EnSafe / Allen & Hoshall
a joint venture for professional services

5720 Summer Trees Dr. Suite 8 Memphis, TN 38134
(901) 383-9115 Fax (901)383-1743

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NAS PENSACOLA
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October 12, 1992

Commanding Officer, Southern Division
Naval Facilities Engineering Command
Attn: Ms. Linda Martin, code 18211
2155 Eagle Drive
Charleston, South Carolina 29411

RE: Final Sampling and Analysis Plan, Operable Unit 10, NAS-Pensacola, Contract # N62467-89-D-0318/048

Dear Ms. Martin:

Enclosed please find the response to comments and two copies of the Final Sampling and Analysis Plan, Remedial Investigation/Feasibility Study, Operable Unit 10 for the Naval Air Station Pensacola in Pensacola, Florida. The amendment to include Site 13 as a part of Operable Unit 10 is being sent under a separate cover.

If you should have any questions or need any additional information regarding the plan, please do not hesitate to call me.

Sincerely,
EnSafe\Allen & Hoshall

Henry H. Beiro

Henry H. Beiro
Task Order Manager

Enclosure
Final Sampling and Analysis Plan

cc:
Ron Joyner, NASP - 6 copies
Eric Nuzie, FDER - 2 copies
Tom Moody, FDER - 1 copy
Allison Drew, EPA - 2 copies
Waynon Johnson, NOAA - 1 copy
Lynn Griffin, FDNR - 1 copy
File - 1 copy

SAMPLING AND ANALYSIS PLAN
FOR OPERABLE UNIT 10
FINAL

Prepared for:

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN)
NAVAL SUPPORT ACTIVITY
NAVAL AIR STATION
PENSACOLA, FLORIDA

CONTRACT N62467-89D-0318

Prepared by:

ENSAFE/ALLEN & HOSHALL
5724 Summer Trees Drive
Memphis, Tennessee 38134
(901) 372-7962

October 12, 1992

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1.0 INTRODUCTION

As part of the U.S. Navy Comprehensive Long-Term Environmental Action Navy (CLEAN) Program, a Remedial Investigation/Feasibility Study (RI/FS) will be completed by EnSafe/Allen & Hoshall (E/A&H) at the Industrial Waste Treatment Plant (IWTP) located at the Naval Air Station (NAS) in Pensacola, Florida. A work plan entitled *Remedial Investigation/Feasibility Study Work Plan - Group O,* Naval Air Station, Pensacola, Florida was completed by Ecology & Environment, Inc. and was submitted to the Southern Division Naval Facilities Engineering Command (SOUTHDIV) in February 1992. The investigative work will be completed to fulfill the requirements of the work plan and will be completed in accordance with the procedures in the EPA document *Standard Operating* Procedures *and Quality* Assurance *Manual,* February 1, 1991 (SOP/QAM). A copy of the SOPQAM will be kept onsite during all field operations.

The investigation consists of three broad tasks: physical surveys, field sampling, and analysis. The physical surveys are screening tools and will consist of a preliminary air survey, radiation survey, habitat/biota survey, and a soil *gas* survey. The physical surveys will be conducted at NEESA Level B (EPA Level 2) protocol. The field sampling is used to delineate the extent of contamination in the selected media. The media to be sampled include surface water, sediment, soils, and groundwater. The analytical tasks will be performed by a NEESA certified laboratory. Laboratory analysis of samples collected will be performed using approved methods. Field sampling, analytical methods, and reports will be conducted at NEESA Level D (EPA Level 4) protocol.

A report will be submitted to SOUTHDIV upon completion of the investigative work and laboratory analysis summarizing the activities, results and conclusions of the investigation. The report will provide the basis for an FS to be completed at the site.

This sampling and analysis plan will provide guidance on sampling and analytical techniques to be used at the site. In addition, this plan will outline proper documentation procedures for all phases of the investigation.

