitas
ARECACEAE	*Coccothrinax alta* (O. F. Cook) Becc.	Palma plateada; Silver palm
RHAMNACEAE	*Colubrina arborescens* (Mill.) Sarg.	Abeyuelo, Sanguinaria; Greenheart
COMMELINACEAE	*Commelina diffusa* Burm. F.	Cohitre; Blue day flower
CONVOLVULACEAE	*Convolvulus nodiflorus* Desr.	Aguinaldo blanco
BORAGINACEAE	*Cordia collococca* L.	Capá rojo, Ateje; Red manjack
FABACEAE	*Crotalaria lotifolia* L.	Cascabelillo axilar
EUPHORBIACEAE	*Croton astroites* Dryand.	Maná

Table 4 (Continued)
List of All Plants Recorded in the PAOC-S (Along the Pipeline) Area in the Former VNTR, 19 September 2005 (116 Species Total)

Family	Species	Common Name(s)
EUPHORBIACEAE	*Croton betulinus* Vahl	—
EUPHORBIACEAE	*Croton discolor* Willd.	Lechecillo
EUPHORBIACEAE	*Croton rigidus* (Muell. Arg.) Britton	Guayacancillo; Yellow balsam
ASCLEPIDACEAE	*Cryptostegia grandiflora* R. Brown	Bejuco de goma; Rubber vine
EUPHORBIACEAE	*Dalechampia scandens* L.	—
POACEAE	*Digitaria bicornis* (L.) R. & S.	—
POACEAE	*Digitaria insularis* (L) Mez	Zorra; Silky grass
POACEAE	*Digitaria* sp.	—
BIGNONIACEAE	*Distictis lactiflora* (Vahl) DC.	Liana fragante
CELASTRACEAE	*Elaeodendrum xylocarpum* (Vent.) DC.	Coscorrón; Marble-tree
OLEACEAE	*Forestiera segregata* (Jacq.) Krug & Urban	Florida privet, Ink-bush
FABACEAE	*Galactia dubia* DC.	—
FABACEAE	*Galactia striata* (Jacq.) Urban	—
RHAMNACEAE	*Gouania lupuloides* (L.) Urban	Bejuco de indio; Soap stick
NYCTAGINACEAE	*Guapira fragans* (Dum.-Cours.) Little	Corcho; Black mampoo
BORAGINACEAE	*Heliotropium indicum* L.	Cotorrera; Indian heliotrope
CACTACEAE	*Hylocereus trigonus* (Haw.) Safford.	Pitajaya; Night blooming cereus
FABACEAE	*Indigofera suffruticosa* Miller	Añil; Índigo
CONVOLVULACEAE	*Ipomoea hederifolia* L.	Cambustera
CONVOLVULACEAE	*Ipomoea triloba* L.	Bejuquillo de puerco
CONVOLVULACEAE	*Jacquemontia havanensis* (Jacq.) Urban	Aguinaldo de costa
THEOPHRASTACEAE	*Jacquinia armillaris* Jacq.	Azúcares
EUPHORBIACEAE	*Jatropha gossypiifolia* L.	Higuereta cimarrona
RHAMNACEAE	*Krugiodendron ferreum* (Vahl) Urban	Bariaco; Black iron-wood
COMBRETACEAE	*Laguncularia racemosa* (L.) Gaertn.	Mangle blanco; Mangrove
VERBENACEAE	*Lantana camara* L.	Cariaquillo; Yellow sage
VERBENACEAE	*Lantana involucrata* L.	Santa María; Button sage
MIMOSACEAE	*Leucaena leucocephala* (Lam.) De Wit	Acacia pálida; Wild tamarind
VERBENACEAE	*Lippia nodiflora* (L.) Michx.	Cidrón, Yerba de sapo; Cape-wood
BIGNONIACEAE	*Macfadyena unguis-cati* (L.) A. Gentry	Bejuco de gato; Cat's claw
MALVACEAE	*Malvastrum* sp.	—
SAPINDACEAE	*Melicoccus bijugatus* Jacq.	Quenepa; Ginep
STERCULIACEAE	*Melochia nodiflora* Sw.	Bretónica prieta
STERCULIACEAE	*Melochia pyramidata* L.	Bretónica piramidal
STERCULIACEAE	*Melochia tomentosa* L.	Bretónica afelpada
CONVOLVULACEAE	*Merremia quinquefolia* (L) Hall. F.	Batatilla blanca
CACTACEAE	*Opuntia repens* Bello	Olaga; Suckers
POACEAE	*Panicum maximum* Jacq.	Yerba de guinea; Guinea grass
POACEAE	*Paspalum vaginatum* Sw.	—
PASSIFLORACEAE	*Passiflora suberosa* L.	Flor de pasion
FABACEAE	*Pictetia aculeata* (Vahl) Urban	Tachuelo; Fustic
CACTACEAE	*Pilosocereus royenii* (L.) Byles & Rowley	Dildo; Organ pipe
FABACEAE	*Piscidia carthagenensis* Jacq.	Ventura; Fish poison
NYCTAGINACEAE	*Pisonia subcordata* Sw.	Corcho blanco; Water mampoo

Table 4 (Continued)
List of All Plants Recorded In the PAOC-S (Along the Pipeline) Area in the Former VNTR, 19 September 2005 (116 Species Total)

Family	Species	Common Name(s)
MIMOSACEAE	*Pithecellobium unguis-cati* (L.) Mart.	Escambrón colorado; Black bean
MIMOSACEAE	*Prosopis juliflora* (Sw.) DC.	Mesquite
RUBIACEAE	*Randia aculeata* L.	Tintillo; Ink-berry
APOCYNACEAE	*Rauvolfia viridis* Willd. Ex Roem. & Schultes	Cachimbo blanco; Bitter bush
RHIZOPHORACEAE	*Rhizophora mangle* L.	Mangle rojo; Red mangrove
FABACEAE	*Rhynchosia minima* (L.) DC.	Frijolillo
FABACEAE	*Rhynchosia reticulata* (Sw.) DC.	Frijolillo de monte
PHYTOLACCACEAE	*Rivina humilis* L.	Carmín; Cat's blood
ACANTHACEAE	*Ruellia tuberosa* L.	Saltaperico; Many-roots
CELASTRACEAE	*Schaefferia frutescens* Jacq.	Cafeíllo; Florida boxwood
SAPINDACEAE	*Serjania polyphylla* (L.) Radlk.	Bejuco de canastas; Basket wood
MALVACEAE	*Sida cordifolia* L.	Escoba acorazonada
MALVACEAE	*Sida stipularis* Cav.	Escoba blanca; Wire weed
MALVACEAE	*Sidastrum multiflorum* (Jacq.) Fryxell	Sidastro
SAPOTACEAE	*Sideroxylon obovatum* Lam.	Alquitrán; Break hill
SOLANACEAE	*Solanum lanceifolium* Jacq.	Tomatillo de paloma
POACEAE	*Spartina patens* (Ait.) Muhl.	Yerba de sal; Salt grass
POACEAE	*Sporobolus jacquemontii* Kunth	Pelo de burro
VERBENACEAE	*Stachytarpheta jamaicensis* (L.) Vahl	Bretónica
MALPIGHIACEAE	*Stigmaphyllon emarginatum* (Cav.) A. Juss.	Bejuco de San Pedro
FABACEAE	*Stylosanthes hamata* (L.) Taubert in Verh.	Zarzabacoa enana; Pencil flower
FABACEAE	*Tephrosia cinerea* (L.) Pers.	Añil cenizo
BROMELIACEAE	*Tillandsia fasciculata* Sw.	Nido parásito; Bunch moss
BORAGINACEAE	*Tournefortia volubilis* L.	Nigua enredadera
EUPHORBIACEAE	*Tragia volubilis* L.	Pringamoza; Stinging vine
POACEAE	*Tragus berteronianus* Schult.	Hincadora; Prickle grass
PHYTOLACACEAE	*Trichostigma octandrum* (L.) H. Walt.	Bejuco de nasa
FABACEAE	*Vigna luteola* (Jacq.) Benth.	Frijol silvestre
MALVACEAE	*Wissadula periplocifolia* (L.) Presl	Pichona
RUTACEAE	*Zanthoxylum monophyllum* (Lam.) P. Wilson	Carrubio, Espino rubial; Yellow prickle
COMMELINACEAE	Indet.*	—

*In sterile condition (no flowers or fruits available for determination)

Note: Following the conservation of the name *Leguminosae* for the family (*sensu latiore*) in the third edition of the International Code of Botanical Nomenclature, many authors treat the three families of *Order Fabales* as one single family (*Leguminosae*) with three subfamilies. However, we distinguish three different families (*Fabaceae*, *Caesalpinaceae* and *Mimosaceae*) as does A. Cronquist in *The Evolution and Classification of Flowering Plants* (1988).

Table 5
List of All Animals Recorded in the PAOC-S (Along the Pipeline) Area in the Former VNTR, 19 September 2005

Common Name	Scientific Name	POCS
Mammals		
Mongoose	*Herpestes auropunctatus*	x
Reptiles and Amphibians		
Anole (Common Lizard)	*Anolis cristatellus*	x
Grass Anole (Garden Lizard)	*Anolis pulchellus*	x
Anole (Spotted Lizard)	*Anolis stratulus*	x
Birds		
Green Mango	*Anthracothorax viridis*	x
Cattle Egret	*Bubulcus ibis*	x
Green Heron	*Butorides virescens*	x
Killdeer	*Charadrius vociferus*	x
Mangrove Cuckoo	*Coccyzus minor*	x
Bananaquit	*Coereba flaveola*	x
Scaly-naped pidgeon	*Columba squamosa*	x
Adelaide's Warbler	*Dendroica adelaidae*	x
Caribbean Elaenia	*Elaenia martinica*	x
Scaly-Breasted Thrasher	*Margarops fuscus*	x
Puerto Rico Woodpecker	*Melanerpes portoricensis*	x
Northern Mockingbird	*Mimus polyglottos*	x
Yellow-crowned Night-Heron	*Nyctanassa violacea*	x
Greater Antillean Grackle	*Quiscalus niger*	x
Puerto Rican Lizard Cuckoo	*Saurothera vieilloti*	x
Gray Kingbird	*Tyrannus dominicensis*	x
White-winged dove	*Zenaida asiatica*	x

4.4.4 Protected Species

The site was located within thorn scrub and mangrove fringe forest habitat. There were no federally protected species observed at this site. The former pipeline ran near the edge of a mangrove community which appreared to have potential habitat for *Stahlia,* however a extensive search during this field effort and previous searches conducted in 2001 did not find any plants. The orginal construction of the pipeline would have potentially impacted *Stahlia* habitat.

4.5 PAOC J AND K (CAMP GARCIA SITES)

4.5.1 Site Description

This site is located at or adjacent to the former Camp Garcia Area (**Figure 5**). The area is maintained mowed grassland. Around the perimeter of Camp Garcia and parts of the PAOC-J and PAOC-K had been recently scraped of vegetation and pushed into piles to create a clear zone around the fence. The cleared vegetation had not grown back.

4.5.2 Vegetation Community Description

The vegetation at PAOC-J and PAOC-K is a moderately sparse thorn scrub community dominated by Mesquite (*Prosopis juliflora),* aroma *(Acacia farnesian)* and twisted acacia (*A. tortuosa).* These species create a sparse canopy with adequate light penetration for good herbaceous ground cover. Camp Garcia

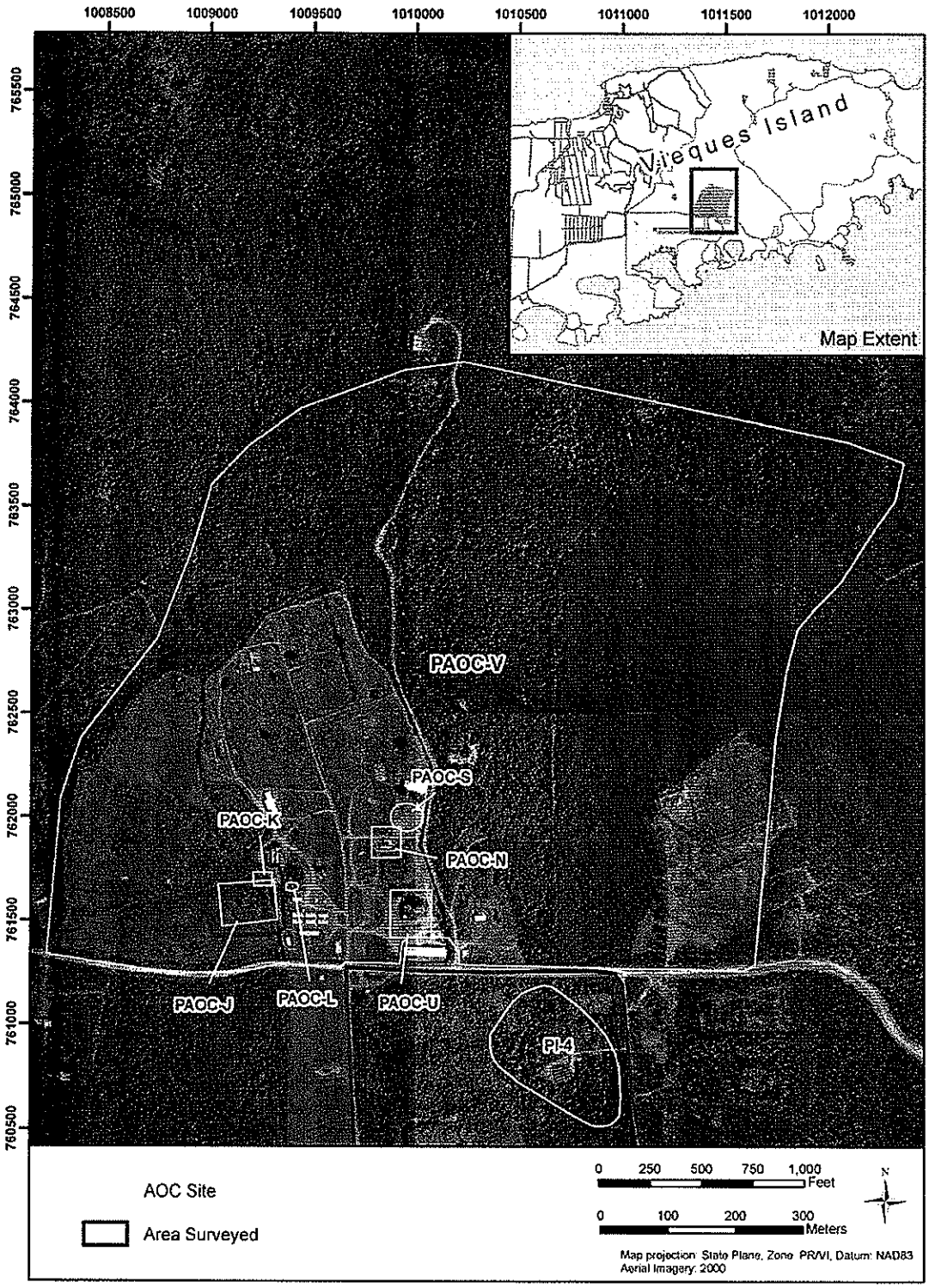

Figure 5. Locations of PAOC J, K and PI-4 at or Adjacent to the Former Camp Garcia Area.

is a combination of a grassy maintained area with few isolated trees around the housing and utilitarian facilities. A wire fence transformed in a sort of paradise for weedy climbers, and a highly disturbed, relatively open area surrounding the whole site, dominated by mesquite and two *Acacia* species. Due to the heavy human impact, this area has the largest percentage of herbaceous and weedy vegetation recored for any of the sites visited (74%). There were 54 species of plants recored for all two sites (**Table 6**).

Table 6
List of All Plants Recorded in the PAOC J and K Areas of the former Camp Garcia Area within the Former VNTR, 20 September 2005 (54 Species Total)

Family	Species	Common Name(s)
MALVACEAE	*Abutilon hirtum* (Lam.) Sweet	Buenos días
MIMOSACEAE	*Acacia farnesiana* (L.) Willd.	Aroma; Casha
MIMOSACEAE	*Acacia tortuosa* (L.) Willd.	Casia; Twisted acacia
AMARANTHACEAE	*Achyrantes aspera* L.	Rabo de gato; Man-better man
ANNONACEAE	*Annona reticulata* L.	Corazón; Custard apple
NYCTAGINACEAE	*Boerhavia coccinea* Mill.	Mata de pavo
POACEAE	*Bothriochloa pertusa* (L.) A. Camus	Huracán; Hurricane grass
ASCLEPIADACEAE	*Calotropis procera* (Ait.) Ait. f.	Algodón de seda; Giant milkweed
SCROPHULARIACEAE	*Capraria biflora* L.	Té del país; Goat weed
CARICACEAE	*Carica papaya* L.	Lechosa, Papaya; Papaw
FLACOURTIACEAE	*Casearia sylvestris* Sw.	Cafeíllo cimarrón, Sarna de perro
POACEAE	*Cenchrus brownii* Roem. & Schult.	Abrojito
FABACEAE	*Centrosema virginianum* (L.) Benth.	Conchita de Virginia
CAESALPINACEAE	*Chamaecrista diphylla* (L.) Greene	Hediondilla; Wild peaunt
CAESALPINACEAE	*Chamaecrista nictitans* (L.) Moench	Moriviví bobo
EUPHORBIACEAE	*Chamaesyce hirta* (L.) Millsp.	Lechecillo
EUPHORBIACEAE	*Chamaesyce hyssopifolia* (L.) Small	Lechera
EUPHORBIACEAE	*Chamaesyce prostrata* (Ait.) Small	Lechecillo
POACEAE	*Chloris inflata* Link	Horquetilla morada; Blue grass
CAPPARACEAE	*Cleome gynandra* L.	Jazmín de río; White massambee
CAPPARACEAE	*Cleome viscosa* L.	Volantín amarillo; Yellow massambee
COMMELINACEAE	*Commelina diffusa* Burm. f.	Cohitre; Blue day flower
BORAGINACEAE	*Cordia collococca* L.	Capá rojo, Ateje; Red manjack
FABACEAE	*Crotalaria incana* L.	Cascabelillo vacio; Shake-shake
FABACEAE	*Crotalaria retusa* L.	Cascabelillo
CUCURBITACEAE	*Cucumis anguria* L.	Pepinillo silvestre
POACEAE	*Cynodon dactylon* (L.) Pers.	Yerba bermuda; Bermuda grass
EUPHORBIACEAE	*Dalechampia scandens* L.	—
POACEAE	*Digitaria insularis* (L) Mez	Zorra; Silky grass
POACEAE	*Eleusine indica* (L.) Gaertn.	Grama de caballo; wire grass
EUPHORBIACEAE	*Euphorbia heterophylla* L.	Acerca amor
RUBIACEAE	*Hedyotis corymbosa* (L.) Lam.	Graciosa
BORAGINACEAE	*Heliotropium indicum* L.	Cotorrera; Indian heliotrope
CONVOLVULACEAE	*Ipomoea tiliacea* (Willd.) Choisy in DC.	Bejuco blanco; Goat foot
OLEACEAE	*Jasminum fluminense* Vell.	Jasmín oloroso
EUPHORBIACEAE	*Jatropha gossypiifolia* L.	Higuereta cimarrona

Table 6 (Continued)
List of All Plants Recorded in the PAOC J and K Areas of the former Camp Garcia Area within the Former VNTR, 20 September 2005 (54 Species Total)

Family	Species	Common Name(s)
VERBENACEAE	*Lantana camara* L.	Cariaquillo; Yellow sage
STERCULIACEAE	*Melochia nodiflora* Sw.	Bretónica prieta
CONVOLVULACEAE	*Merremia quinquefolia* (L) Hall. f.	Batatilla blanca
CUCURBITACEAE	*Momordica charantia* L.	Cundeamor; Wild balsam apple
POACEAE	*Panicum maximum* Jacq.	Yerba de guinea; Guinea grass
POACEAE	*Paspalum fimbriatum* Kunth	Pata de conejo
PASSIFLORACEAE	*Passiflora foetida* L.	Tagua-Tagua
EUPHORBIACEAE	*Phyllanthus amarus* Schumach.	Quinino de pobre
PORTULACACEAE	*Portulaca oleracea* L.	Verdolaga; Purs lane
MIMOSACEAE	*Prosopis juliflora* (Sw.) DC.	Mesquite
RUBIACEAE	*Randia aculeata* L.	Tintillo; Ink-berry
FABACEAE	*Rhynchosia minima* (L.) DC.	Frijolillo
ACANTHACEAE	*Ruellia tuberosa* L.	Saltaperico; Many-roots
MALVACEAE	*Sida abutifolia* Mill.	Escoba tendida
MALVACEAE	*Sida stipularis* Cav.	Escoba blanca; Wire weed
FABACEAE	*Stylosanthes hamata* (L.) Taubert in Verh.	Zarzabacoa enana; Pencil flower
PHYTOLACACEAE	*Trichostigma octandrum* (L.) H. Walt.	Bejuco de nasa
ASTERACEAE	*Tridax procumbens* L.	Pancha, San Juan del monte; Tridax

Note: Following the conservation of the name *Leguminosae* for the family (*sensu latiore*) in the third edition of the International Code of Botanical Nomenclature, many authors treat the three families of *Order Fabales* as one single family (*Leguminosae*) with three subfamilies. However, we distinguish three different families (*Fabaceae*, *Caesalpinaceae* and *Mimosaceae*) as does A. Cronquist in *The Evolution and Classification of Flowering Plants* (1988).

4.5.3 Wildlife Description

Several common wildlife species were observed utilizing the grassy areas and thorn scrub habitat. Wildlife observed at PAOC-J and PAOC-K, and adjacent habitat, are presented in **Table 7**.

4.5.4 Protected Species

The PAOC-K and PAOC-J sites were located in thorn scrub forest habitat. There were no federally protected species or preferred habitats observed at PAOC-K or PAOC-J.

4.6 PI-4 (Former Waste Treatment Site)

4.6.1 Site Description

This site is located south of Camp Garcia across the road and north of the east end of the airfield (**Figure 6**). This site appears to have been a former treatment area for waste. There are numerous concrete foundations, concrete culverts with ditches and piles of rubble and soil.

4.6.2 Vegetation Community Description

Habitat in this area is mostly a man-made *Prosopis/Acacia* successional savanna with a dense, nearly continuous herbaceous layer (*Commelina diffusa)* covering most ground surface including the concrete pads. There are also many invasive shrubs and vines, the great majority of which are weeds (22% of all

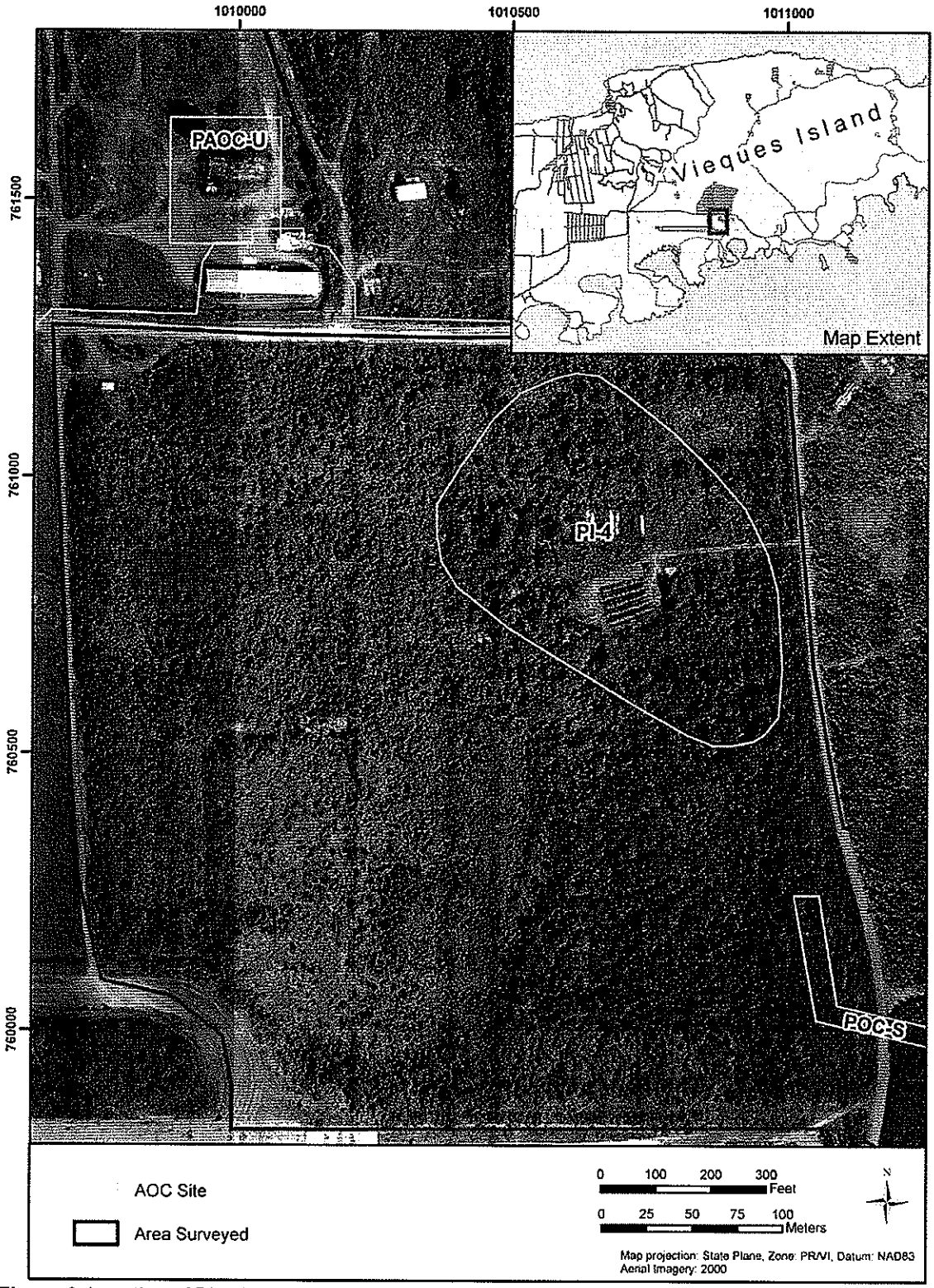

Figure 6. Location of PI-4 (Former Waste Treatment Site).

Table 7
List of All Animals Recorded in the former Camp Garcia Area within the Former VNTR, 20 September 2005

Common Name	Scientific Name	PAOC-J,K,S
Mammals		
Mongoose	*Herpestes auropunctatus*	x
Reptiles and Amphibians		
Anole (Common Lizard)	*Anolis cristatellus*	x
Grass Anole (Garden Lizard)	*Anolis pulchellus*	x
Anole (Spotted Lizard)	*Anolis stratulus*	x
Birds		
Bananaquit	*Coereba flaveola*	x
American Kestrel	*Falco sparverius*	x
Northern Mockingbird	*Mimus polyglottos*	x
Puerto Rican Flycatcher	*Myiarchus antillarum*	x
Greater Antillean Grackle	*Quiscalus niger*	x
Gray Kingbird	*Tyrannus dominicensis*	x
Zenaida Dove	*Zenaida aurita*	x

species recorded here correspond to creepers, climbers and vines). Non-leguminous trees are scarce, both in number of species and individuals. These are: *Ziziphus mauritiana*, *Bucida buceras*, *Citharexylum fruticosum*, *Zanthoxylum monophyllum*, *Bursera simaruba*, and *Bourreria succulenta*. Low herbaceous plants dominate the area surrounding the former runway. There were 99 species of plants recorded from this area (**Table 8**).

Table 8
List of All Plants Recorded in the PI-4 and Runway Areas within the former VNTR, 20 September 2005 (99 Species Total)

Family	Species	Common Name(s)
MIMOSACEAE	*Acacia tortuosa* (L.) Willd.	Casia; Twisted acacia
EUPHORBIACEAE	*Acalypha indica* L.	—
AMARANTHACEAE	*Achyrantes aspera* L.	Rabo de gato; Man-better man
FABACEAE	*Aeschynomene americana* L.	Yerba Rosario
ASTERACEAE	*Ageratum conyzoides* L. ssp. *conyzoides*	Mentastro, Yerba de cabrío
STERCULIACEAE	*Ayenia insulicola* Cristóbal	—
ASTERACEAE	*Bidens alba* (L.) DC. var. (CH. Schulz) Melchert	Margarita
NYCTAGINACEAE	*Boerhavia coccinea* Mill.	Mata de pavo
NYCTAGINACEAE	*Boerhavia diffusa* L.	Mata pavo; Hog-weed
POACEAE	*Bothriochloa pertusa* (L.) A. Camus	Huracán; Hurricane grass
BORAGINACEAE	*Bourreria succulenta* Jacq.	Palo de vaca
POACEAE	*Bouteloua americana* (L.f.) Scribn.	Yerba mesquite
POACEAE	*Brachiaria fasciculata* (Sw.) S. T. Blake	—
POACEAE	*Brachiaria* sp.	—
COMBRETACEAE	*Bucida buceras* L.	Úcar
BURSERACEAE	*Bursera simaruba* (L.) Sarg.	Almácigo
ASCLEPIADACEAE	*Calotropis procera (Ait.) Ait. f.*	Algodón de seda; Giant milkweed
CAPPARACEAE	*Capparis flexuosa* (L.) L.	Palinguán; Caper tree

Table 8 (Continued)
List of All Plants Recorded in the PI-4 and Runway Areas within the former VNTR, 20 September 2005 (99 Species Total)

Family	Species	Common Name(s)
SCROPHULARIACEAE	*Capraria biflora* L.	Té del país; Goat weed
POACEAE	*Cenchrus myosuroides* HBK.	Abrojo de espiga; Spiked-bur grass
FABACEAE	*Centrosema pubescens* Benth.	Flor de conchitas; Butterfly pea
FABACEAE	*Centrosema virginianum* (L.) Benth.	Conchita de Virginia
CAESALPINACEAE	*Chamaecrista diphylla* (L.) Greene	Hediondilla; Wild peaunt
CAESALPINACEAE	*C. nictitans* ssp. *pattelaria* (Colladon) Irwin & Barneby	Moriviví bobo
EUPHORBIACEAE	*Chamaesyce hirta* (L.) Millsp.	Lechecillo
EUPHORBIACEAE	*Chamaesyce hyssopifolia* (L.) Small	Lechera
EUPHORBIACEAE	*Chamaesyce turpinii* (Boiss.) Millsp.	Lecherita
POACEAE	*Chloris inflata* Link	Horquetilla morada; Blue grass
POACEAE	*Chloris radiata* (L.) Sw.	Grama de costa; Plush-grass
VITACEAE	*Cissus verticillata* (L.) Nicolson & Jarvis	Bejuco de Caro; Pinakoop
VERBENACEAE	*Citharexylum fruticosum* L.	Palo de guitarra; Old woman bitter
CAPPARACEAE	*Cleome viscosa* L.	Volantín amarillo; Yellow massambee
COMMELINACEAE	*Commelina diffusa* Burm. f.	Cohitre; Blue day flower
TILIACEAE	*Corchorus hirsutus* L.	Malvaté
BORAGINACEAE	*Cordia collococca* L.	Capá rojo, Ateje; Red manjack
FABACEAE	*Crotalaria retusa* L.	Cascabelillo
CUCURBITACEAE	*Cucumis anguria* L.	Pepinillo silvestre
FABACEAE	*Dalbergia ecastaphyllum* (L.) Taub.	Maraimaray
EUPHORBIACEAE	*Dalechampia scandens* L.	—
POACEAE	*Digitaria insularis* (L) Mez	Zorra; Silky grass
POACEAE	*Eragrostis pilosa* (L.) Beauv.	—
POACEAE	*Eragrostis* sp.	—
CONVOLVULACEAE	*Evolvulus convolvuloides* Stearn	—
FABACEAE	*Galactia dubia* DC.	—
FABACEAE	*Galactia striata* (Jacq.) Urban	—
BORAGINACEAE	*Heliotropium curassavicum* L.	Cotorra de playa; Seaside heliotrope
CONVOLVULACEAE	*Ipomoea indica* (Burm. f.) Merr.	Bejuco de gloria
CONVOLVULACEAE	*Ipomoea tiliacea* (Willd.) Choisy in DC.	Bejuco blanco; Goat foot
CONVOLVULACEAE	*Ipomoea triloba* L.	Bejuquillo de puerco
OLEACEAE	*Jasminum fluminense* Vell.	Jasmín oloroso
EUPHORBIACEAE	*Jatropha gossypiifolia* L.	Higuereta cimarrona
VERBENACEAE	*Lantana camara* L.	Cariaquillo; Yellow sage
VERBENACEAE	*Lantana involucrata* L.	Santa María; Button sage
MIMOSACEAE	*Leucaena leucocephala* (Lam.) De Wit	Acacia pálida; Wild tamarind
BIGNONIACEAE	*Macfadyena unguis-cati* (L.) A. Gentry	Bejuco de gato; Cat´s claw
MALVACEAE	*Malvastrum americanum* (L.) Torrey	Malva silvestre
MALVACEAE	*Malvastrum corchorifolium* (Desr.) Britton	Escoba blanca
STERCULIACEAE	*Melochia nodiflora* Sw.	Bretónica prieta
STERCULIACEAE	*Melochia pyramidata* L.	Bretónica piramidal

Table 8 (Continued)
List of All Plants Recorded in the PI-4 and Runway Areas within the former VNTR, 20 September 2005 (99 Species Total)