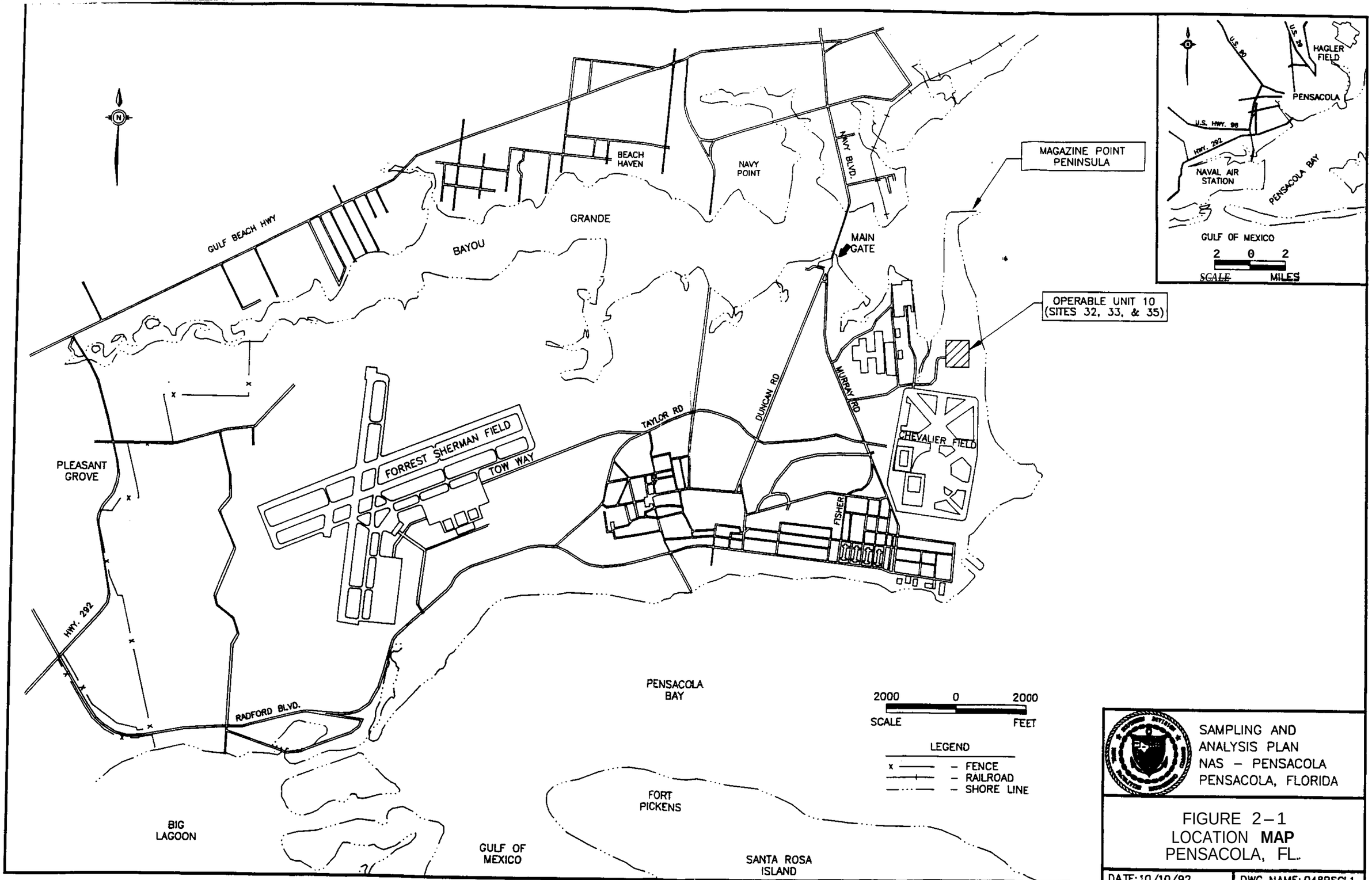
2.0 BACKGROUND INFORMATION

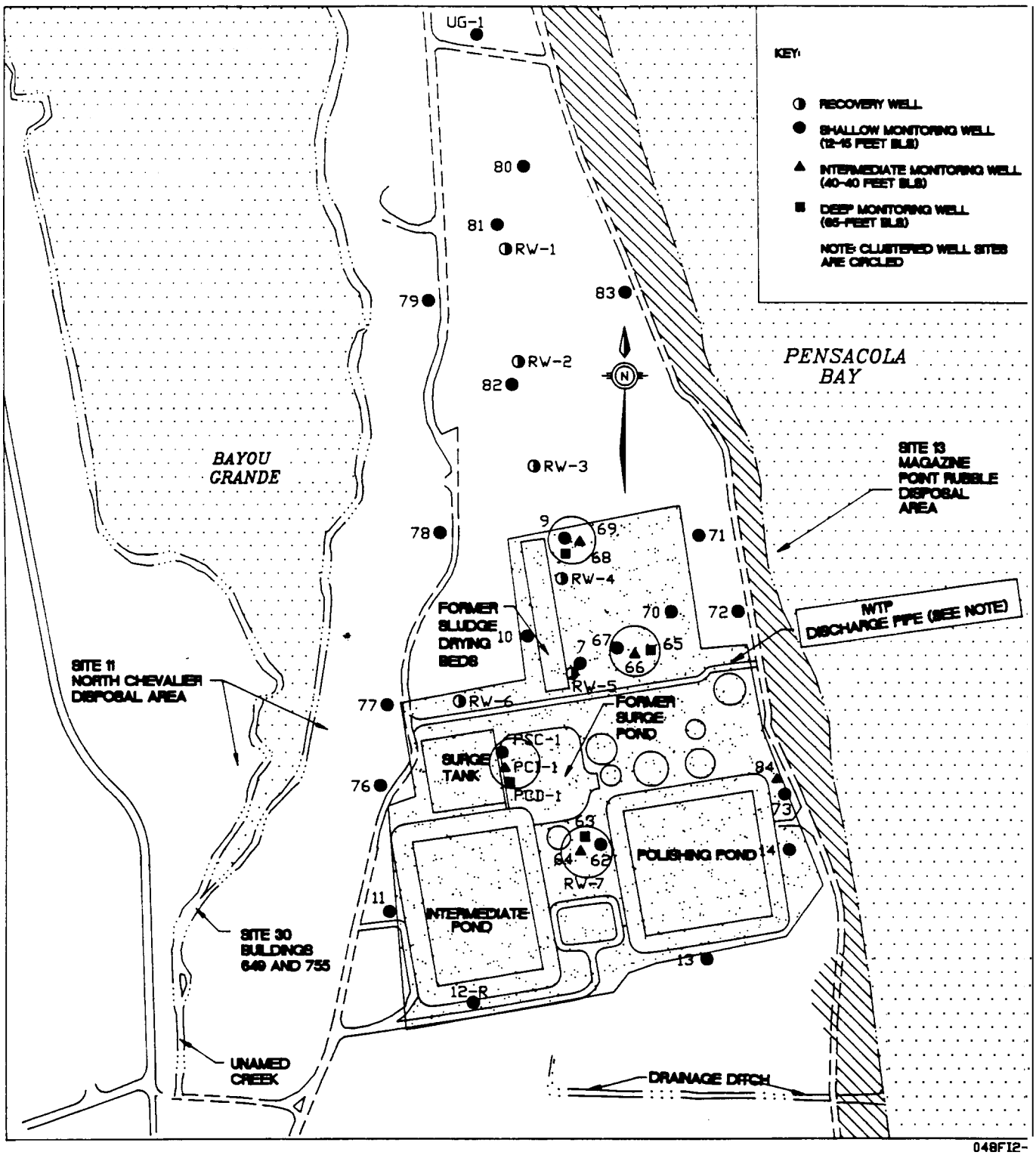
2.1 Site Description

The IWTP located at NAS in Pensacola, Escambia County, Florida is comprised of three sites. These sites are the Industrial Sludge Drying Beds (Site 32), Wastewater Treatment Plant (WWTP) Ponds (Site 33) and the miscellaneous IWTP SWMUs (Site 35). These three sites at the IWTP comprise Operable Unit 10. Operable Unit 10 is located on Magazine Point Peninsula and occupies approximately 26 acres. The location of Operable Unit 10 is shown on Figure 2-1.

Three additional sites are located adjacent to Operable Unit 10 and are shown in Figure 2-2. Site 13 (Magazine Point Rubble Disposal Area) is located to the east of Operable Unit 10, the northern end of Site 30 (Buildings 649 and 755) occupies the southwestern border, and Site 11 (north of the Chevalier Disposal Area) occupies the western border. Located at or adjacent to Operable Unit 10 are 23 shallow monitoring wells, five intermediate monitoring wells, four deep monitoring wells, and seven groundwater recovery wells. The terms shallow, *intermediate,* and deep refer to the zone of placement within the surficial sand and gravel aquifer. These terms correspond to three zones of differing permeability; a surficial zone (~20'), a low permeability zone (~40'), and a producing zone (~65').

Magazine Point Peninsula is surrounded by Bayou Grande to the north and west and Pensacola Bay to the east. The coastal waters are classified as Class III waters. According to the Florida Depaxtment of Environmental Regulation (FDER), Class III waters are used for recreational purposes and the maintenance of a well-balanced fish and wildlife population.


GULF BEACH HWY
BEACH HAVEN
NAVY POINT
NAVY BLVD.
GRANDE
BAYOU
MAIN GATE
MAGAZINE POINT PENINSULA
OPERABLE UNIT 10 (SITES 32, 33, & 35)
PLEASANT GROVE
FORREST SHERMAN FIELD
TOW WAY
TAYLOR RD
DUNCAN RD
MURRAY RD
CHEVALIER FIELD
FISHER
HWY. 292
RADFORD BLVD.
PENSACOLA BAY
BIG LAGOON
GULF OF MEXICO
FORT PICKENS
SANTA ROSA ISLAND
2000 0 2000
SCALE FEET
LEGEND
x — FENCE
— RAILROAD
— SHORE LINE
U.S. 90
U.S. 29
HAGLER FIELD
PENSACOLA
U.S. HWY. 98
HWY. 292
NAVAL AIR STATION
PENSACOLA BAY
GULF OF MEXICO
2 0 2
SCALE MILES
SAMPLING AND ANALYSIS PLAN
NAS – PENSACOLA
PENSACOLA, FLORIDA
FIGURE 2–1
LOCATION MAP
PENSACOLA, FL.
DATE: 10/10/92
DWG NAME: 048PSCL1



SOURCE: GERAGHTY & MILLER INC., 1988

SCALE
0 300 600

SHADED AREA ENCOMPASSES SITES 32,33,AND 35

NOTE: DISCHARGE POINT FOR THIS PIPE LOCATED APPROXIMATELY 2,500' OUT IN PENSACOLA BAY.

IWTP INDUSTRIAL WASTEWATER TREATMENT PLANT

FIGURE 2-2 SITE MAP - NAS PENSACOLA, SITES 32,33,AND 35

2.2 Previous Investigations

2.2.1 Chronology of Site Activities

1971 The IWTP Sludge Drying Beds, WWTP Ponds, and miscellaneous IWTP SWMUs were constructed for tertiary treatment of industrial waste and secondary treatment of domestic wastes.