Family	Species	Common Name(s)
CUCURBITACEAE	*Melothria pendula* L.	Pepinillo
CONVOLVULACEAE	*Merremia quinquefolia* (L) Hall. f.	Batatilla blanca
POACEAE	*Panicum maximum* Jacq.	Yerba de guinea; Guinea grass
CAESALPINACEAE	*Parkinsonia aculeata* L.	Palo de rayo; Horsebean, Jerusalem thorn
POACEAE	*Paspalum distichum* L.	—
POACEAE	*Paspalum fimbriatum* Kunth	Pata de conejo
POACEAE	*Paspalum virgatum* L.	Cortadero, Matojo blanco
POACEAE	*Paspalum* sp.	—
PASSIFLORACEAE	*Passiflora suberosa* L.	Flor de pasion
MALVACEAE	*Pavonia spinifex* (L.) Cav.	Cadillo espinoso
EUPHORBIACEAE	*Phyllanthus niruri* L.	Quinino; Gale of the wind
PORTULACACEAE	*Portulaca oleracea* L.	Verdolaga; Purs lane
PORTULACACEAE	*Portulaca quadrifida* L.	Yerba de pollo; Chicken weed
PORTULACACEAE	*Portulaca teretifolia* Kunth	Yerba verdolaga
MIMOSACEAE	*Prosopis juliflora* (Sw.) DC.	Mesquite
RUBIACEAE	*Randia aculeata* L.	Tintillo; Ink-berry
APOCYNACEAE	*Rauvolfia viridis* Willd. ex Roem. & Schultes	Cachimbo blanco; Bitter bush
FABACEAE	*Rhynchosia reticulata* (Sw.) DC.	Frijolillo de monte
PHYTOLACCACEAE	*Rivina humilis* L.	Carmín; Cat´s blood
ACANTHACEAE	*Ruellia tuberosa* L.	Saltaperico; Many-roots
POACEAE	*Setaria geniculata* (Lam.) Beauv.	Arrocillo; Yellow fostail
MALVACEAE	*Sida ciliaris* L.	Escoba peluda, Escoba pestañosa
MALVACEAE	*Sidastrum multiflorum* (Jacq.) Fryxell	Sidastro
POACEAE	*Spartina patens* (Ait.) Muhl.	Yerba de sal; Salt grass
RUBIACEAE	*Spermacoce verticillata* L.	Botón blanco
LOGANIACEAE	*Spigelia anthelmia* L.	Lombricera; Waterweed
POACEAE	*Sporobolus jacquemontii* Kunth	Pelo de burro
VERBENACEAE	*Stachytarpheta jamaicensis* (L.) Vahl	Bretónica
POACEAE	*Stenotaphrum secundatum* (Walt.) Kuntze	Cinta; Crab grass
MALPIGHIACEAE	*Stigmaphyllon emarginatum* (Cav.) A. Juss.	Bejuco de San Pedro
FABACEAE	*Stylosanthes hamata* (L.) Taubert in Verh.	Zarzabacoa enana; Pencil flower
FABACEAE	*Tephrosia cinerea* (L.) Pers.	Añil cenizo
BORAGINACEAE	*Tournefortia volubilis* L.	Nigua enredadera
EUPHORBIACEAE	*Tragia volubilis* L.	Pringamoza; Stinging vine
MALVACEAE	*Urena lobata* L.	Cadillo; Bur
STERCULIACEAE	*Waltheria indica* L.	Basora prieta, Malvavisco
MALVACEAE	*Wissadula periplocifolia* (L.) Presl	Pichona
RUTACEAE	*Zanthoxylum monophyllum* (Lam.) P. Wilson	Carrubio, Espino rubial; Yellow prickle
RHAMNACEAE	*Ziziphus mauritiana* Lam.	Aprín; Indian jujube

*In sterile condition (no flowers or fruits available for determination)

Note: Following the conservation of the name *Leguminosae* for the family (*sensu latiore*) in the third edition of the International Code of Botanical Nomenclature, many authors treat the three families of *Order Fabales* as one single family (*Leguminosae*) with three subfamilies. However, I distinguish three different families (*Fabaceae*, *Caesalpinaceae* and *Mimosaceae*) as does A. Cronquist in *The Evolution and Classification of Flowering Plants* (1988). This treatment is strongly supported by molecular data and no doubt will prevail in the future.

4.6.3 Wildlife Description

Common wildlife species were observed utilizing the vegetated areas, pipes, concrete pads, concrete culverts, edges of the cleared areas, and soil piles, and vegetation. Wildlife observed at PI-4, and adjacent habitat, is presented in **Table 9**.

Table 9
List of All Animals Recorded in the PI-4 Area within the former VNTR, 20 September 2005

Common Name	Scientific Name	PI-4
Mammals		
Mongoose	*Herpestes auropunctatus*	x
Reptiles and Amphibians		
Anole (Common Lizard)	*Anolis cristatellus*	x
Grass Anole (Garden Lizard)	*Anolis pulchellus*	x
Anole (Spotted Lizard)	*Anolis stratulus*	x
Birds		
Cattle Egret	*Bubulcus ibis*	x
Killdeer	*Charadrius vociferus*	x
Mangrove Cuckoo	*Coccyzus minor*	x
Bananaquit	*Coereba flaveola*	x
Scaly-naped pidgeon	*Columba squamosa*	x
Common ground Dove	*Columbina passerina*	x
Smooth-billed Ani	*Crotophaga ani*	x
Adelaide's Warbler	*Dendroica adelaidae*	x
Caribbean Elaenia	*Elaenia martinica*	x
American Kestrel	*Falco sparverius*	x
Scaly-Breasted Thrasher	*Margarops fuscus*	x
Puerto Rico Woodpecker	*Melanerpes portoricensis*	x
Northern Mockingbird	*Mimus polyglottos*	x
Puerto Rican Flycatcher	*Myiarchus antillarum*	x
Antillean-crested Hummingbird	*Orthorhyncus cristatus*	x
Greater Antillean Grackle	*Quiscalus niger*	x
Gray Kingbird	*Tyrannus dominicensis*	x
White-winged dove	*Zenaida asiatica*	x

4.6.4 Protected Species

The site was located within young thorn scrub forest habitat. There were no federally protected species or preferred habitats observed at this site

4.7 PI-7 (FORMER RADAR AND QUARRY SITES)

4.7.1 Site Description

The two PI-7 sites are located northeast of Camp Garcia and east of Vieques township (**Figure 7**). This site appears to have been a former radar and quarry site.

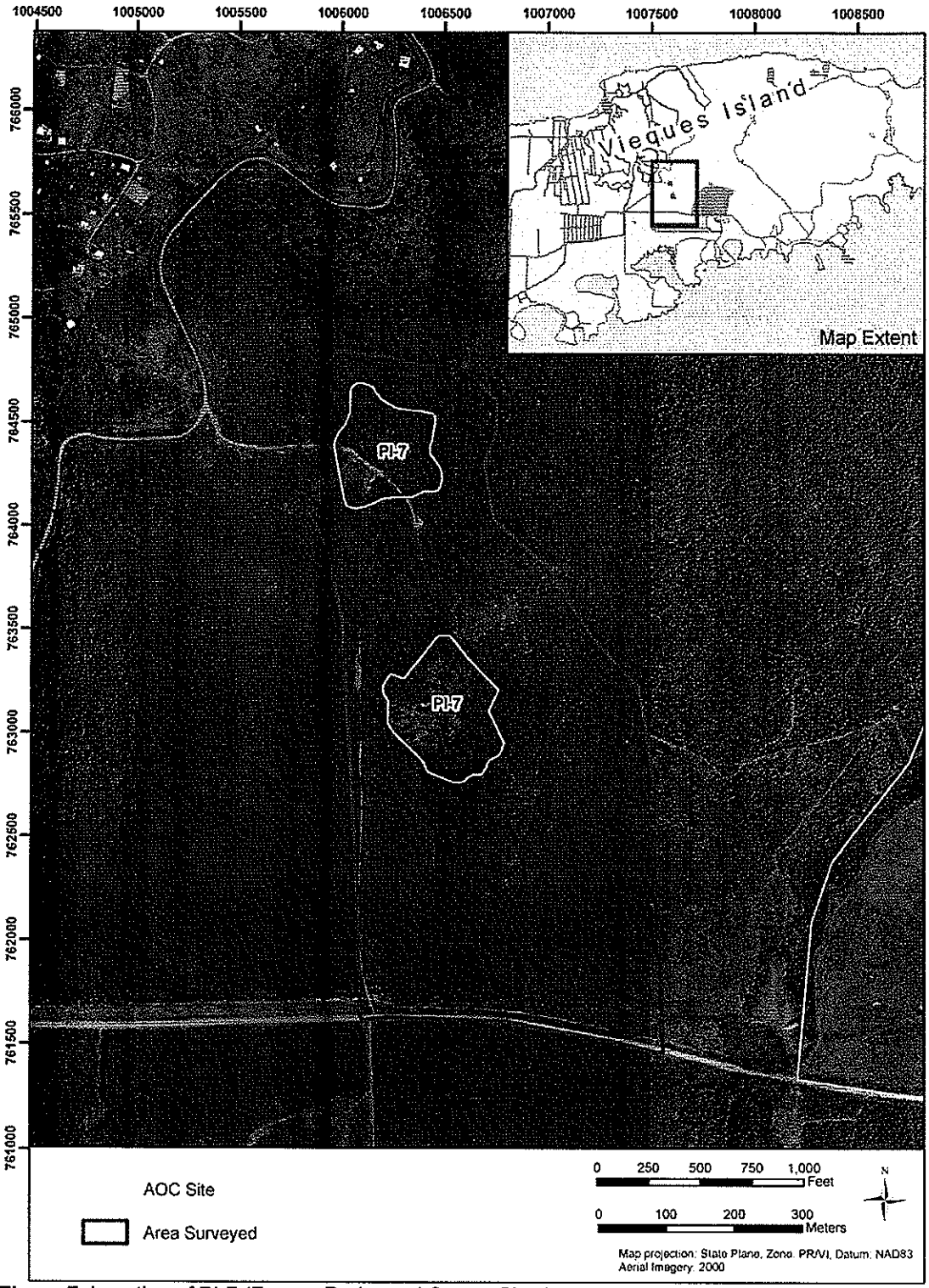

Figure 7. Location of PI-7 (Former Radar and Quarry Sites).

4.7.2 Vegetation Community Description

This site is a mixed woodland/upland forest (moist deciduous formation) which is found mostly on the inner hill and slopes primarily on the western side of Vieques. Dominant vegetation was seasonal forests and woodlands (on the moist humid side of the hills especially along drains and gullies), shrubs, and dense grassland areas on the south and southeastern slopes of radar hill, these were probably established by repeated fires during training activities (GMI 1996; 2001). Common trees are *Andira inermis*, *Prosopis juliflora*, various *Acacia* and *Casearia* species, *Trichilia hirta*, *Cordia collococca*, *Piscidia carthagenensis*, *Bursera simaruba*, *Guapira fragrans*, *Zanthoxylum monophyllum*, *Schaefferia frutescens*, and *Ziziphus mauritiana*. Despite the disturbance of former times and the great number of invasive weeds (mostly along the road and open areas), the site seems to be slowly recovering. There wer 101 plants recorded at these two sites (**Table 10**).

Table 10
List of All Plants Recorded in the PI-7 Area (Radar Hill and Old Quarry/Dumpsite) within the Former VNTR, 20 September 2005 (101 Species Total)

Family	Species	Common Name(s)
FABACEAE	*Abrus precatorius* L.	Ojo de cangrejo; Crab´s eyes
MALVACEAE	*Abutilon umbellatum* (L.) Sweet	Malvavisco cimarrón
MIMOSACEAE	*Acacia farnesiana* (L.) Willd.	Aroma; Casha
MIMOSACEAE	*Acacia macracantha* Humb. & Bonpl.	Tamarindo silvestre; Tamarind
MIMOSACEAE	*Acacia retusa* (Jacq.) Howard	Acacia zarza
MIMOSACEAE	*Acacia tortuosa* (L.) Willd.	Casia; Twisted acacia
AMARANTHACEAE	*Achyrantes aspera* L.	Rabo de gato; Man-better man
FABACEAE	*Andira inermis* (W. Wr.) DC.	Moca; Angelin tree
EUPHORBIACEAE	*Argythamnia fasciculata* (Vahl) Muell. Arg.	----
MALVACEAE	*Bastardia viscosa* (L.) HBK	Escoba babosa; Viscid mallow
NYCTAGINACEAE	*Boerhavia coccinea* Mill.	Mata de pavo
POACEAE	*Bothriochloa pertusa* (L.) A. Camus	Huracán; Hurricane grass
POACEAE	*Brachiaria adspersa* (Trin.) Parodi	Millito; Brown top millet
BURSERACEAE	*Bursera simaruba* (L.) Sarg.	Almácigo
CAPPARACEAE	*Capparis flexuosa* (L.) L.	Palinguán; Caper tree
FLACOURTIACEAE	*Cassearia guianensis* (Aubl.) Urban	Cafeíllo; Wild coffee
FLACOURTIACEAE	*Cassearia sylvestris* Sw.	Cafeíllo cimarrón, Sarna de perro
APOCYNACEAE	*Catharanthus roseus* (L.) G. Don	Playera; Periwinkle
POACEAE	*Cenchrus echinatus* L.	Abrojo; Bur grass
FABACEAE	*Centrosema pubescens* Benth.	Flor de conchitas; Butterfly pea
FABACEAE	*Centrosema virginianum* (L.) Benth.	Conchita de Virginia
CAESALPINACEAE	*Chamaecrista diphylla* (L.) Greene	Hediondilla; Wild peanut
CAESALPINACEAE	*Chamaecrista nictitans* (L.) Moench	Moriviví bobo
EUPHORBIACEAE	*Chamaesyce hirta* (L.) Millsp.	Lechecillo
EUPHORBIACEAE	*Chamaesyce hyssopifolia* (L.) Small	Lechera
POACEAE	*Chloris inflata* Link	Horquetilla morada; Blue grass
VITACEAE	*Cissus trifoliata* L.	Caro costero
VITACEAE	*Cissus verticillata* (L.) Nicolson & Jarvis	Bejuco de Caro; Pinakoop
VERBENACEAE	*Citharexylum fruticosum* L.	Palo de guitarra; Old woman bitter
ARECACEAE	*Coccothrinax alta* (O. F. Cook) Becc.	Palma plateada; Silver palm
COMMELINACEAE	*Commelina diffusa* Burm. f.	Cohitre; Blue day flower
BORAGINACEAE	*Cordia collococca* L.	Capá rojo, Ateje; Red manjack
FABACEAE	*Crotalaria falcata* Vahl ex DC.	Matraca; Rattlebox

Table 10 (Continued)
List of All Plants Recorded in the PI-7 Area (Radar Hill and Old Quarry/Dumpsite) within the Former VNTR, 20 September 2005 (101 Species Total)

Family	Species	Common Name(s)
FABACEAE	*Crotalaria retusa* L.	Cascabelillo
EUPHORBIACEAE	*Croton astroites* Dryand.	Maná
EUPHORBIACEAE	*Croton lobatus* L.	Croto lobulado
BIGNONIACEAE	*Cydista aequinoctialis* (L.) Miers	Bejuco blanco; Guard withe
CYPERACEAE	*Cyperus compressus* L.	—
POACEAE	*Dactyloctenium aegyptium* (L.) Beauv.	Yerba egipcia; Egyptian grass
EUPHORBIACEAE	*Dalechampia scandens* L.	----
MIMOSACEAE	*Desmanthus virgatus* (L.) Willd.	Desmanto
FABACEAE	*Desmodium* sp.	—
POACEAE	*Digitaria bicornis* (L.) R. & S.	—
POACEAE	*Digitaria insularis* (L) Mez	Zorra; Silky grass
BIGNONIACEAE	*Distictis lactiflora* (Vahl) DC.	Liana fragante
ERYTHROXYLACEAE	*Erythroxylum brevipes* DC.	Brisselet
EUPHORBIACEAE	*Euphorbia heterophylla* L.	Acerca amor
FABACEAE	*Galactia striata* (Jacq.) Urban	—
NYCTAGINACEAE	*Guapira fragans* (Dum.-Cours.) Little	Corcho; Black mampoo
BORAGINACEAE	*Heliotropium indicum* L.	Cotorrera; Indian heliotrope
FABACEAE	*Indigofera suffruticosa* Miller	Añil; Índigo
CONVOLVULACEAE	*Ipomoea indica* (Burm. f.) Merr.	Bejuco de gloria
CONVOLVULACEAE	*Ipomoea tiliacea* (Willd.) Choisy in DC.	Bejuco blanco; Goat foot
CONVOLVULACEAE	*Ipomoea triloba* L.	Bejuquillo de Puerco
OLEACEAE	*Jasminum fluminense* Vell.	Jasmín oloroso
EUPHORBIACEAE	*Jatropha gossypiifolia* L.	Higuereta cimarrona
MIMOSACEAE	*Leucaena leucocephala* (Lam.) De Wit	Acacia pálida; Wild tamarind
BIGNONIACEAE	*Macfadyena unguis-cati* (L.) A. Gentry	Bejuco de gato; Cat´s claw
FABACEAE	*Macroptilium lathyroides* (L.) Urban	Habichuela parada; Wild bush bean
MALVACEAE	*Malachra alceifolia* Jacq.	Malva de caballo; Bastard okra
MALVACEAE	*Malvastrum* sp.	—
SAPINDACEAE	*Melicoccus bijugatus* Jacq.	Quenepa; Ginep
STERCULIACEAE	*Melochia nodiflora* Sw.	Bretónica prieta
CONVOLVULACEAE	*Merremia quinquefolia* (L) Hall. f.	Batatilla blanca
POACEAE	*Panicum maximum* Jacq.	Yerba de guinea; Guinea grass
POACEAE	*Paspalum fimbriatum* Kunth	Pata de conejo
EUPHORBIACEAE	*Phyllanthus niruri* L.	Quinino; Gale of the wind
FABACEAE	*Piscidia carthagenensis* Jacq.	Ventura; Fish poison
MIMOSACEAE	*Pithecellobium unguis-cati* (L.) Mart.	Escambrón colorado; Black bean
PORTULACACEAE	*Portulaca oleracea* L.	Verdolaga; Purs lane
MIMOSACEAE	*Prosopis juliflora* (Sw.) DC.	Mesquite
RUBIACEAE	*Randia aculeata* L.	Tintillo; Ink-berry
APOCYNACEAE	*Rauvolfia viridis* Willd. ex Roem. & Schultes	Bitter bush
FABACEAE	*Rhynchosia reticulata* (Sw.) DC.	Frijolillo de monte
PHYTOLACCACEAE	*Rivina humilis* L.	Carmín; Cat´s blood
MIMOSACEAE	*Samanea saman* (Willd.) Merrill	Samán; Rain tree
CELASTRACEAE	*Schaefferia frutescens* Jacq.	Cafeíllo; Florida boxwood
CAESALPINACEAE	*Senna obtusifolia* (L.) Irwin & Barneby	Dormidera, Dormilona