1973 IWTP was receiving and treating wastes generated by the industrial activities at NAS Pensacola.

1979 During maintenance of a clogged industrial waste line, an industrial waste spill of approximately 80,000 gallons occurred because of pump failure at the final industrial waste lift station. The spill was released into a ditch (Site 30) near the lift station that eventually discharged into Bayou Grande. The spill occurred approximately 2,000 feet southwest of the subject site. Although it is believed the spill did not impact Operable Unit 10, it was indicative of the waste received at Operable Unit 10. The release caused a minor fish kill in Bayou Grande (NEESA 1983), but was deemed a minor incident by the FDER. No further investigation was initiated.

1981 The IWTP was designated a Hazardous Waste Surface Impoundment (HWSI) and received an average of 880,000 gallons of wastes per day. The waste water contained high concentrations of organic solvents, phenols, chromium electroplating wastes (including cyanide and other heavy metals), and wastes from an onsite chemical conversion coating process for aluminum. The surge pond located at the IWTP was estimated to be leaking 5,800 gallons per day by Missimer and Associates, Inc. Four shallow monitoring wells were installed by Missimer and Associates, Inc. to meet the requirements of the Resource Conservation and Recovery Act (RCRA). A background well (UG-

1) was installed approximately 1,800 feet north of the surge pond, and three additional monitoring wells (DG-1, DG-2, and DG-3) were installed in the vicinity and immediately northwest of the surge pond. Although monitoring well UG-1 is hydraulically downgradient of the IWTP, the Navy, EPA and FDER agreed upon it as a background well. Analytical results from monitoring well UG-1 indicate it has not been impacted by the IWTP.

1983 Compliance requirements were not met with the previously installed monitoring wells. Three additional monitoring wells were installed by Missimer and Associates, Inc. at the toe of the dike surrounding the surge pond. The newly installed monitoring wells (DG-4 through DG-6) and two previously existing monitoring wells (UG-1 and DG-1) were designated as RCRA detection monitoring wells.

A sulfuric acid spill of approximately 2,200 gallons occurred at the sulfuric acid storage tank located in the chemical storage area near the northeast corner of the surge pond. Sand was used as an absorbent for the spill and lime was used as a neutralizing agent. The contaminated sand was then placed in the sludge drying beds. Water was used to rinse the land surface of the spill area, and the water was allowed to infiltrate (Geraghty & Miller 1988c).

1984 The IWTP sludge drying beds were removed from service.

1984 A water quality assessment program was initiated at the IWTP by Geraghty & Miller to investigate contamination in the upper portion of the shallow aquifer. Seven additional shallow groundwater monitoring wells (GM-8 through GM-14) were installed at the facility by Geraghty & Miller. The

monitoring wells were installed near the former sludge drying beds and the WWTP ponds. Groundwater samples were collected and submitted for laboratory analysis during three sampling events from the existing RCRA detection monitoring wells and the newly installed monitoring wells.

1985 The FDER issued a temporary RCRA operation permit (permit no. HT17-68087) to the U.S. Navy Public Works Department for the surge pond. A permanent permit was issued in September 1987 (permit no. H017-12-7026).

A Phase II groundwater investigation was initiated by Geraghty & Miller to delineate the horizontal and vertical extent of groundwater contamination. Fourteen additional monitoring wells (GM-62, GM-67, GM-70 through GM-73, and GM-76 through GM-83) were installed at the facility. Four intermediate monitoring wells (GM-64, GM-66, GM-69 and GM-75) and four deep monitoring wells (GM-63, GM-65, GM-68 and GM-74) were also installed at the facility.

In September, groundwater samples were collected from monitoring wells DG2, DG3, GM-8 through GM-14, and GM-62 through GM-83 and were submitted for laboratory analysis.

1986 To comply with conditions in the FDER Temporary Operating Permit No. HT17-68087, a Corrective Action Program was implemented at the IWTP. Seven groundwater recovery wells were installed at the facility.

1987 In January, a comprehensive groundwater monitoring evaluation was conducted by the EPA. Groundwater samples were collected from seven

shallow monitoring wells (UG-1, DG-4, GM-12, GM-71, GM-73 and GM-80) and one deep monitoring well (GM-68).

1987 In February, the groundwater recovery system was placed in operation.

In September, the FDER issued permit no. H017-127026 to the U.S. Navy Public Works for operation of the surge pond stipulating the continued operation of the Corrective Action System. In addition, the permit required the implementation of a quarterly groundwater monitoring program.

In November, the first quarterly groundwater sampling event was conducted at the IWTP.

1988 In January, the FDER issued a closure permit to the U.S. Navy Public Works for the following sites: polishing pond, stabilization pond, and the sludge drying beds (permit no. HF17-134657). Upon closure of the ponds and beds, the clay liner and/or subsurface soils were sampled for laboratory analysis.

In February, monitoring wells DG-1, DG2, DG6, GM-74 and GM-75 were abandoned so that a surge tank could be constructed to replace the surge pond.

In April, three temporary monitoring wells were installed by Geraghty & Miller to investigate the source of acidic groundwater. It was determined that the intermediate zone of the surficial aquifer was impacted and that recovery well RW-5 was recovering the acid.

In November, a closure permit was issued to the U.S. Navy Public Works by the FDER for the surge pond (permit no. HF17-148989). Upon closure of the pond, the clay liner and/or subsurface soils were sampled for laboratory analysis.

In December, a semi-annual report for the corrective action and compliance monitoring program was submitted by Geraghty & Miller. The report recommended the addition of five recovery wells to enhance the effectiveness of the corrective action system.

1989 In May, monitoring of the corrective action program was transferred to Ecology & Environment (E & E).

1990 In March, three additional monitoring wells were installed by E & E on the western flank of the former surge pond. These wells were installed to replace those wells abandoned during the construction of the surge tank.

From July 1990 to November 1991 the recovery system was inoperative.

1991 In September, the FDER issued permit no. HF17-170951, changing the monitoring requirement from quarterly to semi-annually. Appendix IX analyses were also deleted from the monitoring program.

1992 In February, a report entitled ***Remedial Investigation/Feasibility Study Wonk Plan — Group O, Naval Air Station Pensacola*** was submitted by E & E.

2.2.2 Previous Analytical Results

soils

Soil samples were collected in accordance with the closure permits for the former sludge drying beds, former surge ponds, and the polishing and stabilization ponds. Laboratory analysis detected only low concentrations of phenols in the clay liner and subsurface soils beneath the polishing and stabilization ponds. Therefore, these two impoundments were given clean closure status. Soils collected beneath the sludge drying beds had detected concentrations of cyanide (0.3 mg/kg); 1,3-dichlorobenzene (18 pglkg); 1,4-dichlorobenzene (14 μg/kg); and toluene (10 pglkg). Soils beneath the surge pond had detected concentrations of cyanide (1.2 mg/kg); 1,2-dichlorobenzene (28 μg/kg); 1,3-dichlorobenzene (67 μg/kg); and 1,4-dichlorobenzene (10 pglkg).

Groundwater

The following is a summary of the analytical results for sampling events conducted during the past 8 years at the IWTP. Monitoring wells at the IWTP have been designated into 3 categories, with different laboratory analyses, based upon their use under the RCRA permits. These designations are point-of-compliance (POC), corrective action (CA) and background (UG-1). The discussion is divided into 4 major groups: metals, inorganics, VOCs and BNAs. Only the parameters that exceeded their respective Florida Primary Drinking Water Standards (FPWDS) or their Florida Secondary Drinking Water Standards (FSWDS) will be discussed. A summary of the standard exceedances is presented in Appendix A.

Metals

Sodium, cadmium, chromium, lead, arsenic and mercury have been detected at concentrations that exceed their respective FPDWSs. During the July and October 1984 sampling events, samples were filtered and submitted for laboratory analysis of dissolved

metals. In all other sampling events the samples were not filtered and were submitted for laboratory analysis of total metals.

Sodium was detected at concentrations that exceeded the FPDWS of 160 mg/l in monitoring wells PCS-1, PCI-1, PCD-1, GM-14, GM-63, GM-64, GM-65, GM-66, GM-68, GM-69, GM-71, GM-72, GM-76, GM-77 and GM-84 during the monitoring program.

Cadmium was detected at concentrations that exceeded the FPDWS of 10 pgll in monitoring well GM-66. Cadmium concentrations have increased from being not detected in March of 1989 to 57 µg/l in March of 1991.

Chromium concentrations have exceeded the FPDWS of 50 µg/l in monitoring well GM-66. Detected concentrations have increased from 210 pgll in March 1990 to 360 µg/l in March 1991.

The detected concentration of lead in monitoring well GM-66 increased from 2.6 pgll in March 1990 to 400 µg/l in March 1991. The detected concentration exceeded the FPDWS of 50 pgll in March 1991.

In monitoring well UG-1 arsenic was detected in May 1990 in concentrations (51 µg/l) that exceeded the FPDWS of 50 µg/l. Concentrations of arsenic have decreased to 2.6 µg/l (November 1990) or to below the method detection limit in subsequent sampling events.

The detected concentration (4.4 µg/l) of mercury exceeded the FPDWS (2 µg/l) in monitoring well GM-11 during the sampling event in November 1989. Mercury was not detected above the method detection limit in subsequent sampling events.

The FSDWS of 50 µg/l for manganese has been exceeded by the detected concentrations in monitoring wells UG-1, DG-1, DG2, DG3, DG-4, DG-5, DG6, GM-8, GM-9, GM-10, GM-12R, GM-13, GM-14, GM-62, GM-64, GM-66, GM-69, GM-75, GM-63, GM-65 GM-68, PCS-1, PCI-1, and PCD-1.

The detected concentrations of iron have exceeded the FSDWS of 300 µg/l in monitoring wells UG-1, GM-8, GM-9, GM-10, GM-12R, GM-13, GM-14, GM-62, GM-63, GM-65, GM-66, GM-67, GM-68, GM-69, PCS-1, PCI-1 and PCD-1.

Inorganics

Of the additional inorganic parameters analyzed for (i.e., nitrate, cyanide, sulfate, chloride), only chloride and sulfate have been detected at levels that exceed their respective FSDWS.

Detected levels of chloride have exceeded the FSDWS of 250 mg/l in monitoring wells GM-63, GM-64, GM-65, GM-66, GM-69, GM-71, GM-72, GM-76, GM-77, GM-84, PCI-1 and PCD-1. The FSDWS for sulfate of 250 mg/l was exceeded by the detected concentration of sulfate in monitoring well GM-66.

Volatile Organic Compounds

Detected concentrations of vinyl chloride; 1,1-dichloroethene; 1,2-dichloroethane; chloroform; carbon tetrachloride; trichloroethene; 1,1,1-trichloroethene; tetrachloroethene; and benzene have exceeded their respective FPDWS at Operable Unit 10. The following section discusses standard exceedances by the VOC parameters.

Detected concentrations of vinyl chloride exceeded the FPDWS of 1µg/l in monitoring wells DG-1, DG-2, DG-4, DG-5, DG-6, GM-8, GM-9, GM-10, GM-64, GM-66, GM-69, and GM-75. The detected concentrations have decreased during the monitoring program.

Monitoring wells GM-66 and GM-69, which are intermediate zone wells have generally had higher detected concentrations of vinyl chloride.

Detected concentrations of 1,1-dichloroethene have exceeded the FPDWS of 7 μg/l in monitoring wells DG-1, DG6, GM-8, GM-9, GM-64, GM-66, GM-67, and GM-69. Detected concentrations of 1,1-dichloroethene have been generally decreasing since May 1989.

1,2-dichloroethane has been detected at concentrations that exceeded the FPDWS of 3 μg/l in monitoring wells GM-9 and GM-64.

The detected concentration of chloroform in monitoring well DG-1 exceeded the FPDWS of 100 μg/l in October 1984. Chloroform was not detected above the method detection limit in a subsequent sampling event in February 1988.

Carbon tetrachloride was detected above the FPDWS (3 μ/l) in monitoring wells DG-1 and DG-6 in October 1984. In the February 1988 sampling event, carbon tetrachloride was not detected above the method detection limit.

Detected levels of trichloroethene exceeded the FPDWS of 3 μg/l in monitoring wells UG-1, DG-1, DG2, DG-4, DG6, GM-8, GM-12R, GM-65, GM-66, GM-67, GM-68, and GM-69. The concentrations have decreased in the shallow and deep zones while increasing in the intermediate zone since August 1988. The intermediate zone has also had significantly higher detected concentrations of trichloroethene.

Detected concentrations of 1,1,1-trichloroethane have exceeded its FPDWS of 200 μg/l in monitoring wells DG-1, DG6, GM-8, and GM-9. The concentrations have decreased since 1984 in the DG series wells and since 1989 in the GM series wells.

Detected concentrations of tetrachloroethene have exceeded the FPDWS of 3 µg/l in monitoring wells DG-1, DG2, DG3, DG-4, DG6, GM-8, GM-9, GM-62, Gm-63, GM-64, GM-66, GM-69 GM-81, GM-82, and GM-83. Detected concentrations have generally decreased since September 1985 in all of the site monitoring wells.