Table 10 (Continued)
List of All Plants Recorded in the PI-7 Area (Radar Hill and Old Quarry/Dumpsite) within the Former VNTR, 20 September 2005 (101 Species Total)

Family	Species	Common Name(s)
CAESALPINACEAE	*Senna occidentales* (L.) Link	Hedionda; Coffee sena
SAPINDACEAE	*Serjania polyphylla* (L.) Radlk.	Bejuco de canastas; Basket wood
POACEAE	*Setaria setosa* (Sw.) Scribn.	Yerba suave
MALVACEAE	*Sida abutifolia* Mill.	Escoba tendida
MALVACEAE	*Sida ciliaris* L.	Escoba peluda, Escoba pestañosa
MALVACEAE	*Sida cordifolia* L.	Escoba acorazonada
MALVACEAE	*Sida rhombifolia* L.	Escoba colorada
MALVACEAE	*Sidastrum multiflorum* (Jacq.) Fryxell	Sidastro
SAPOTACEAE	*Sideroxylon salicifolium* (L.) Lam.	Sabina; Willow bustic
RUBIACEAE	*Spermacoce verticillata* L.	Botón blanco
POACEAE	*Sporobolus jacquemontii* Kunth	Pelo de burro
VERBENACEAE	*Stachytarpheta jamaicensis* (L.) Vahl	Bretónica
MALPIGHIACEAE	*Stigmaphyllon emarginatum* (Cav.) A. Juss.	Bejuco de San Pedro
FABACEAE	*Stylosanthes hamata* (L.) Taubert in Verh.	Zarzabacoa enana; Pencil flower
FABACEAE	*Tephrosia cinerea* (L.) Pers.	Añil cenizo
EUPHORBIACEAE	*Tragia volubilis* L.	Pringamoza; Stinging vine
MELIACEAE	*Trichilia hirta* L.	Retamo; Broomstick
ASTERACEAE	*Tridax procumbens* L.	Pancha, Tridax
TILIACEAE	*Triumpheta semitriloba* Jacq.	Cadillo de perro
STERCULIACEAE	*Waltheria indica* L.	Basora prieta, Malvavisco
RUTACEAE	*Zanthoxylum monophyllum* (Lam.) P. Wilson	Carrubio, Espino rubial; Yellow prickle
RHAMNACEAE	*Ziziphus mauritiana* Lam.	Aprí; Indian jujube
COMMELINACEAE	Indet.*	----

*In sterile condition (no flowers or fruits available for determination)

Note: Following the conservation of the name *Leguminosae* for the family (*sensu latiore*) in the third edition of the International Code of Botanical Nomenclature, many authors treat the three families of *Order Fabales* as one single family (*Leguminosae*) with three subfamilies. However, we distinguish three different families (*Fabaceae*, *Caesalpinaceae* and *Mimosaceae*) as does A. Cronquist in *The Evolution and Classification of Flowering Plants* (1988

4.7.3 Wildlife Description

Several common wildlife species were observed utilizing woody vines, brush piles and soil piles, and vegetation. Wildlife observed at PI-7 are presented in **Table 11**.

4.7.4 Protected Species

The site was located within thorn scrub forest habitat. There were no federally protected species or preferred habitats observed at this site.

Table 11
List of All Plants Recorded In the PI-7 Area (Radar Hill and Old Quarry/Dumpsite) within the former VNTR, 20 September 2005

Common Name	Scientific Name	PI-7
Mammals		
Mongoose	*Herpestes auropunctatus*	x
Reptiles and Amphibians		
Anole (Common Lizard)	*Anolis cristatellus*	x
Grass Anole (Garden Lizard)	*Anolis pulchellus*	x
Anole (Spotted Lizard)	*Anolis stratulus*	x
Birds		
Green Mango	*Anthracothorax viridis*	x
Bananaquit	*Coereba flaveola*	x
Scaly-naped pidgeon	*Columba squamosa*	x
Common ground Dove	*Columbina passerina*	x
Smooth-billed Ani	*Crotophaga ani*	x
Adelaide's Warbler	*Dendroica adelaidae*	x
Caribbean Elaenia	*Elaenia martinica*	x
Puerto Rico Woodpecker	*Melanerpes portoricensis*	x
Northern Mockingbird	*Mimus polyglottos*	x
Puerto Rican Flycatcher	*Myiarchus antillarum*	x
Greater Antillean Grackle	*Quiscalus niger*	x
Zenaida Dove	*Zenaida aurita*	x

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HABITAT CHARACTERIZATION OF SOLID WASTE MANAGEMENT UNITS (SWMU) 4, SWMU 5, SWMU 6, SWMU 7, AND THE PUBLIC WORKS AREA NAVAL AMMUNITION SUPPORT (NASD) DETACHMENT, VIEQUES ISLAND, PUERTO RICO

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INTRODUCTION

As part of a Resource Conservation and Recovery Act (RCRA) facility investigation at Vieques, ecological risk assessments were conducted at 10 solid waste management unit (SWMU) sites. A habitat characterization was conducted at each SWMU in order to determine the presence of plant and animal species and to determine whether preferred habitat was present for any federally endangered or threatened plant and animal species.

SITE LOCATION

The Naval Ammunition Support Department (NASD) Vieques (approximately 8,265 acres) is operated by the Weapons Department of Naval Station Roosevelt Roads and is located on the western end of Vieques (Figure 1). This report covers 10 SWMU sites located at NASD Vieques (Figure 2). Four of the sites were separated geographically and were located in different plant communities. These sites, SWMU 4, SWMU 5, SWMU 6, and SWMU 7, are presented individually in this report. SWMU 4, an inactive Waste Explosive Open Burn/Detonation Range, is approximately 3 acres in size and is located on the western side of NASD. SWMU 5, IRFNA/MAF-4 Disposal Site, is located adjacent to magazine Building 422 and consists of a small ditch and ravine. SWMU 6, Mangrove Disposal Site, is located north of Laguna Kiani. SWMU 7, Quebrada Disposal Site, is approximately 5.0 acres in size and is located within a quebrada (drainage channel or ravine) located about 0.5 miles west of the NASD Public Works area.

Six of the SWMU sites (SWMU 10, SWMU 14, SWMU 15, AOC C, AOC E-UST, and AOC F-UIC) were located near each other and were potentially contaminated with similar debris; therefore, they were analyzed as one unit and are presented in the same section of this report. These six sites were all located in the NASD Public Works area.

METHODS

Vegetation communities were initially characterized into broad community types based on color signatures from true-color aerial photographs (November 4, 1999: scale, RF=1:6000). Different vegetation communities were delineated based on species composition and structure by viewing magnified stereo pairs of aerial photography. The community types were marked on overlying acetate for use in the field (May 15-19, 2000). Personnel walked transects through each of these SWMU to:

1. verify that the community types were identified and delineated correctly from the true color aerial photography;
2. identify the species composition of the dominant vegetation;
3. identify the wildlife species present in the SWMU sites;

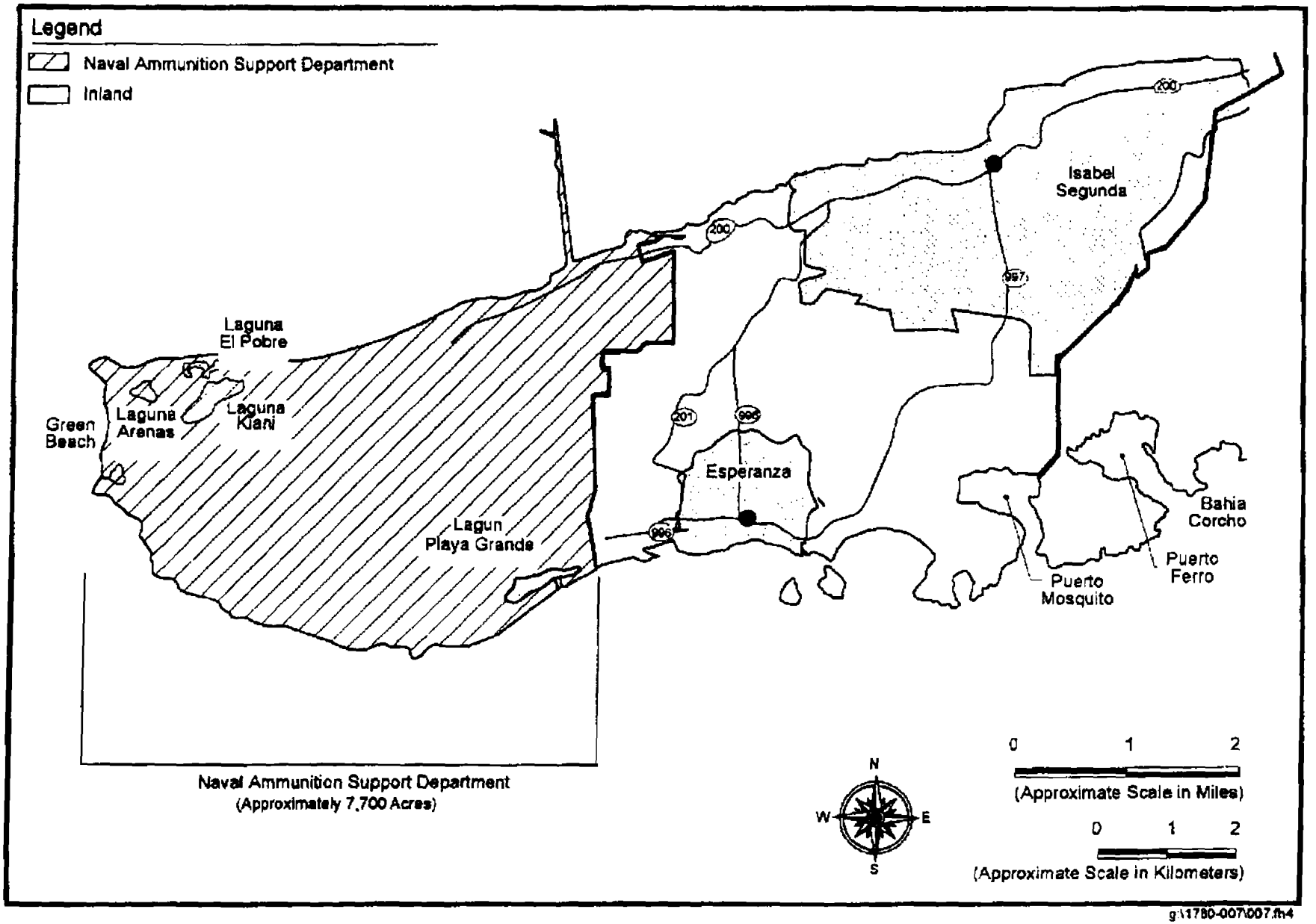

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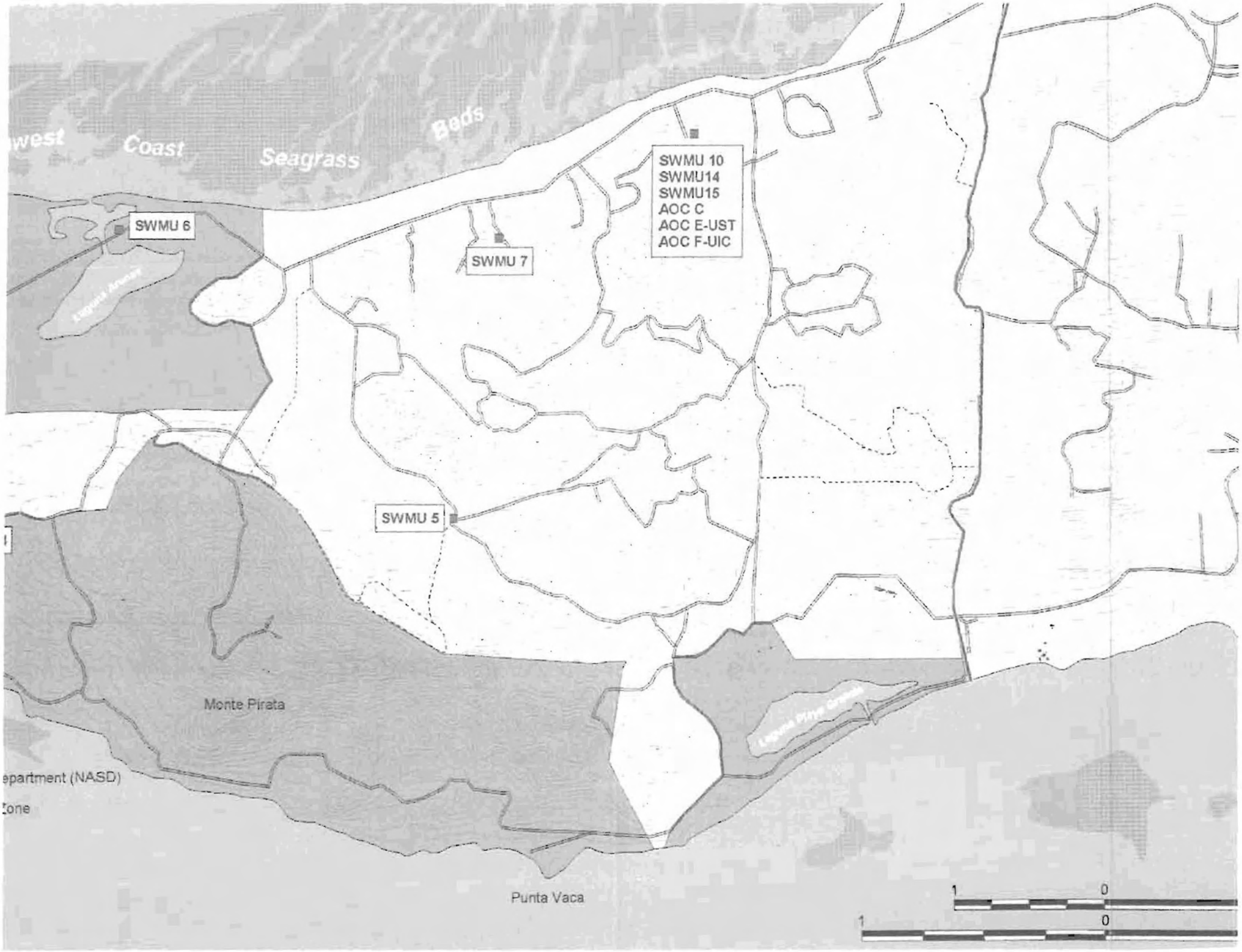
Figure 1. NASD Location.


west Coast Seagrass Beds
SWMU 6
SWMU 7
SWMU 10
SWMU14
SWMU15
AOC C
AOC E-UST
AOC F-UIC
SWMU 5
Monte Pirata
epartment (NASD)
Zone
Punta Vaca
1 0
1 0

4. identify habitat that may potentially support federally designated threatened and endangered species within and contiguous to each SWMU; and
5. identify any obvious impacts potentially related to previous waste management activities.

The vegetation communities were verified by walking surveys through each community type previously identified with aerial photography. Most species were identified in the field; however, some specimens were collected for identification using reference books (Liogier 1985, 1988, 1994, 1995, 1997; Little and Wadsworth 1964; Little et al. 1964; and Acevedo-Rodriguez 1996) and herbarium specimens. Relative dominance and species structure were characterized from the visual observations within each community type and SWMU.

Wildlife species residing within or utilizing each SWMU habitat, and wildlife habitat were identified during the vegetation field surveys. A wildlife biologist characterized the habitats and determined the types of wildlife that could potentially inhabit the plant communities or SWMU sites. Any wildlife species that were observed were identified in the field with the use of 8 x 40 binoculars and reference guides (Raffaele 1989 and Raffaele et al 1998).

Fourteen federally listed species are known to occur or have the potential to occur on NASD Vieques (Table 1). Prior to conducting the fieldwork, a literature search was conducted for each federally protected species. During the May 15-19 surveys, biologists walked transects through each site and identified any federally protected species seen and noted the presence or absence of preferred habitat for the species.