BNAs

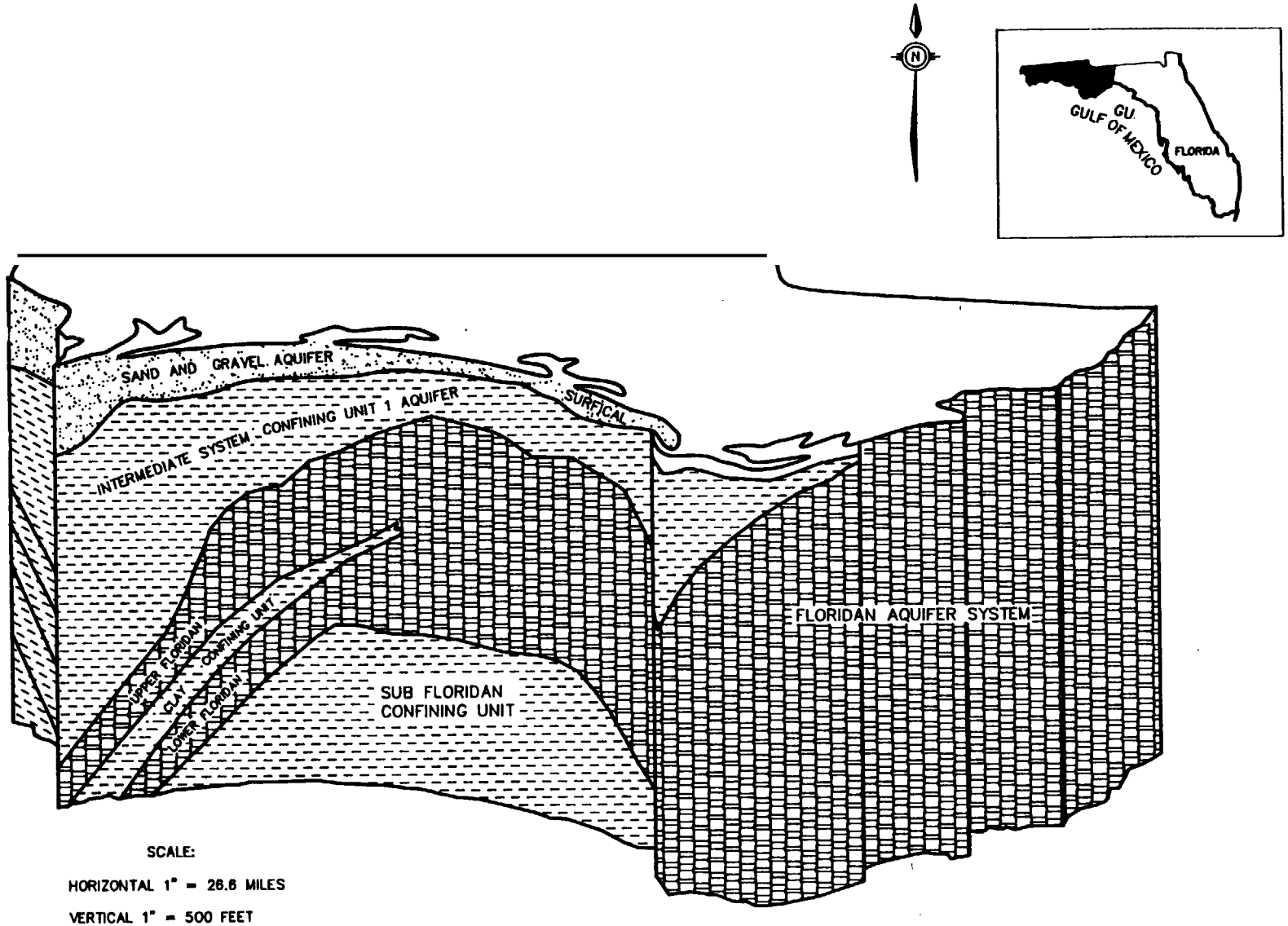
1,4-dichlorobenzene has been detected at concentrations exceeding the FPDWS of 75 µg/l in monitoring wells DG-1, DG2, DG-4, DG5, DG-6, GM-8, GM-9, GM-10, GM-67 and GM-69. There is no significant trend in the concentrations of 1,4-dichlorobenzene; however, the overall trend is for decreasing concentrations.

2.2.3 Conclusions

Analytical work completed at the site has determined the presence of contamination in the shallow and intermediate zones of the surficial aquifer. Excluding sodium, the deep zone of the surficial aquifer has not had any parameters detected above applicable state standards. Primary areas of groundwater contamination include the area east of the former sludge drying beds and the area near the former surge pond.

2.3 Geology and Hydrology

Surface soils across the site are classified by the U.S. Department of Agricultures's (USDA) Soil Conservation Service as undifferentiated coastal soils bordering salt water. Three principal hydrogeologic units underlie NAS Pensacola. In descending order they are the surficial/sand-and-gravel aquifer, the intermediate system and the Floridan Aquifer System. A generalized cross-section illustrating the hydrogeologic units is provided in Figure 2-3.



SCALE:

HORIZONTAL 1" = 26.6 MILES

VERTICAL 1" = 500 FEET

VERTICAL EXAGGERATION = 281

SAMPLING AND
ANALYSIS PLAN
NAS-PENSACOLA
PENSACOLA, FLORIDA

FIGURE 2-3
GENERALIZED GEOLOGIC CROSS-SECTION OF
HYDROGEOLOGIC UNITS IN
NORTHWEST FLORIDA

DATE: 10/12/92 | DWG NAME: 048FI2-3

SOURCE: John Berkadale 1988.

Surficial/Sand-and-Gravel Aquifer

The surficial aquifer is composed of unconsolidated clastic deposits and is approximately 300 feet thick. The surficial aquifer is a major source of drinking water for this area of Florida. The Surficial Aquifer is comprised of three zones of differing permeabilities. These zones are the surficial zone, the low permeability zone, and the main producing zone. A generalized cross-section of the Surficial Aquifer is presented in Figure 2-4. The three zones are discussed below.

General Surficial Zone

The surficial zone is approximately 40 to 70 feet thick and is composed of a poorly graded quartz sand. The depth to groundwater is less than 1 foot to 20 feet depending on ground surface elevation and tidal influence. This zone has a high permeability, and groundwater velocity is generally high.

Site Specific Surficial Zone

At Operable Unit 10, the surficial zone of the Sand-and-Gravel aquifer is composed of white to light brown, fine-to medium-grained quartz sand (E & E, 1991g) and extends to a depth of approximately 38 to 48 feet. Depth to groundwater in this zone is between 1 to 4 feet below the ground surface depending on the tidal influence and the ground surface elevation. Groundwater flow in this zone is to Bayou Grande or Pensacola Bay, depending on proximity to the water bodies. The average estimated groundwater velocity for March 1990 in the shallow zone is 0.0950 feet/day or 34.67 feet/year (E & E 1991g). The groundwater flow can be locally influenced by the seven well recovery system. From February 1987 to November 1988, the recovery system was operating at an average withdrawal rate of 1.86 million gallons per month (G & M 1988d). The recovery system is currently in operation.

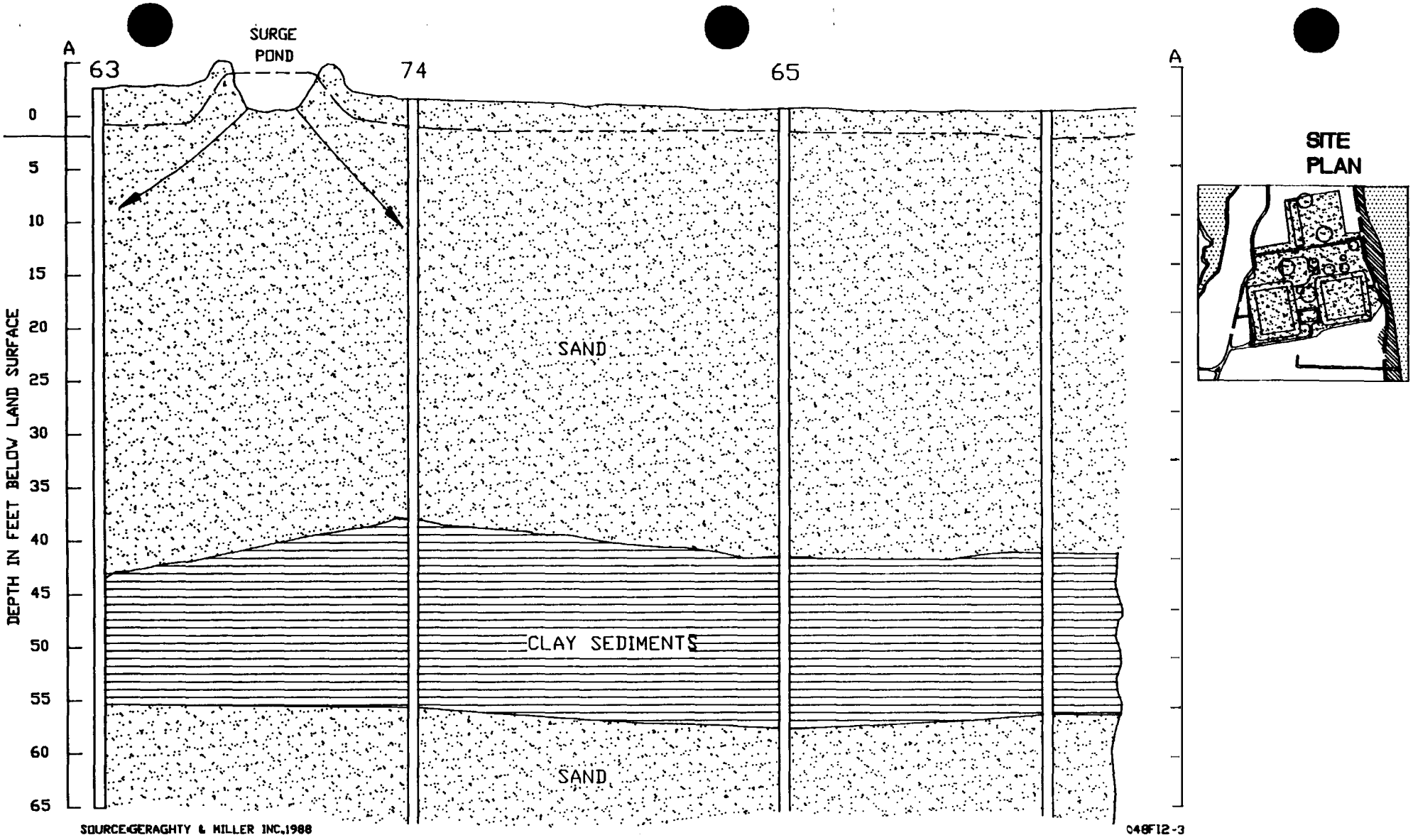


FIGURE 2-4 GENERALIZED GEOLOGIC CROSS-SECTION OF SURFICIAL ZONE OF SAND-AND-GRAVEL AQUIFER IN THE VICINITY OF NAS PENSACOLA SITES 32,33,AND 35.

The intermediate zone is located at the lowermost portion of the Surficial/Sand-and-Gravel aquifer. Groundwater flow in the intermediate zone across Operable Unit 10 is to Pensacola Bay. The average estimated groundwater velocity for this zone in March 1990 is 0.0027 feet/day or 0.9855 feet/year (E& E 1991g).

General Low Permeability Zone

Underlying the surficial zone is the low permeability zone. This zone is composed of clay and silt-size material which are lower permeability sediments. Vertical hydraulic conductivities are lower than the surficial zone. Therefore, this unit acts as a confining or semiconfining unit. The thickness of this zone ranges from 8 to 40 feet, and the unit is believed to be continuous across the site.

Site Specific Low Permeability Zone

The low permeability zone extends to an approximate depth of 55 to 60 feet beneath the ground surface at Operable Unit 10. The low permeability zone is composed of gray, clayey silt which contains shell fragments. No groundwater wells are screened in the intermediate zone. Therefore, the direction and velocity of groundwater flow are not known.

General Main Producing Zone

Underlying the low permeability zone is the main producing zone. This zone is composed of sand and gravel with thin beds of silt and clay. The depth to the top of this zone ranges from 60 to approximately 120 feet. The thickness of the zone is variable but it is estimated to be approximately 300 feet at NAS Pensacola. This zone is the principal source of water supply for the Pensacola area. Because of the overlying low permeability zone, the main producing zone is under confined or semiconfined conditions. A downward hydraulic gradient has been locally observed at NAS Pensacola from the surficial zone to the main producing zone; thus, there is a potential for vertical migration of contaminants.

Site Specific Main Producing Zone

The main producing zone at Operable Unit 10 occurs approximately 55 to 60 feet beneath the ground surface. The lithology of the zone based on monitoring wells completed at Operable Unit 10 is gray, fine to medium grained sand. Groundwater flow is generally to the south across the site. Groundwater velocity is estimated for March 1990 to be 0.0019 feet/day or 0.6935 feet/year (E & E 1991g).

Intermediate System

The intermediate system underlies the sand and gravel aquifer. This unit is regionally extensive and has a lower permeability. Fine grain sediments comprise this system and it functions as a confining unit for the underlying Floridan Aquifer System. The unit does not generally contain water, but local sand seams may exist which would yield small quantities of water. The intermediate system is approximately 1,100 feet thick in the NAS Pensacola area. There are no wells completed into the intermediate system at Operable Unit 10.

Floridan Aquifer System

Underlying the intermediate system is the Floridan Aquifer. The depth to the top of the Floridan Aquifer is approximately 1,500 feet, and the unit is composed of limestone. Groundwater within the Floridan System is highly mineralized in the area of NAS Pensacola and is not used for water supply. There are no wells completed into the Floridan Aquifer System at Operable unit 10.

3.0 PHYSICAL SURVEY

3.1 Preliminary Site Survey

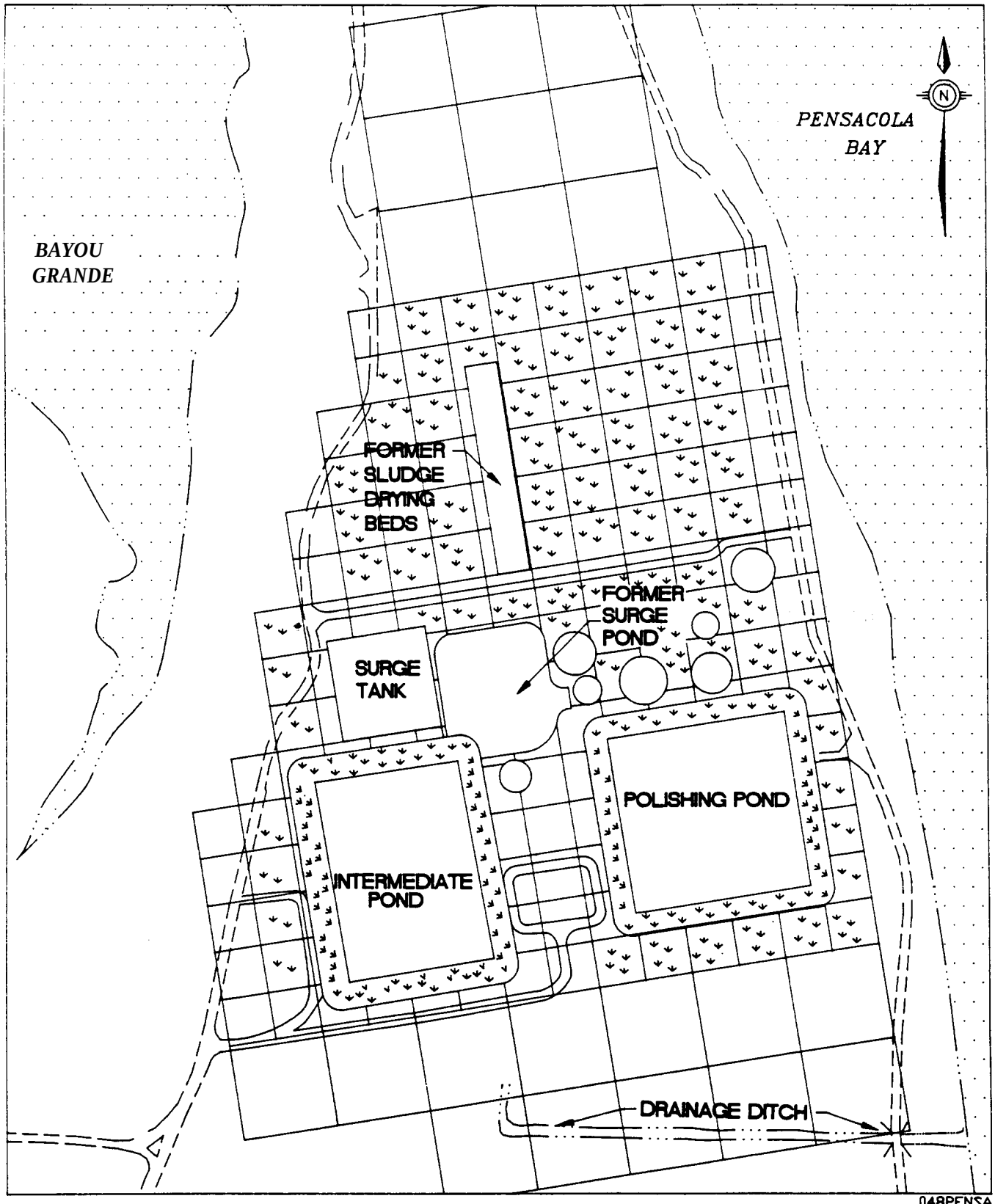
A preliminary survey will be conducted to determine any potential contaminant sources and any present or past waste streams at the facility. The survey will include a review of previous investigative reports, interviews with present and former NAS Pensacola personnel, aerial photo analysis, and a utility survey.