Table 1

Federally Listed Species Occurring or Potentially Occurring at NASD Vieques

***Scientific Name* (Common Name)**	**Federal Status**
Plants	
Chaemacrista glandulosa var. *mirabilis* (Herb)	Endangered
Stahlia monosperma (Cobana negra)	Threatened
Calyptranthes thomasiana (Tree)	Endangered
Eugenia woodburyana (Evergreen tree)	Endangered
Reptiles and Amphibians	
Chelonia mydas (Green sea turtle)	Threatened
Dermochelys coriacea (Leatherback sea turtle)	Endangered
Eretmochelys imbricata (Hawksbill sea turtle)	Endangered
Birds	
Falco peregrinus tundrius (Arctic peregrine)	Threatened
Pelecanus occidentalis occidentalis (Brown pelican)	Endangered
Sterna dougalli dougalli (Roseate tern)	Endangered
Mammals	
Physeter macrocephalus (Sperm whale)	Endangered
Balaenoptera physalus (Fin whale)	Endangered
Megaptera novaeangliae (Humpback whale)	Endangered
Trichechas manatus (West Indian manatee)	Endangered

Source: NASD 1996

Past management activities at the SWMU sites may have potentially impacted the current vegetation communities. During the field surveys the biologists made visual observations to characterize the health of the plants in the SWMU sites. Indications of altered plant communities include: chlorotic leaves, epinasty (deformities of leaves and stems), patches of altered plant growth, absence of plants (bare ground), and changes in species composition. To determine if the SWMU sites contained altered plant communities, a nearby representative site was selected as a control. When altered plant communities were identified, the biologists made an effort to determine and record the probable cause (i.e., chemical, soil compaction, natural causes, etc.).

In addition to identification of wildlife in the field, existing literature sources were used to identify any additional species that may have occurred on the SWMU sites but were not observed. Most of the wildlife occurring in the area is bird species and these are presented in Appendix A. Species information and field data was used to generate a simplified food web for the sites. A food web is an interlocking pattern of several to many food chains that is helpful in determining ecosystem processes including those that may occur when a contaminant is introduced to a system.

RESULTS AND DISCUSSION

SWMU 4

Vegetation Community Description

SWMU 4, an inactive Waste Explosive Open Burn/Detonation Range, is approximately 3 acres in size and is located on the western side of NASD. The area is located on a hill that is completely surrounded by dirt roads (Figure 3). A high density of thorny shrubs dominates species composition on the site. Many areas of the site had both a shrub and a herbaceous stratum. With the two strata combined, the canopy cover was approximately 75-95 percent. Dominant shrubs identified on the site included *Acacia farnenciana, Prosopis glandulosa, Pithicellobium dulce,* and *Zanthoxylum brevipes*. Another shrub that was present throughout the site and was dominant in some areas was *Leucaena leucocephala*. In certain areas there was an herbaceous stratum that was dominated by *Bothriochloa ischaemum, Commelina erecta, C. diffusa,* and *Lasiacis divaricata*. Vegetation photos are presented in Figures 4 and 5. The vegetation observed at SWMU 4 is presented in Table 2.

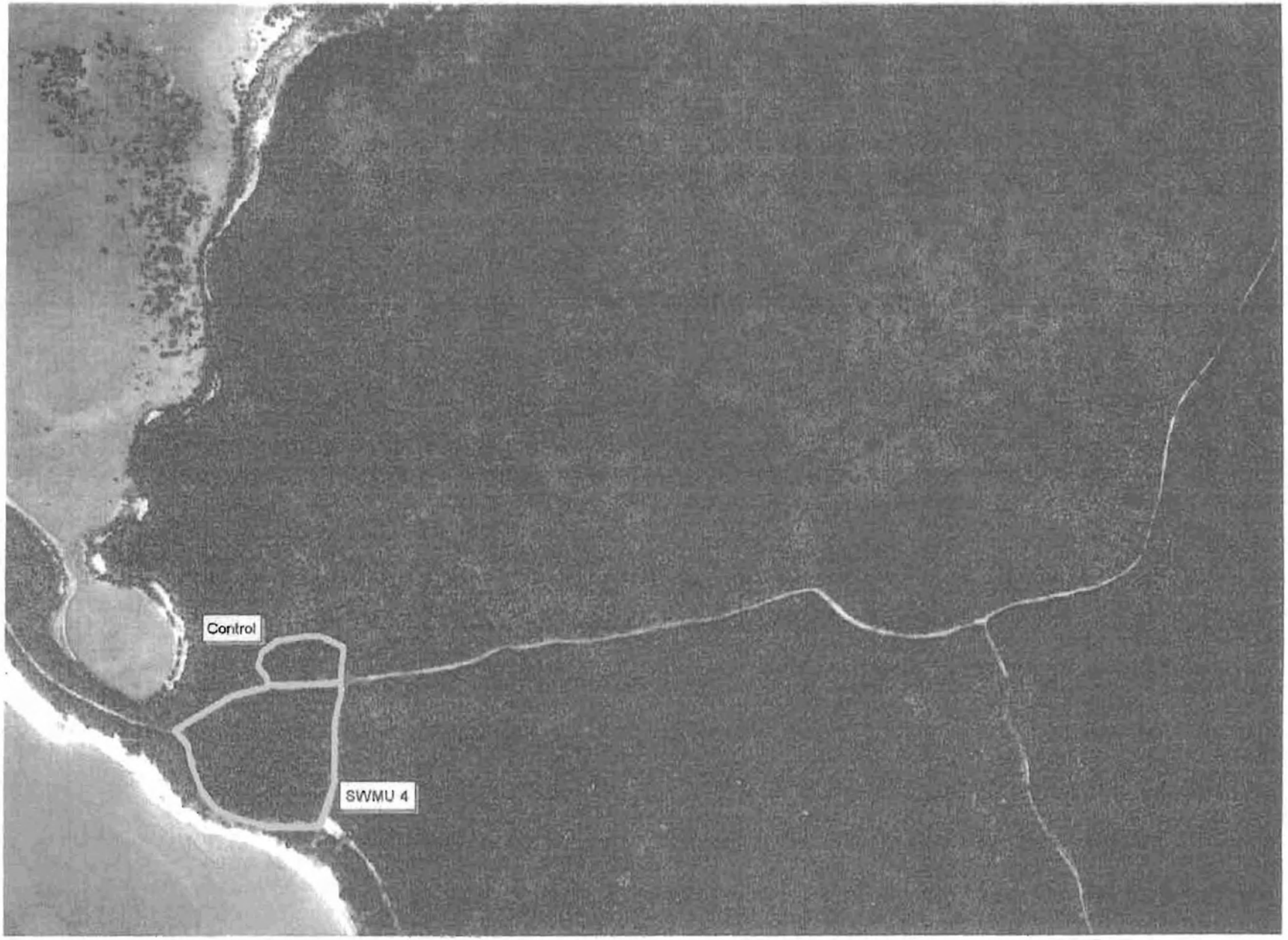


Figure 3. Location of SWMU 4 and control site at NASD Vieques Island, Puerto Rico.

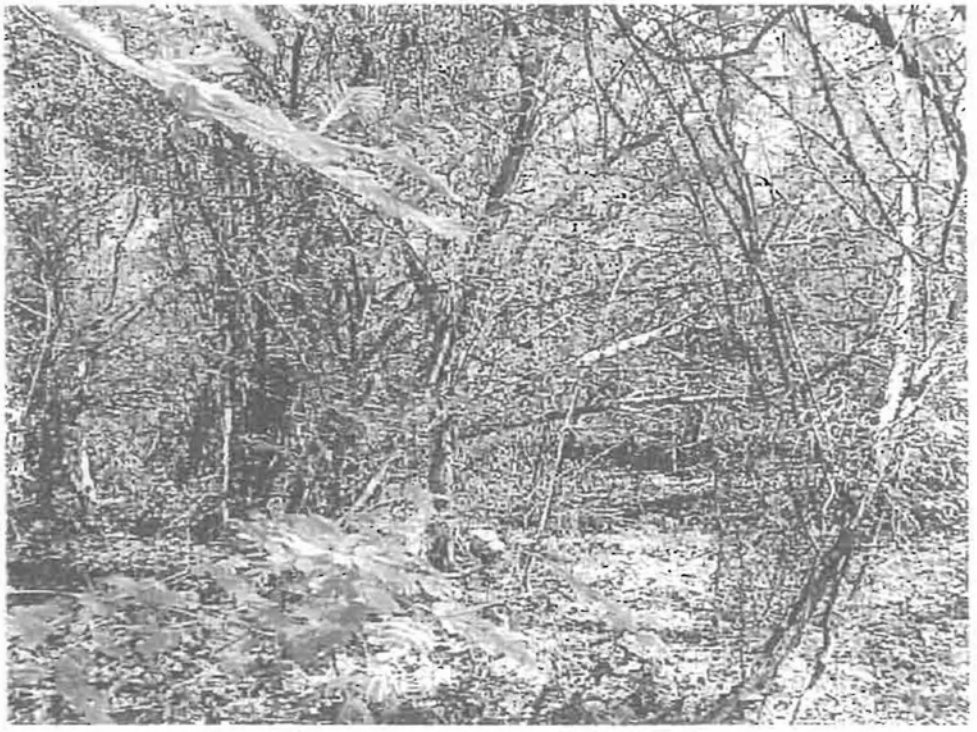


Figure 4. SWMU 4, typical vegetation at the site.

Figure 5. SWMU 4, typical scrub at the edge of the site

Table 2

Vegetation Observed at SWMU 4

Common Name	Scientific Name	Stratum
Aroma, sweet acacia	*Acacia farnenciana*	S
None	*Acalypha poiretti*	H
Kitty katies, Tibet tree	*Albizia lebbek*	T
Bastard mahogany	*Andira inermis*	T
None	*Bothriochloa ischaemum*	H
Turpentine tree, almácigo	*Bursera simaruba*	T
Papaya	*Carica papaya*	S
Palo Blanco	*Casearia gianensis*	T
Trumpet tree	*Cecropia shreberiana*	T
Wist vine	*Centrosema virginianum*	V
Lime bush	*Citrus aurantifolia*	S
None	*Citrus lima*	T
French grass	*Commelina* spp.	H
Purple allamanda	*Cryptostegia grandiflora*	S
Brazilette, brizzlet, wild cherry	*Erythroxylum brevipes*	S
Wild coffee	*Faramea occidentalis*	S
Bastard cedar	*Guazuma ulmifolia*	T
Sea pusley	*Heliotropium curassavicum*	H
None	*Ipomea* sp.	V
Jazmin oloroso	*Jasminum fluminense*	V
Belly ache bush	*Jatropha gossypifolia*	S
Belly ache bush, body catta	*Jatropha gossypifolia*	S
None	*Lasiacis divaricata*	H
Rabbit food, hollow stalk	*Leonotis nepetifolia*	H
Tan tan, tanty, wild tamarind	*Leucaena leucocephala*	S
Beach milk vine	*Matelea maritime*	V
Cundeamor, jumbie pumpkin	*Momordica charantia*	V
None	*Oceoclades maculata*	H
Passion fruit, parcha	*Passiflora* sp.	V
Guamá americano, guamuchil	*Pithicellobium dulce*	S
Mesquite	*Prosopis glandulosa*	S
Christmas tree, tintillo	*Randia aculeata*	S
None	*Rhynchosia reticulata*	V
None	*Stigmaphyllon periplocifolium*	V
Tamarind	*Tamarindus indica*	T
Tamarind	*Tamarindus indica*	T
Basket wiss, hoop vine	*Trichostygma octandrum*	S
Yerba socialista, socialist herb	*Vernonia cinera*	H
Yellow-sanders, aceitillo	*Zanthoxylum brevipes*	S
Yuyubi	*Ziziphus mauritiana*	T

S = shrub
T = tree
H = herbaceous
V = vine

Plant Community Health

Although some debris was found at SWMU 4, there was little evidence of past military activities. There were some indications from openings in the plant communities and from worn trails that horses (*Equus caballus*) occasionally graze throughout the site. No other plant stresses were observed in SWMU 4. The control site for SWMU 4 was located adjacent to the northern boundary of the SWMU 4 site. Canopy openings and trails made by horses were also observed in the control.

Wildlife Description

During the short duration of wildlife surveys conducted on this site, numerous wildlife species were observed utilizing the habitat of this site. Although no individual horses were observed within the site, there was sufficient evidence to indicate that horses use this habitat. The bird species observed at this habitat type consisted of coastal forest and shore species due to the close proximity to ocean. Numerous lizards (*Anolis* species) were also identified. There was no evidence that the SWMU site had an impact on the wildlife diversity or its habitat. Wildlife that was observed at SWMU 4 is presented in Table 3.

Table 3
Wildlife Observed at SWMU 4

English Name	Scientific Name	Local Name
Mammals		
Horse	*Equus caballus*	Horse, caballo
Mongoose	*Herpestes auropunctatus*	Not known
Reptiles and Amphibians		
Lizards	*Anolis* sp.	Not known
Birds		
Bananaquit	*Coereba flaveola*	Reinita Común
Common-ground Dove	*Columbina passerina*	Rolita
Smooth-billed Ani	*Crotophaga ani*	Garrapatero (Judio)
Adelaidae Warbler	*Dendroica adelaidae*	Reinita Mariposera
Yellow Warbler	*Dendroica petechia*	Canario de Mangle
Pearly-eyed Thrasher	*Margarops fuscatus*	Zorzal Pardo
Greater Antillean Grackle	*Quiscalus niger*	Mozambique (Chango)
Puerto Rican Lizard Cuckoo	*Saurothera vieilloti*	Pájaro Bobo Mayor
Loggerhead Kingbird	*Tyrannus caudifasciatus*	Clérigo
Gray Kingbird	*Tyrannus dominicensis*	Pitirre

Protected Species

Stahlia monosperma (Cobana negra), a federally threatened tree, has been found between the boundary of black mangrove (*Avicennia germinans*) communities, salt flats and the upland communities at NASD Vieques. This species is also known to occur in coastal forests of southeastern Puerto Rico (Little and Wadsworth 1964). The preferred habitat for *Stahlia monosperma* was not present at this site.

Federally threatened and endangered sea turtles such as the Green (*Chelonia mydas*), Hawksbill (*Eretmochelys imbricata*), and Leatherback sea turtles (*Dermochelys coriacea*), and endangered marine mammals such as the West Indian manatee (*Trichechas manatus*), Sperm whale (*Physeter macrocephalus*), Fin whale (*Balaenoptera physalus*), and Humpback whale (*Megaptera novaeangliae*) would not occur at this site because they require marine habitats.

The Artic peregrine falcon (*Falco peregrinus tundrius*) has been observed nearby at NAVSTA Roosevelt Roads (U.S. Navy 1998b). This species utilizes open grassland areas for potential feeding areas. This type of habitat was not present at or near this site.

Federally endangered marine birds such as the Brown pelican (*Pelecanus occidentalis occidentalis*) and the Roseate tern (*Sterna dougalli dougallii*) would most likely not occur at this terrestrial site. During these surveys, Brown pelicans were observed flying over the adjacent marine habitat, but not at SWMU 4.

Food Web

The information in a food web is very important when considering the potential for contaminants existing in the ecosystem. Many contaminants are passed from one trophic level to the next. A contaminant at the soil surface goes through a different process than a contaminant that has leached into the soil. The surface contaminant may be ingested by a decomposer such as a hermit crab and then passed on to the secondary consumer (i.e., a carnivorous bird). Leached contaminates are picked up by the primary producers and are then passed upwards in the food chain.

Figure 6 presents a generalized food web for the forested and herbaceous community types found at all SWMU sites except SWMU 6. The abundance within each of the food groups is represented by the size of their polygon in the figure. Dominant species are listed in each of the food groups except for plants, which were provided previously in this section.

SWMU 5

Vegetation Community Description

SWMU 5, IRFNA/MAF-4 Disposal Site, is located adjacent to magazine Building 422 and consists of a small ditch and ravine (Figure 7). There were two plant communities present at this site – the maintained grasslands of the small ditch and the non-maintained large ravine (quebrada). This ravine occurs as a long depression that was created during the construction of a road that leads to additional magazine buildings (243-249). The elevated road was blocking the natural water flow and was creating a hydrophytic plant community.

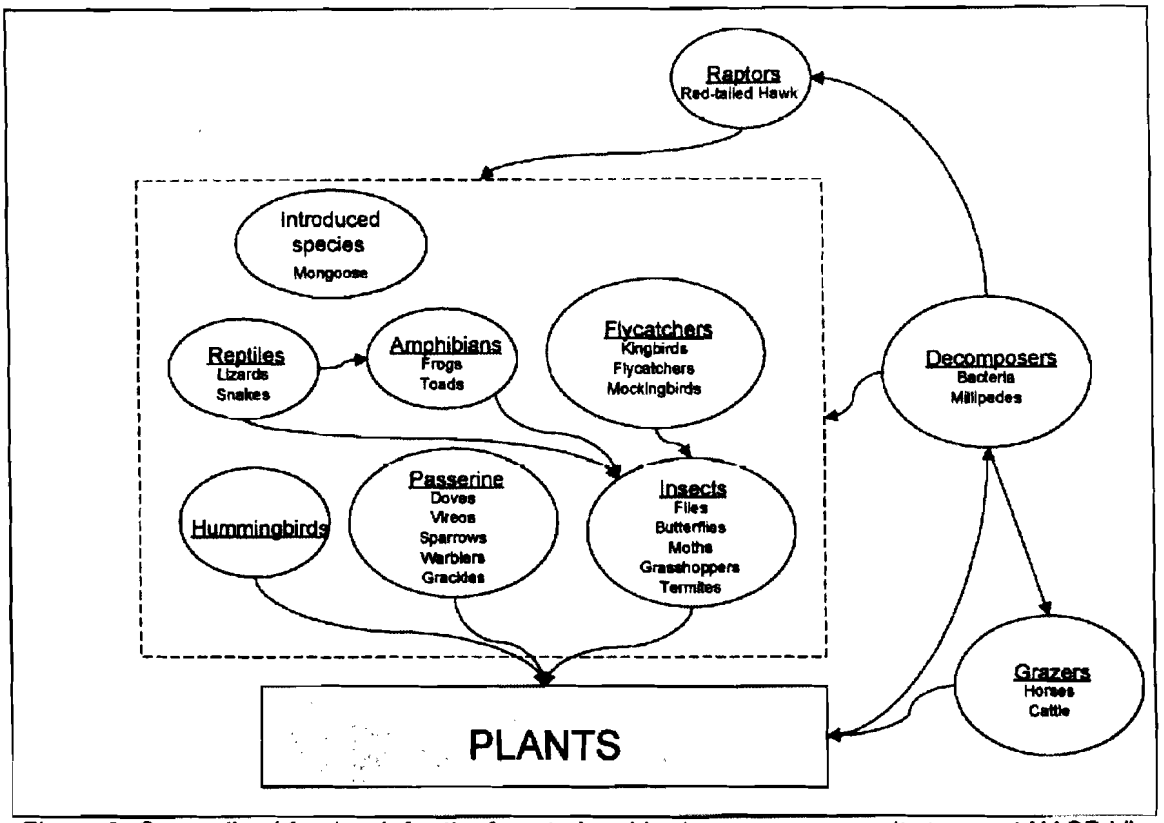


Figure 6. Generalized food web for the forested and herbaceous community types at NASD Vieques.