The contaminant survey will include identification of:

- The location of previous and current underground and overhead piping and utilities.
- Past and presently used chemicals used at the facility.
- Locations of any known surface spills or leaks.
- Locations of any known historical outfalls.
- The locations and contents of any known present or former underground storage tanks.

3.2 Preliminary Air Survey

In conjunction with the establishment of the soil gas survey grid network (See Figure 3-1), a preliminary air survey will be conducted across the site using an organic vapor analyzer (OVA). The surface volatile emissions survey will be conducted when ambient conditions are most conducive to volatilization. This condition would be when the air temperature exceeds 80° F, the wind speed is lower than 5 miles per hour (mph), the preceding several days have been characterized by warm, dry weather, and the sun is shining. The OVA will be calibrated according to manufacturer's specifications before use onsite. Surface coverage features (e.g., buildings, asphalt areas, vegetation, soil discoloration) will be noted and recorded in the field notebook. Prior to collecting readings across the site, background readings will be collected at an upwind location. At each air sampling point, the location and two OVA readings will be recorded in the field logbook. The readings will be collected approximately 2 inches above the



SOURCE:GERAGHTY & MILLER INC.1988

SCALE
0 300 600

SAMPLING GRID

SURFACE WATER BODIES

GRASS

FIGURE 3-1 *GAS* **SURVEY GRID SAMPLING LOCATIONS**
NAS PENSACOLA SITES 32,33 AND 35

ground surface. The first reading will be without a carbon filter on the OVA. The second reading will be with a carbon filter on the OVA. The difference between the two readings will be calculated as the concentration of methane at that sampling point. This procedure will be implemented to differentiate between ionizable organic vapors and methane. Each sampling point will also be screened with an explosivity/oxygen (O_2) meter. If elevated readings are detected at 2 inches above the ground surface, an additional reading will be collected in the breathing zone, approximately 3 to 5 feet, to determine site workers' exposure. The location of the air survey point will also be marked on a site map. The background location shall be checked at regular intervals throughout the survey to document any changes in background concentrations. A contaminant quantification survey will be performed based on the results of the preliminary air survey, the soil gas survey results and the surface soil grain size analyses results. Meteorological conditions will be obtained from other Navy facilities located on the base.

3.3 Radiation Survey

In conjunction with the preliminary air survey, a preliminary radiation survey will be conducted at the soil gas survey node locations. Proposed grid locations for the preliminary air survey, the radiation survey and the soil gas survey were previously presented on Figure 3-1. The initial survey objective is to determine if Radium-226 has been transported via the waste water treatment system to sites 32, 33, or 35. Because Radium-226 has a strong gamma emission, its presence can be detected and mapped with a sodium iodide (NaI) type scintillation detector. "he field screening will be conducted by taking measurements at the surface and 1 meter above the ground surface to estimate site workers' exposure. The Micro-R meter uses a NaI scintillation type detector expressing the amount of ionizing radiation in dose units. Measurements will be collected at each established grid point and recorded in the field notebook. The Micro-R meter shall be calibrated by the manufacturer prior to shipment to the site. Calibration

shall be checked in the field using a manufacturer's check source. The equipment will be operated in accordance with manufacturer's specifications. If a source above exposure criteria is discovered during the surveys, a pressurized ion chamber (PIC) may be used to determine dose.

Soil borings will be located at identified source areas from the radiation and soil gas surveys. If no activity above background is encountered, soil borings will be located based solely on soil gas criteria. A gross alpha, beta, and gamma screening will be conducted on all samples with 10 percent of all samples undergoing a complete gamma spectroscopic analysis.

Gamma spectroscopy is needed to determine the isotopic ratios. If contamination is found, analysis for the specific isotopes will be conducted to assess the specific activity of the source. Specific activity and isotopic ratios are required for risk based assessments. If contamination is found, the nature and extent will be modeled using mathematical models such as the DOE RESRAD model. A certified health physicist will be contracted to review all field and analytical data.

3.4 Habitat/Biota Survey

A habitat/biota survey will be performed during the initial investigation to determine any onsite terrestrial and aquatic habitats and any surrounding habitats that could be affected by contamination migration. Any rare, threatened, and endangered species will be documented in addition to the general population of animals and plants.

3.5 Soil Gas Survey

A soil gas survey serves as an effective screening tool for VOC contamination and is an effective guide for establishing soil and groundwater sampling points. The soil gas survey will require the establishment of a grid system across the site. Initially, the two baselines (x and y axis) will be defined by a Florida registered surveyor. The baselines will be flagged at 100-foot intervals. The grid will then be established using a Brunton compass and measuring tape. The proposed soil gas sampling grid is provided in Figure 3-1. Measurements will be collected at each established grid point and recorded in the field logbook. Areas of elevated soil gas readings shall be further investigated by redefining the survey grid to a smaller interval. The intervals shall be decreased to 10 feet, and additional soil gas readings shall be collected. The survey shall be decreased to smaller intervals as needed. The sampling point will be noted on a site map. The baselines and other key elements of the grid will be documented by a Florida registered surveyor for inclusion on report maps.

The soil gas probes will be installed using either a manual disposable drive point method or a hydraulically powered, van-mounted Geoprobe. One duplicate sample will be collected every 20 samples. One rinsate blank sample will be collected every 20 samples to check decontamination procedures. Based on the results of the survey, locations will be selected for *heated headspace analysis*. The *heated headspace analysis* method is described in Appendix B. This method requires the collection of groundwater samples through the stainless steel sampling point for analysis by the portable gas chromatograph (GC). The quality assurance plan (QAP) for soil gas services is included in Appendix B.

Perforated Drive Point Procedures

The following procedures will be followed at each of the soil gas sampling locations if the perforated drive point method is used:

- The soil gas sampling point will be constructed of stainless steel pipe with a disposable sampling point.

- The point will be decontaminated prior to use and will be advanced to slightly above the water table.

- Teflon tubing will connect the soil gas sampling point to the sample collection device. A vacuum pump will be attached to the sample collection device by additional Teflon tubing.

- At least three volumes will be purged from the sampling apparatus before sample collection.

- Following purging, the sample collection device will be closed to its connection to the vacuum pump. After approximately five minutes, the sample collection device will be sealed.

- The sample will be analyzed with a portable GC.

- Upon completion of the sample collection, the sampling point will be removed.

- **At selected sampling locations based on soil gas readings and proximity to source areas, the soil gas sampling points will be advanced into the water table. Groundwater samples will be collected through the sampling point and analyzed using the *heated headspace* method.**

- Between each sampling location, the soil gas probe will be decontaminated. Teflon tubing will not be reused. The materials will be placed in 55-gallon drums for future disposal by the Navy.