The non-maintained area consisting of a patchy canopy of various trees, shrubs, and herbaceous ground cover had 100 percent cover. *Eriochloa polystachya, Leonotis nepetifolia, Panicum maximum,* and *Commelina erecta* dominated the herbaceous canopy. Shrubs were mostly found along the hillside edges; these included *Randia aculeata, Erythroxylum brevipes, Acacia farnenciana,* and *Leucaena leucocephala.* There were a few trees (*Bursera simaruba* and *Tamarindus indica*) found throughout the site.

The small drainage ditch is located within the area where regular grounds maintenance occurs around Magazine Building 422. A regular disturbance by mowing has created a low-growing herbaceous community. Grasses dominated this drainage ditch and included *Bothriochloa ischaemum, Sporobolus indicus, Cynodon dactylon,* and *Scleria* sp. Vegetation photos are presented in Figures 8 and 9. The vegetation observed at SWMU 5 is presented in Table 4

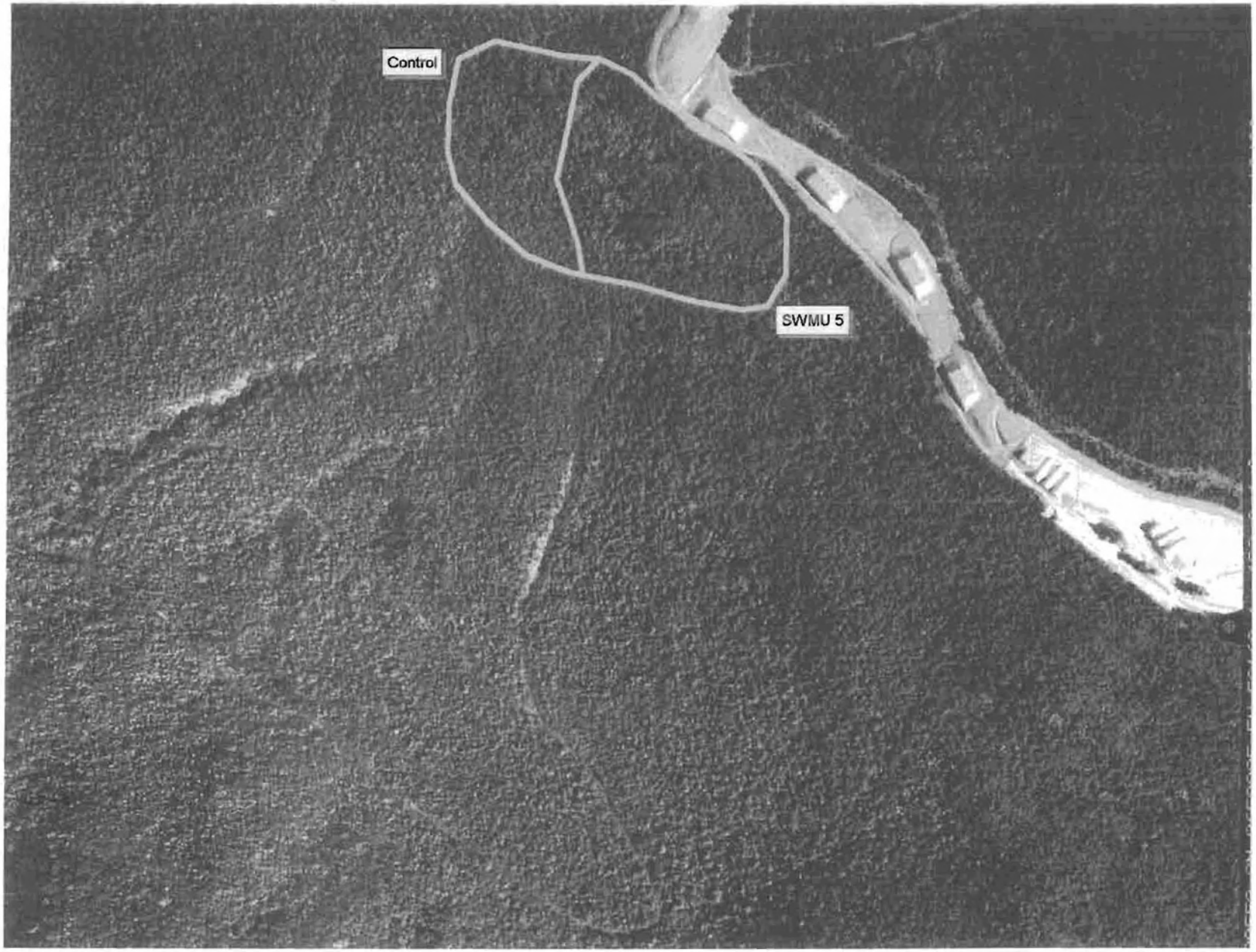


Figure 7. Location of SWMU 5 and control site at NASD Vieques Island, Puerto Rico.

Figure 8. SWMU 5, maintained area.

Figure 9. SWMU 5, shrubby area within site.

Table 4

Vegetation Observed at SWMU 5

Common Name	Scientific Name	Stratum
Aroma, sweet acacia	*Acacia farnenciana*	S
Better man better	*Achyranthes aspera*	H
Bastard mahogany, dog almond	*Andira inermis*	T
Cralita, coral vine, Mexican creeper	*Antigonon leptopus*	V
None	*Bothriochloa ischaemum*	H
Almácigo, turpentine-tree	*Bursera simaruba*	T
Pataka	*Cissampelos pareira*	No data
Palo blanco	*Casearia gianensis*	No data
Cafeillo Cimarron	*Casearia sylvestris*	No data
Wist vine	*Centrosema virginianum*	V
French grass	*Commelina erecta*	H
Bermuda grass	*Cynodon dactylon*	H
None	*Eriochloa polystachya*	H
Brazilette, brizzlet, wild cherry	*Erythroxylum brevipes*	S
Jazmin oloroso	*Jasminum fluminense*	V
Sage, cariaquillo	*Lantana* sp.	S
Quinino del pasto, lion's ear	*Leonotis nepetifolia*	H
Tan tan, tanty, wild tamarind	*Leucaena leucocephala*	S
Cundeamor	*Momordica charantia*	V
None	*Panicum maximum*	H
Parcha, passion fruit	*Passiflora edulis*	V
Indigo berry, ink berry	*Passiflora suberosa*	V
Christmas tree, tintillo, ink berry	*Randia aculeata*	S
Licorice, rain tree	*Samanea saman*	T
None	*Scleria* sp.	H
White button	*Spermacocce verticilata*	H
None	*Sporobolus indicus*	H
Chichery grape	*Tournefortia hirsutissima*	V
Tamarind	*Tamarindus indica*	T
Baske wiss	*Trichostigma octrandrum*	S,V
Aprín, India jujube	*Ziziphus mauritiana*	T

S = shrub
T = tree
H = herbaceous
V = vine

Plant Community Health

It was difficult to select a control for this community due to the effects of anthropomorphic disturbances (previous road construction) on the existing topography. The selected control was split into two areas so that an adequate comparison of the site could be accomplished. The non-maintained control area was adjacent to the southern border of the SWMU 5 site. The maintained control area was adjacent to the northern border of the maintained portion of SWMU 5. There were no differences identified in the vegetation communities with the exception of the drainage ditch that had some soil disturbance from recent soil removal during which some of the vegetation was trampled or cut.

Two dead animals were found near the site. One of the remains was the skeleton of a bovine species that died some time ago. The other animal was a horse that looked to have died more recently (less than five

days old). The cause of mortality of these animals is unknown, but they probably died due to natural causes and their presence was most likely coincidental.

Wildlife Description

During the short duration of wildlife surveys conducted on this site, numerous wildlife species were observed utilizing the habitat of this site. Although no horses were observed within the site, there was sufficient evidence to indicate that they frequent this habitat. The bird species observed at this habitat type consisted of coastal forest species (Table 5). Numerous lizards were also identified at this site. There was no evidence that the SWMU site had an impact on the wildlife or its habitat.

Table 5
Wildlife Observed at SWMU 5

English Name	Scientific Name	Local Name
Mammals		
Horse	*Equus caballus*	Horse, caballo
Mongoose	*Herpestes auropunctatus*	Not known
Reptiles and Amphibians		
Lizards	*Anolis sp.*	Not known
Birds		
Bananaquit	*Coereba flaveola*	Reinita Común
White-crowned Dove	*Columba leucocephala*	Paloma Cabeciblanca
Adelaidae Warbler	*Dendroica adelaidae*	Reinita Mariposera
Puerto Rican Lizard Cuckoo	*Saurothera vieilloti*	Pájaro Bobo Mayor
Gray Kingbird	*Tyrannus dominicensis*	Pitirre
Zenaida Dove	*Zenaida aurita*	Tórtola Cardosantera

Protected Species

There were no federally protected species or preferred habitat observed at this site. Terrestrial forested plant community dominated the sites and is not preferred habitat for any of the species.

Food Web

Figure 6 presents a generalized food web for the forested community type found at this SWMU site.

SWMU 6

Vegetation Community Description

SWMU 6, Mangrove Disposal Site, consists of black and red mangrove (*Rhizophora mangle*) communities located between Laguna Kiani and the Caribbean Sea (Figure 10). These communities were separated by position within the landscape. The red mangroves were located near open water where there is saturation/inundation of seawater while the black mangroves were located on higher land.

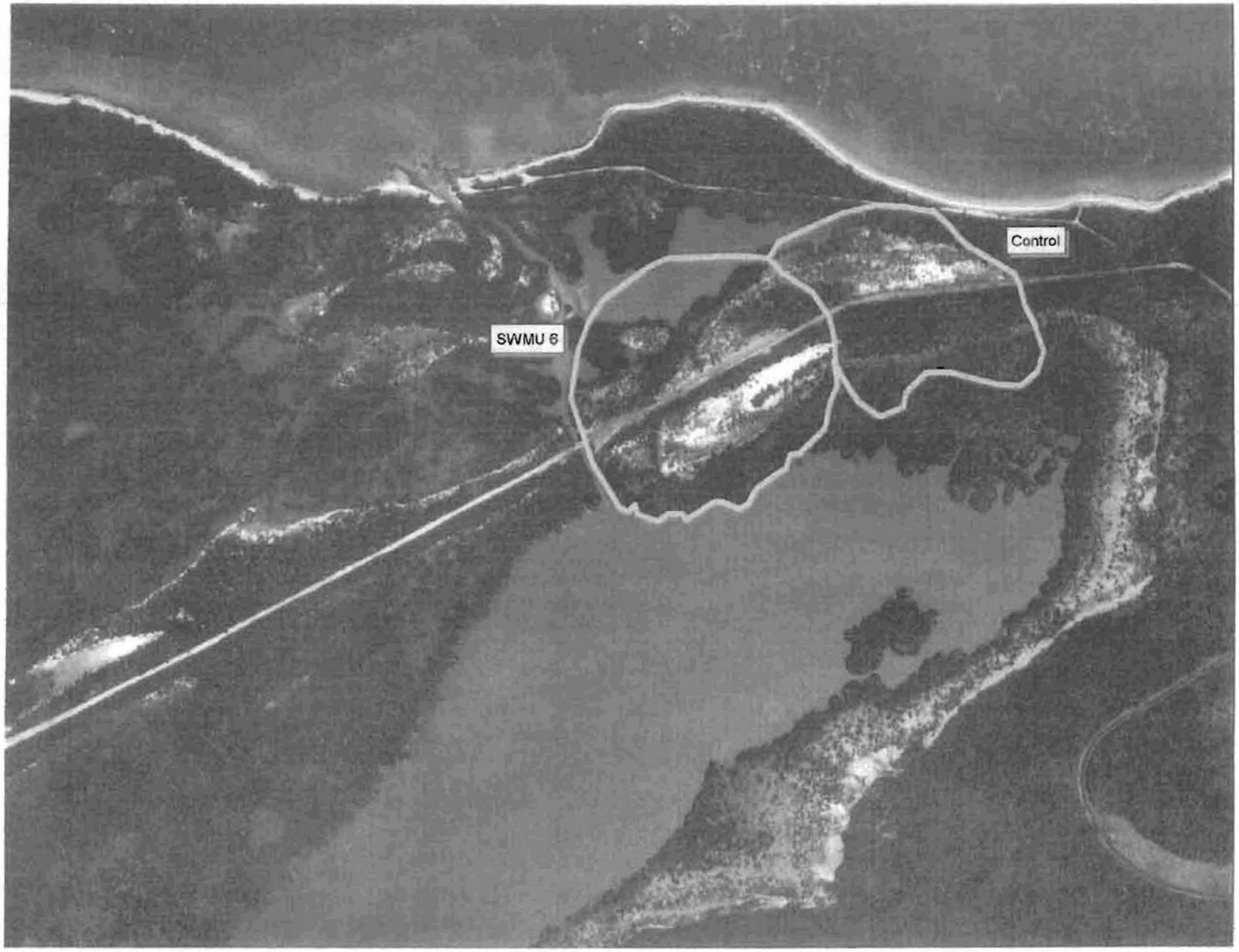


Figure 10. Location of SWMU 6 and control site at NASD Vieques Island, Puerto Rico.

The red mangrove community was sparsely vegetated (approximately 25 percent cover) with large pools of open water. Nearly all vegetation included short shrubs of red mangrove; no other vegetation was observed in this community. Numerous red mangrove seedlings were also observed in this site.

The black mangrove community also had sparse vegetation cover; there was approximately 25 percent cover. Plants were predominately short shrubs (8-15 feet); however, there were some patches of herbaceous vegetation located on higher topography. Black mangrove dominated the shrub vegetation, with *Laguncularia racemosa*, *Prosopis glandulosa*, and *Acacia farnesiana* also present. The herbaceous vegetation was dominated by *Sporobolus virginicus*. *Heliotropium curassavicum*, *Sesuvium portulacastrum*, and *Blutaparon vermiculare* were also identified. Vegetation photos are presented in Figures 11 and 12. The vegetation observed at SWMU 6 is presented in Table 6.

Plant Community Health

A control site was selected along the eastern boundary of the SWMU 6 site for plant community comparisons. These sites (control vs. SWMU 6) were essentially the same. The only difference was that the open areas of the black mangrove community in SWMU 6 were covered in debris. There were no observed stresses in the plant communities.

Wildlife Description

During the short duration of wildlife surveys conducted on this site, numerous wildlife species such as birds and lizards were observed utilizing the habitat of this site (Table 7). The mangrove communities had significant crab activity. The red mangrove community, with more water present, had more crab holes than the black mangrove. There was no evidence that the SWMU site had an impact on the wildlife or habitat.

Figure 11. SWMU 6, typical vegetation, mangroves

Figure 12. SWMU 6, typical vegetation, drier area.

Table 6

Vegetation Observed at SWMU 6

Common Name	Scientific Name	Stratum
Aroma, sweet acacia	*Acacia farnesiana*	S
Black mangrove	*Avicennia germinans*	S
Bay flower	*Blutaparon vermiculare*	H
None	*Croton discolor*	S
Purple allamanda	*Cryptostegia grandiflora*	S
None	*Heliotropium curassavicum*	H
None	*Jacquinia arborea*	S
None	*Jasminum fluminense*	V
Belly ache bush, body catta	*Jatropha gossypifolia*	S
White mangrove	*Laguncularia racemosa*	S
Sage, cariaquillo	*Lantana* sp.	S
None	*Mikania cordifolia*	V
Mesquite	*Prosopis glandulosa*	S
Tintillo, Christmas tree	*Randia aculeata*	S
Red mangrove	*Rhizophora mangle*	S
Bay flower, sea purslane, sea pusley	*Sesuvium portulacastrum*	H
None	*Sporobolus virginicus*	H
None	*Stigmaphyllon periplocifolium*	V
White cedar, pink cedar	*Tabebuia heterophylla*	T
Emajaguilla, portiatree	*Thespesia populnea*	T

S = shrub
T = tree
H = herbaceous
V = vine

Table 7
Wildlife Observed at SWMU 6

English Name	Scientific Name	Local Name
Reptiles and Amphibians		
Lizards	*Anolis* sp.	Not known
Birds		
Green Heron	*Butorides virescens*	Martinete
Bananaquit	*Coereba flaveola*	Reinita Común
White-crowned Dove	*Columba leucocephala*	Paloma Cabeciblanca
Yellow Warbler	*Dendroica petechia*	Canario de Mangle
Common Moorhen	*Gallinula chloropus*	Gallareta Común
Pearly-eyed Thrasher	*Margarops fuscatus*	Zorzal Pardo
Zenaida Dove	*Zenaida aurita*	Tórtola Cardosantera

Protected Species

There were no federally protected species or preferred habitat observed at this site which was dominated by black and red mangrove communities.

The federally endangered tree *Stahlia monosperma* is known to occur in coastal forests of southeastern Puerto Rico (Little and Wadsworth 1964). One of the two known *Stahlia monosperma* populations is

located on the eastern boundary of Laguna Kiani which is fairly close to SWMU 6. No individuals of *Stahlia monosperma* were found at SWMU 6. Although *Stahlia monosperma* has been found at NASD Vieques (in the boundary between black mangrove communities, salt flats and the upland communities), the habitat at SWMU is a mixed mangrove and therefore not preferred habitat.

Brown pelicans and roseate terns, both federally endangered marine birds, would most likely not occur at the site, but have been found adjacent to the site at Laguna Kiani. During the surveys, brown pelicans were observed flying over the adjacent marine habitat, but not at SWMU 7.

Food Web

Figure 13 presents a generalized food web for the mangrove community type found at SWMU 6. The abundance within each of the food groups is represented by the size of their polygon in the figure. Dominant species are listed in each of the food groups except for plants, which were provided previously in this section.

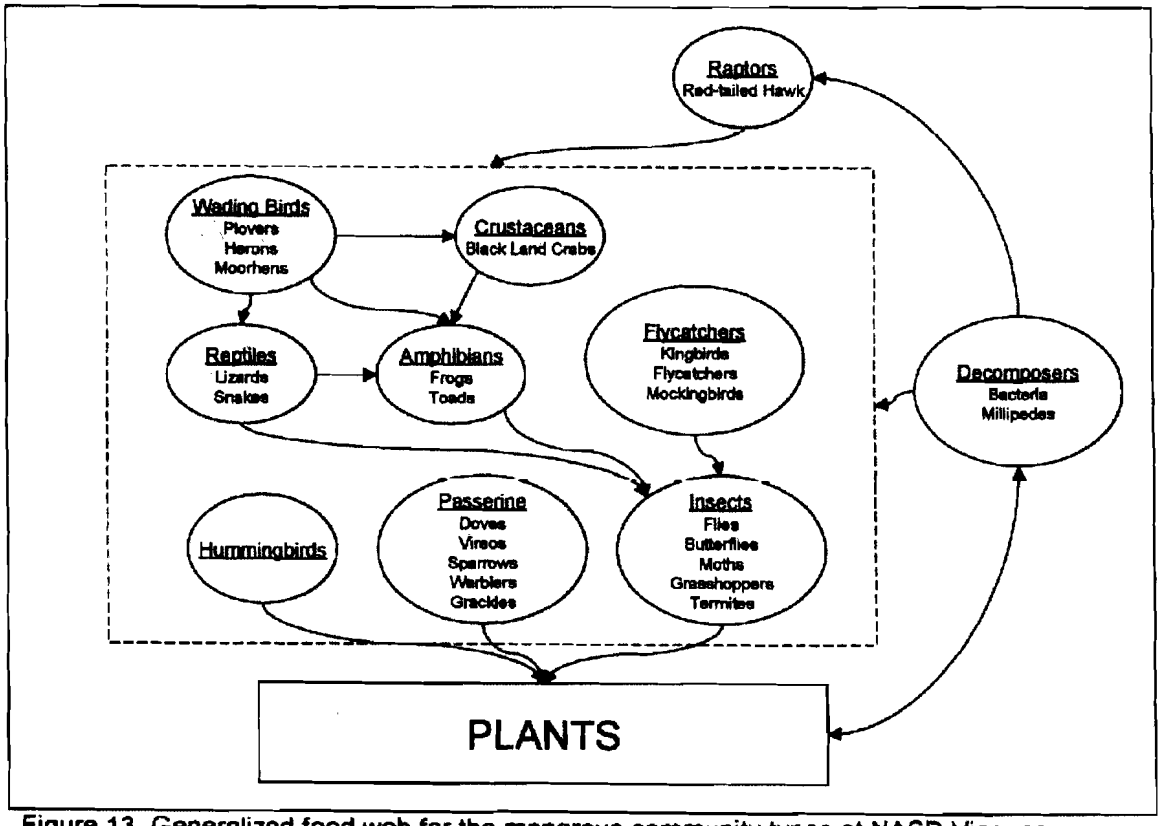


Figure 13. Generalized food web for the mangrove community types at NASD Vieques.

SWMU 7

Vegetation Community Description

SWMU 7, Quebrada Disposal Site, is located within a ravine approximately 0.5 miles west of the NASD Public Works area (Figure 14). This site had historically been used as a trash dump, but had been abandoned long enough for a shrub plant community to establish. This plant community had two canopy layers (shrub and tree), which provided nearly 100 percent canopy cover. The dense shrub and tree canopies provided little sunlight for an herbaceous canopy. *Lasiacis divaricata* was present in some areas. The dominant shrubs in this community consisted of *Leucaena leucocephala*, *Foresteria eggersiana*, and *Acacia retusa*. There was a lower density of trees than shrubs, but dominant tree species such as *Andira inermis* and *Cordia collococca* provided a significant contribution to the plant community. Vegetation photos are presented in Figures 15 and 16. The vegetation observed at SWMU 7 is presented in Table 8.

Table 8

Vegetation Observed at SWMU 7

Common Name	Scientific Name	Stratum
Catch and keep, white police	*Acacia retusa*	S
None	*Acacia westiana*	S
Bastard mahogany	*Andira inermis*	T
Palo blanco	*Casearia gianensis*	T
Pataka	*Cissampelos pareira*	V
Manjack, red manjack	*Cordia collococca*	T
Brazilette, brizzlet	*Erythroxylum brevipes*	S
Rocio, brizzlet	*Erthroxylum rotundifolium*	S
None	*Foresteria eggersiana*	S
Guaba,	*Inga vera*	T
None	*Ipomea* sp.	V
None	*Lasiacis divaricata*	H
Quinino del pasto, lion's ear	*Leonotis nepetifolia*	H
Zarcilla, tan tan, wild tamarind	*Leucaena leucocephala*	S
Congo root	*Petiveria alliacea*	SS
Black wattle	*Piper amalago*	S
Prickly mampoo	*Pisonia aculeata*	V/S
Christmas tree, tintillo	*Randia aculeata*	S
Cat's blood	*Rivina humilis*	H,S
None	*Securidaca virgata*	S
Basket wiss	*Serjania polyphylla*	V
Black hoopwood	*Trichostigma octrandrum*	T
Bran nettle, creeping cowitch	*Tragia volubilis*	H
Aceitillo, yellow-sanders	*Zanthoxylum monophyllum*	S
Yuyubi	*Ziziphus mauritiana*	T

S = shrub
T = tree
H = herbaceous
V = vine

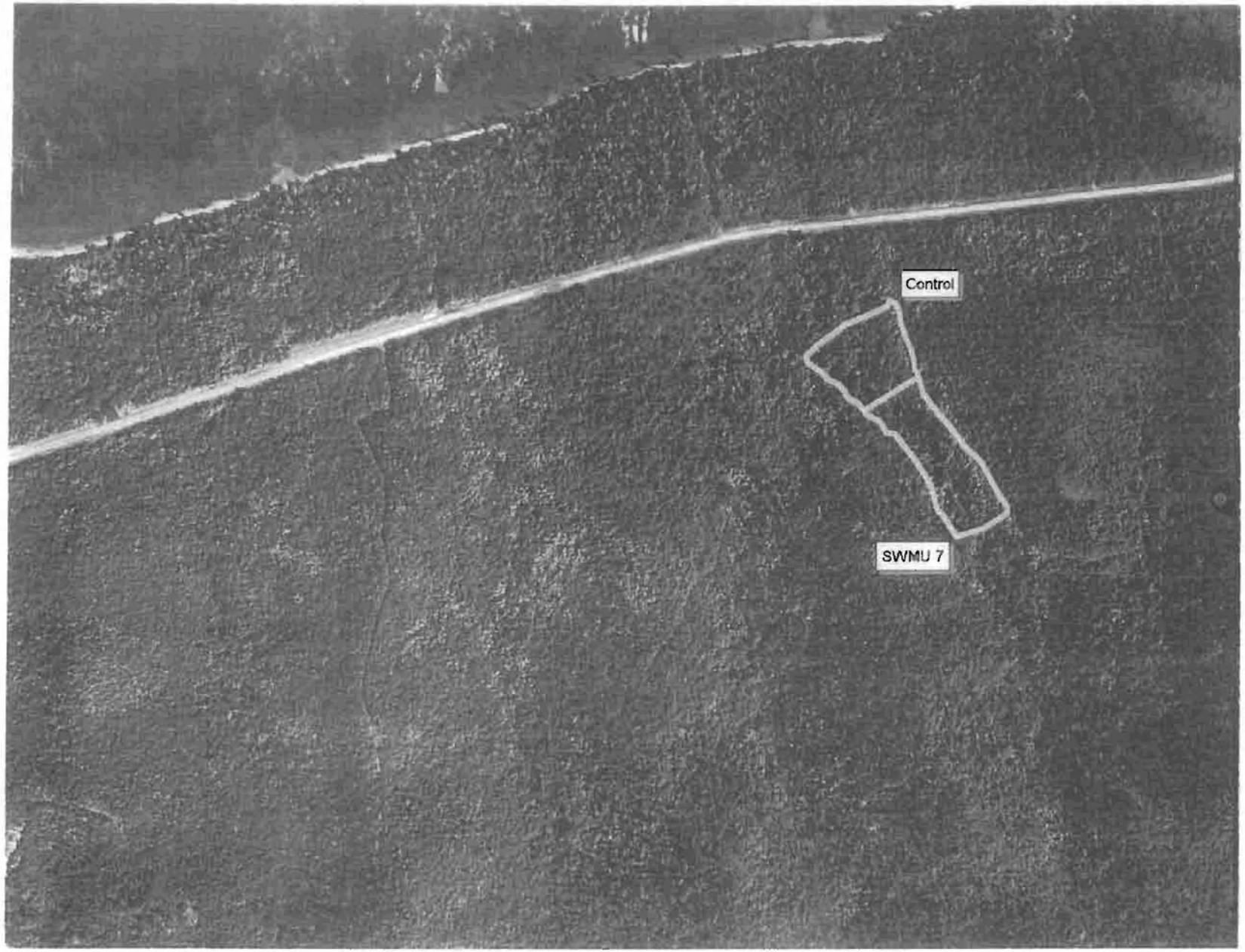


Figure 14. Location of SWMU 7 and control site at NASD Vieques Island, Puerto Rico.

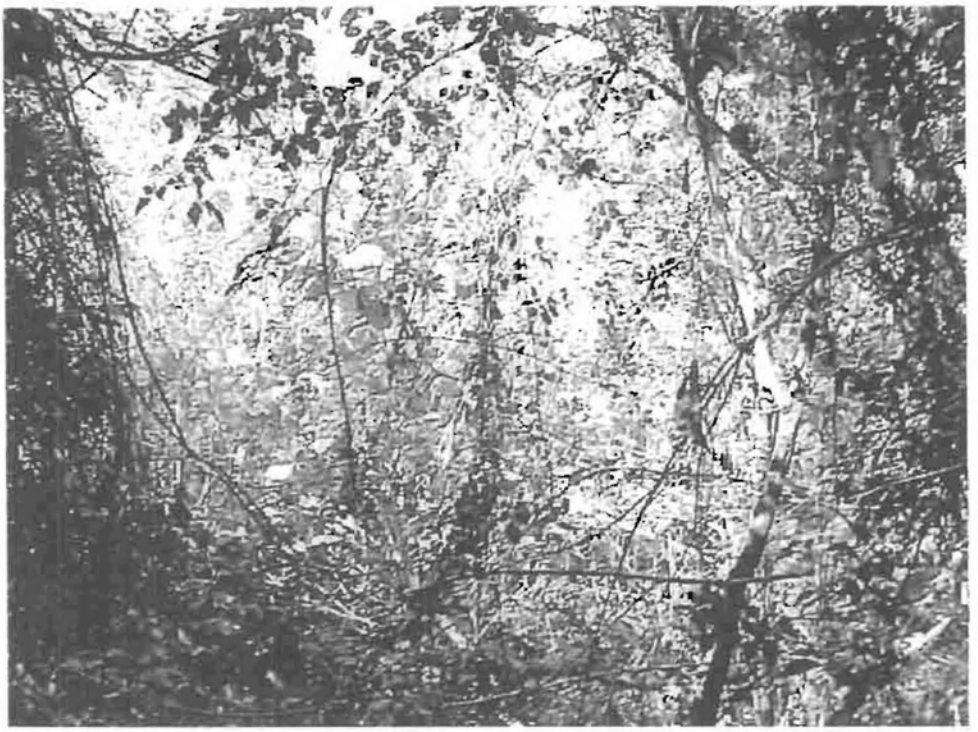


Figure 15. SWMU 7, typical vegetation

Figure 16. SWMU 7, vegetation located downstream

Plant Community Health

Despite a large amount of debris at the site, the plant community was very similar to the control. The control was located downstream of the site along the same ravine. The species composition and structure was similar between the two sites (control and SWMU 7). Additionally, no vegetation stresses were observed in SWMU 7.

Wildlife Description

During the short duration of wildlife surveys conducted on this site, numerous wildlife species were observed utilizing the habitat of this site. The bird species observed at this habitat type consisted of coastal forest and shore species due to the close proximity to Caribbean Sea (Table 9). Numerous lizards were also identified at this site. There was no evidence that the SWMU site had an impact on the wildlife or its habitat.

Table 9

Wildlife Observed at SWMU 7

English Name	Scientific Name	Local Name
Reptiles and Amphibians		
Lizards	*Anolis* sp.	Not known
Birds		
Red-tailed Hawk	*Buteo jamaicensis*	Guaraguao de Cola Roja
Bananaquit	*Coereba flaveola*	Reinita Común
Adelaidae Warbler	*Dendroica adelaidae*	Reinita Mariposera
Green-throated Carib	*Eulampis holosericeus*	Zumbador de Pecho Azul
Pearly-eyed Thrasher	*Margarops fuscatus*	Zorzal Pardo
Northern Mockingbird	*Mimus polyglottos*	Ruiseñor
Puerto Rican Lizard Cuckoo	*Saurothera vieilloti*	Pájaro Bobo Mayor
Louisiana Waterthrush	*Seiurus motacilla*	Pizpita de Rio
Loggerhead Kingbird	*Tyrannus caudifasciatus*	Clérigo
Gray Kingbird	*Tyrannus dominicensis*	Pitirre
White-winged Dove	*Zenaida asiatica*	Tórtola Aliblanca

Protected Species

There were no federally protected species or preferred habitat observed at this site. Terrestrial forested plant community dominated the sites, which is not preferred habitat for any of the species.

The federally endangered Brown pelican would most likely not occur at this terrestrial site. During the surveys Brown pelicans were observed flying over the adjacent marine habitat, but not at SWMU 7.

Food Web

Figure 6 presents a generalized food web for the forested community type found at this SWMU site.

AOC Sites, SWMU 10, SWMU 14, and SWMU 15

Vegetation Community Description

The SWMU 10, SWMU 14, SWMU 15, AOC C, AOC E-UST, and AOC F-UIC sites contained similar plant communities and were all located in the NASD Public Works area (Figure 17). The plant communities at these sites were dominated by herbaceous species due to grounds maintenance activities (mowing). A few shrubs with decumbent growth were present and there was approximately 70 to 85 percent ground cover at all the sites. The herbaceous plant community was dominated by several species, including: *Bothriochloa ishaemum, Digitaria ciliaris, Cynodon dactylon*, and *Commelina erecta*. Vegetation photos are presented in Figures 18 through 23. The vegetation observed at the NASP Public Works sites is presented in Table 10.

Plant Community Health

These sites have experienced regular disturbance from mowing that has resulted in a grassland community. Grounds maintenance is the primary factor affecting the species composition and structure of the sites. A control plot was selected within the Public Works area, southeast of SWMU 14.

There was no significant difference between any of the sites and the control. SWMU 10 had a greater abundance of *Digitaria ciliaris* and *Commelina erecta* than the control; however, this was most likely due to the shading effect of the associated buildings (both species are shade tolerant). AOC-C had a higher abundance of hydrophytic vegetation than the other sites because of the drainage ditches on the site. There was some surface disturbance (bare ground) at both AOC-E UST and AOC-F UIC due to the installation/maintenance of monitoring wells.

SWMU 15 was the only site that had evidence of stressed vegetation. There was a greater abundance of *Cynodon dactylon* growing at this site than other sites. *Cynodon dactylon* is an exotic and invasive grass species that can tolerate variable growing conditions. The presence of this species on any of these sites is not significant, due to the grounds maintenance. The decumbent growth form of this species indicates that this area had been compacted from heavy vehicles driving or parking on the site. The soil compaction may also limit the species diversity of the site. It appears that the site may have been a parking lot or vehicle staging area because some small patches of oil stained soil were observed in the south central portion of the site.

Wildlife Description

Wildlife observed at these sites is typical for developed areas on Vieques and is presented in Table 11. Two mammal species (horse and mongoose) were observed utilizing the sites. Numerous birds including

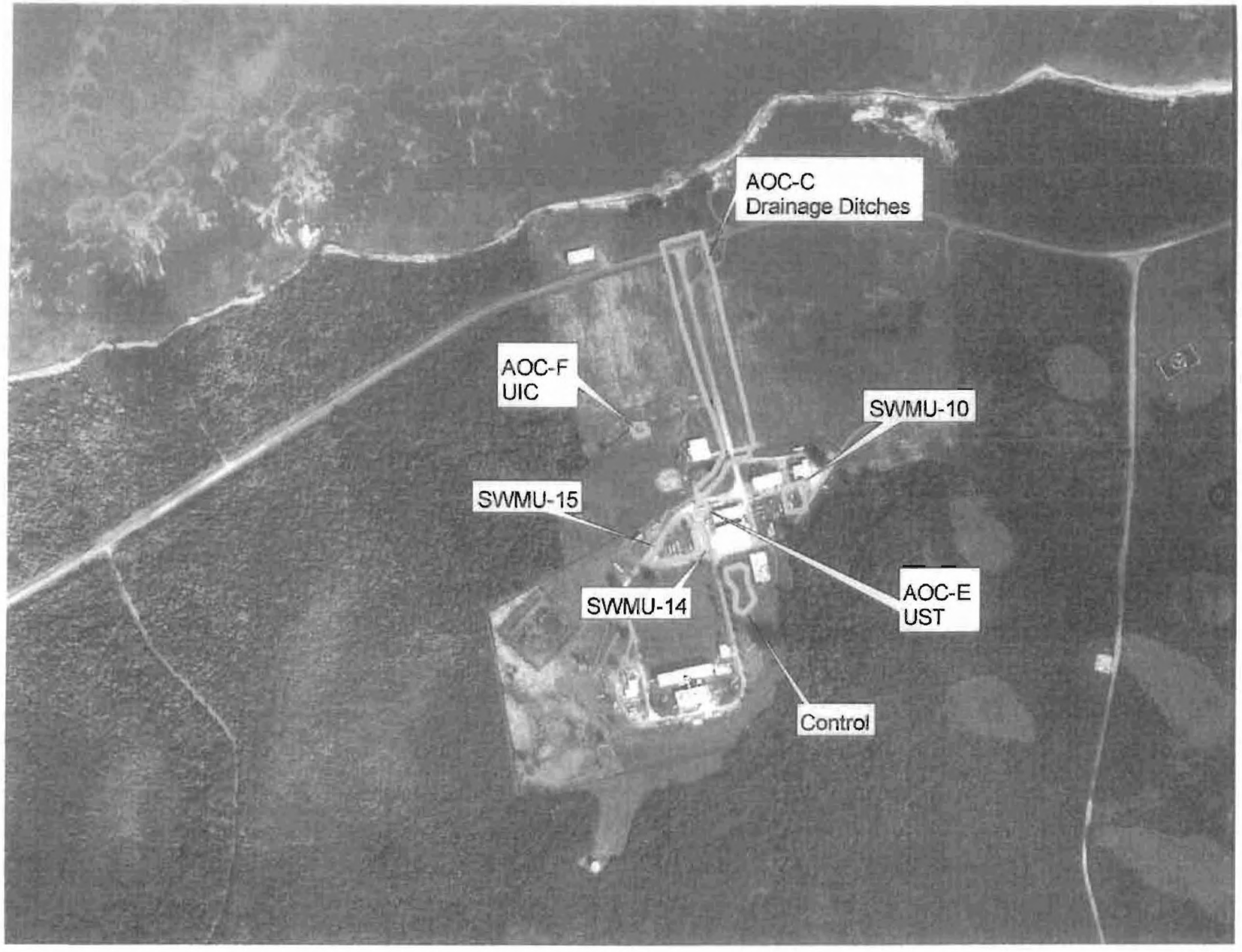


Figure 17. Location of SWMUs, AOCs, and control located in the NASD Public Works Area, Vieques Island, Puerto Rico.

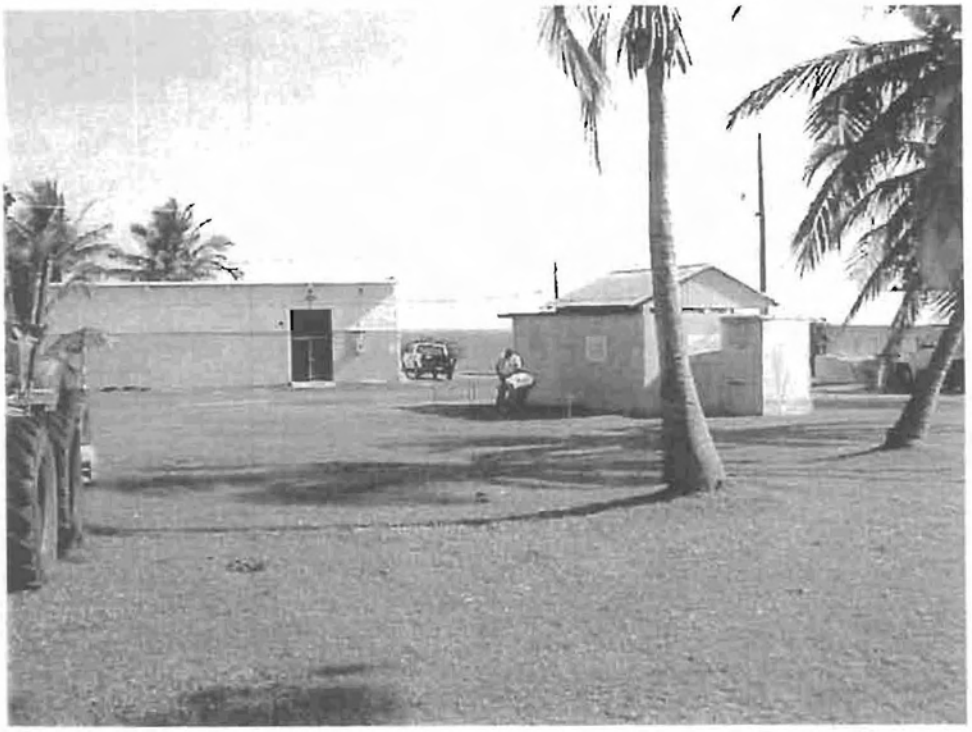


Figure 18. SWMU 10, maintained area

Figure 19. SWMU 14 (wash rack to left) and SWMU 15 (right of light gravel area)

Figure 20. Control area southwest of SWMU 14.

Figure 21. AOC C, drainage looking North

Figure 22. AOC E-UST, wash rack

Figure 23. AOC F-UIC, showing bare areas

Table 10

Vegetation Observed at SWMU 10, SWMU 14, SWMU 15, AOC C, AOC E-UST, and AOC F-UIC

Common Name	Scientific Name	Stratum
Better man better	*Achyranthes aspera*	H or SS
Lumboo, whitey mary	*Amaranthus viridis*	H
None	*Bothriochloa ischaemum*	H
Sandbur, sand-spurs	*Cenchrus brownii*	H
Sandbur	*Cenchrus echinatus*	H
None	*Cissus sicyoides*	H
French grass	*Commelina erecta*	H
Bermuda grass	*Cynodon dactylon*	H
None	*Cyperus flexuosus*	H
None	*Datura inoxia*	H
Jimsonweed	*Datura stramonium*	H
Zarzabacoa común, tick trefoil	*Desmodium incanum*	H/S
None	*Digitaria ciliaris*	H
None	*Echinochloa colona*	H
Dutch grass	*Eleusine indica*	H
None	*Eragrostis amabilis*	H
Siempreviva silvestre	*Gomphrena serrata*	H
Willy vine	*Ipomea tiliacea*	H
None	*Ludwigia octovalus*	H
None	*Panicum diffisum*	H
None	*Paspalidium germinatum*	H
Jump up an' kiss me, purslane	*Portulaca oleracea*	H
Mesquite	*Prosopis glandulosa*	S
Christmas pride	*Ruellia tuberosa*	H
None	*Schrankia uncinata*	H
Bejuco de sopla, jaboncillo	*Securidaca virgata*	S
None	*Sida* sp.	H
None	*Sporobulus indicus*	H
Barrow, node weed	*Synedrella nodiflora*	H
White cedar, pink cedar	*Tabebuia heterophylla*	T
None	*Tridax procumbens*	H
Yerba socialista, socialist herb	*Vernonia cinerea*	H
None	*Vernonia lineria*	H

S = shrub
T = tree
H = herbaceous
V = vine

Table 11

Wildlife Observed at AOC Sites, SWMU 10, SWMU 14, and SWMU 15

English Name	Scientific Name	Local Name
Mammals		
Mongoose	*Herpestes auropunctatus*	Not known
Horse	*Equus Caballus*	Caballo
Birds		
Red-tailed Hawk	*Buteo jamaicensis*	Guaraguao de Cola Roja
Killdeer	*Charadrius vociferous*	Playero Sabanero
Common-ground Dove	*Columbina passerina*	Rolita
Scaley-naped Pigeon	*Columda squamosa*	Paloma Turca
Snowy Egret	*Egretta thula*	Garza Blanca
Puerto Rico Woodpecker	*Melanerpes portoricensis*	Carpintero de Puerto Rico
Northern Mockingbird	*Mimus polyglottos*	Ruiseñor
Greater Antillean Grackle	*Quiscalus niger*	Mozambique (Chango)
Gray Kingbird	*Tyrannus dominicensis*	Pitirre
Black-Whiskered Vireo	*Vireo altiloquus*	Bien-te-veo
White-winged Dove	*Zenaida asiatica*	Tórtola Aliblanca
Zenaida Dove	*Zenaida aurita*	Tórtola Cardosantera

the Red-tailed hawk (*Buteo jamaicensis*) and Killdeer (*Charadrius vociferous*) were present during the surveys and utilize the habitat for feeding and/or breeding.

Protected Species

There were no federally protected species or preferred habitat observed at this site. The sites were dominated by maintained herbaceous grassland, which is not preferred habitat for any of the species except the Arctic peregrine falcon.

The Arctic peregrine falcon has been observed nearby at NAVSTA Roosevelt Roads (U.S. Navy 1998b), and potential feeding habitat for these birds is present on the site. Arctic peregrine falcons prefer edges and open areas to hunt prey. Foraging habitat within and/or near the site is present since the majority of the area is open grassland.

Food Web

Figure 6 presents a generalized food web for the herbaceous community type found at this SWMU site.

CONCLUSION

The past activities at all to the SWMU sites presented in this report have some degree of impacts on their ecosystems. However, these impacts appear to be limited to changes in species composition based on physical disturbances. The construction of roads and grounds maintenance is the only disturbances that

have caused noticeable differences. Wildlife at these sites seems to be healthy and utilizing the habitats to their fullest extent. Through these surveys, no federally protected species were identified at these sites.

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APPENDIX A

Birds Potentially Occurring at NASD Vieques

Pied-billed grebe (*Podilymbus podiceps*)
Red-billed tropicbird (*Phaethon aethereus*)
Brown pelican (*Pelecanus occidentalis*)
Brown booby (*Sula leucogaster*)
Magnificent frigatebird (*Fregata magnificens*)
Great blue heron (*Ardea herodias*)
Louisiana heron (*Hydranassa tricolor*)
Snowy egret (*Egretta thula*)
Great egret (*Egretta alba*)
Striated heron (*Butorides striatus*)
Little blue heron (*Florida caerulea*)
Cattle egret (*Bubulcus ibis*)
Least bittern (*Ixobrychus exilis*)
Yellow-crowned night heron (*Nyctanassa violacea*)
Black-crowned night heron (*Nycticorax nycticorax*)
White-cheeked pintail (*Anas bahamensis*)
Blue-winged teal (*Anas discors*)
American widgeon (*Anas americana*)
Red-tailed hawk (*Buteo jamaicensis*)
Osprey (*Pandion haliaetus*)
Merlin (*Falcon columbarius*)
Clapper rail (*Rallus longirostris*)
American coot (*Fulica americana*)
Caribbean coot (*Fulica caribaea*)
Common gallinule (*Gallinula chloropus*)
Piping plover (Charadrius melodus)
Semipalmated plover (*Charadrius semipalmatus*)
Black-bellied plover (*Squatarola squatarola*)
Wilson's plover (*Charadrius wilsonia*)
Killdeer (*Charadrius vocifera*)
Ruddy turnstone (*Arenaria interpres*)
Black-necked stilt (*Himantopus himantopus*)
Whimbrel (*Numenius phaeopus*)
Spotted sandpiper (*Actitis macularia*)
Semipalmated sandpiper (*Calidris pusilla*)
Short-billed dowitcher (*Limnodromus griseus*)
Greater yellowlegs (*Tringa melanoleauca*)
Lesser yellowlegs (*Tringa flavipes*)
Willet (*Catoptrophorus semipalmatus*)
Stilt sandpiper (*Micropalama himantopus*)
Pectoral sandpiper (*Calidris melanotos*)
Laughing gull (*Larus atricilla*)
Royal tern (*Thalasseus maximus*)
Sandwich tern (*Thalasseus sandvicensis*)
Bridled tern (*Sterna anaethetus*)
Least tern (*Sterna albifrons*)
Brown noddy (*Anous stolidus*)
White-winged dove (*Zenaida asiatica*)
Zenaida dove (*Zenaida aurita*)
White-crowned pigeon (*Columba leucocephala*)
Mourning dove (*Zenaida macroura*)
Red-necked pigeon (*Columba squamosa*)
Common ground dove (*Columba passerina*)
Bridled quail dove (*Geotrygon mystacea*)

Birds Potentially Occurring at NASD Vieques (Continued)

Ruddy quail dove (*Geotrygon montana*)
Caribbean parakeet (*Aratinga pertinax*)
Smooth-billed ani (*Crotophaga ani*)
Yellow-billed cuckoo (*Coccyzus americanus*)
Mangrove cuckoo (*Coccyzus minor*)
Short-eared owl (*Asio flammeus*)
Chuck-will's-widow (*Caprimulgus carolinensis*)
Common nighthawk (*Chordeiles minor*)
Antillean crested hummingbird (*Orthorynchus cristatus*)
Green-throated carib (*Sericotes holosericeus*)
Antillean mango (*Anthracothorax dominicus*)
Belted kingfisher (*Ceryle alcyon*)
Gray kingbird (*Tyrannus dominicensis*)
Loggerhead kingbird (*Tyrannus caudifasciatus*)
Stolid flycatcher (*Myiarchus stolidus*)
Caribbean elaenia (*Elaenia martinica*)
Purple martin (*Progne subis*)
Cave swallow (*Petrochelidon fulva*)
Barn swallow (*Hirundo rustica*)
Northern mockingbird (*Mimus polyglottos*)
Pearly-eyed thrasher (*Maragarops fuscatus*)
Red-legged thrush (*Mimocichla plumbea*)
Black-whiskered vireo (*Vireo altiloquus*)
American redstart (*Setaophaga ruticilla*)
Parula warbler (*Parula americana*)
Prairie warbler (*Dendroica discolor*)
Yellow warbler (*Dendroica petechia*)
Magnolia warbler (*Dendroica magnolia*)
Cape May warbler (*Dendroica tigrina*)
Black-throated blue warbler (*Dendroica caerulescens*)
Adelaide's warbler (*Dendroica adelaidae*)
Palm warbler (*Dendroica palmarum*)
Black and white warbler (*Mniotilta varia*)
Ovenbird (*Seiurus aurocapillus*)
Northern water thrush (*Seiurus noveboracensis*)
Bananaquit (*Coerba flaveola*)
Striped-headed tanager (*Spindalis zena*)
Shiny cowbird (*Molothrus bonariensis*)
Black-cowled oriole (*Icterus dominicensis*)
Greater Antillean grackle (*Quiscalis niger*)
Yellow-shouldered blackbird (*Agelaius xanthomus*)
Hooded mannikin (*Lonchura cucullata*)
Yellow-faced grassquit (*Tiaris olivacea*)
Black-faced grassquit (*Tiaris bicolor*)
Least sandpiper (*Calidris minutilla*)
Western sandpiper (*Calidris mauri*)
Puerto Rican woodpecker (*Melanerpes portoricensis*)
Rock dove (*Columba livia*)
Puerto Rican emerald (*Chlorostilbon maugeus*)
Puerto Rican flycatcher (*Myiarchus antillarum*)
Pin-tailed whydah (*Vidua macroura*)
Spice finch (*Lonchura punctulata*)
Ruddy duck (*Oxyura jamaicensis*)
Peregrine falcon (*Falco peregrinus*)

Birds Potentially Occurring at NASD Vieques (Continued)

Marbled godwit (*Limosa fedoa*)
Puerto Rican lizard cuckoo (*Saurothera vieilloti*)
Prothonotary warbler (*Protonotaria citrea*)
Green-winged teal (*Anas carolinensis*)
Orange-cheeked waxbill (*Estrilda melpoda*)
Least grebe (*Tachybaptus dominicus*)
West Indian whistling duck (*Dendrocygna arborea*)
Puerto Rican screech owl (*Otus nudipes*)
Puerto Rican tody (*Todus mexicanus*)

Sources: Vila and Azar 1974, Weaver et al 1976, Pace 1989