Van-Mounted Geoprobe Procedures

If the van-mounted Geoprobe is used, the following procedures will be followed:

- The stainless steel pipe, outfitted with a disposable tip on the lead drive point, will be advanced in the ground with the percussion hammer.

- After the pipe has been advanced to the desired depth, the pipe will be withdrawn 8 to 10 inches to create a void in which soil gases *can* collect.

- Teflon tubing will be inserted in the pipe into the expendable tip holder. The tubing will be connected to a sample collection device. Additional Teflon tubing will be used to attach the sample collection device to a vacuum pump.

- At least three volumes will be purged from the sampling apparatus before sample collection.

- Following purging, the sample collection device will be closed to its connection to the vacuum pump. After approximately five minutes, the sample collection device will be *sealed.*

- The sample will be analyzed with a portable GC.

- **At selected sampling locations based on soil gas readings and proximity to the source areas, the soil gas sampling points will be advanced into the water table. Groundwater samples will be collected through the sampling points and analyzed using the *heated headspace* method.**

- Upon completion of the sample collection, the sampling point will be removed.

- All sampling equipment will be decontaminated before use and between each sample collection point. Teflon tubing will not be reused and will be placed in 55-gallon drums for future disposal by the Navy.

4.0 FIELD SAMPLING PLAN

The field sampling plan details the sampling and field measurement procedures to be used during the facility investigation. The field investigation includes advancing soil borings, installing groundwater monitoring wells, and the collection of air, soil, sediment, surface water and groundwater samples using various techniques. The sampling and analytical requirements for this investigation are provided in Table 4-1.

The EPA Contract Laboratory Program (CLP), Target Analyte List (TAL), and Target Compound List (TCL) are used to provide a defensible full spectrum contaminant analysis. The primary surface drainage is chosen for sampling to assess the current and latest affects on the surface water pathway. Soil and groundwater collection points were selected to asses the soil and groundwater pathways. All soil and water samples will be analyzed for the full TAL/TCL list with additional non-CLP analyses also being conducted.

Table 4-2, taken from E&E's approved work plan, details the rationale for proposed groundwater monitoring wells. These proposed locations of the monitoring wells may change based on the soil gas survey results. Soil gas survey results may indicate a trend that requires further investigation with either soil borings or groundwater monitoring wells.

Medium	No. of Samples[a]	Duplicates	Trip Blanks[b]	Fiid Blanks	Rinsate Blank.	Total	Analytical Suite	Comments
Surface Water	12 (1) (2)	1 — NR	— 1 NR	1 — NR	1 — NR	15 (1), 1 (2)	A B C	1 sampling event at high, intermediate, and low tides
Sediment	4 (1) (2)	1 — NR	— 1 NR	NR NR NR	1 — NR	6 (1), 1 (2)	A B D	
Soil[a]	36 (1) 5 (12) 2	5 — NR NR NR	— 3 NR NR NR	NR NR NR NR NR	3 — NR NR NR	44 (1), 3 5 (12) 2	A B E D F	 Surface Soils Shelby tubes
Groundwater[d]	24 (1) (9)	3 — NR	— 3 NR	3 — NR	3 — NR	33 (1), 3 (9)	A B C	
TOTAL	83 (29)	10 NR	8 NR	4 NR	8 NR	113 (29)		

Source: Ecology and Environment, Inc., 1992

Notes:
[a] The number of samples shown in parentheses will be analyzed for the additional parameters indicated.

[b] Trip blanks will be analyzed for Appendix IX volatile organic compounds only.

Analytical suites:

A = TCL VOCs; TCL base-neutral/acid extractable organic compounds (BNAs); TCL pesticides and polychlorinated biphenyls (PCBs); TAL metals (total [i.e., unfiltered] and dissolved [i.e., millipore-filtered], water only); cyanide; gross alpha, beta, and gamma radioactivity; total organic carbon; total Kjeldahl nitrogen (TKN; surface water and sediment only); grain size (sediment only); hardness (water only); and alkalinity (water only).

B = Appendix IX VOCs, BNAs, dioxins, pesticides, PCBs, organophosphorus pesticides, herbicides, total metals, cyanide,. and sulfide.

C = Total suspended solids, TKN (groundwater only), ammonia nitrogen, orthophosphate phosphorus, dissolved oxygen (in field), 5-day biological oxygen demand (BOD_5), and chemical oxygen demand (COD).

D = pH, alkalinity, percent moisture, grain size (soil only), BTU content, ash content, total organic halogens, sulfur, ignitability, and cation exchange capacity.

E = Grain size.

F = Permeability.

NR = Not required.

[a] 12 boring locations x 3 depth intervals = 36 samples; and 2 locations x 1 depth interval = 2 samples.

[d] 7 existing wells + 17 new onsite wells (6 shallow, 10 intermediate, and 1 deep) = 24 samples.

Table 4-2
Rationale For Proposed RI Monitoring Well Locations And Groundwiter Sample Analyses

Location Reference Number(s)	Sample Type(s)	Location Rationale	Analytical Suites	Analytical Rationale
1	SGW	Delineate the presence, nature, and extent of possible shallow groundwater contamination east (hydraulically downgradient) of former surge pond.	A	Characterize full spectrum of potential contaminants.
2, 3, 4, 5	SGW	Delineate the presence, nature, and extent of possible shallow groundwater contamination hydraulically downgradient of former sludge drying beds.	A C (4 only)	Characterize full spectrum of potential contaminants. Support potential site remediation planning activities.
6	SGW	Delineate the presence, nature, and extent of possible shallow groundwater contamination east of former surge pond—high VOCs and BNAs detected in this area—and near area of former acid spill.	A C	Characterize full spectrum of potential contaminants. Support potential site remediation planning activities.
7, 8	SGW	Delineate the presence, nature, and extent of possible shallow groundwater Contamination west (hydraulically downgradient) of former surge pond.	A	Characterize full spectrum of potential contaminants.
9, 10	SGW	Delineate the presence, nature, and extent of possible shallow groundwater contamination west (hydraulically downgradient) of former sludge drying beds.	A C (9 only)	Characterize full spectrum of potential contaminants. Support potential site remediation planning activities.
11 (Recovery Well 3, see Figure 4-3)	SGW	Delineate the presence, nature, and extent of possible shallow groundwater contamination north (hydraulically downgradient) of former sludge drying beds.	A C	Characterize full spectrum of potential contaminants. Support potential site remediation planning activities.

Table 4-2
Rationale For Proposed RI Monitoring Well Locations And Groundwater Sample Analyses

Location Reference Number(s)	Sample Type(s)	Location Rationale	Analytical Suites	Analytical Rationale
13, 14, 15	IGW	Delineate the eastern extent of possible intermediate depth groundwater contamination. Proposed wells are located downgradient of former sludge drying beds, former surge ponds, end intermediate and polishing ponds.	A C (14 and 15)	Characterize full spectrum of potential contaminants. Support potential site remediation planning activities.
12, 17	IGW	Delineate the southern extent of possible intermediate depth groundwater contamination. Proposed wells are located cross-gradient from intermediate pond and polishing ponds.	A	Characterize full spectrum of potential contaminants.
16	IGW	Delineate the presence, nature and extent of possible intermediate depth groundwater contamination east (hydraulically downgradient) of the former surge pond.	A B C	Characterize full spectrum of potential contaminants. Satisfy RCRA requirements. Support potential site remediation planning activities.
18	IGW	Delineate the western extent of possible intermediate depth groundwater contamination. Proposed well is located hydraulically upgradient from intermediate pond.	A	Characterize full spectrum of potential contaminants.
19, 20	IGW	Delineate the western extent of possible intermediate depth groundwater contamination. Proposed wells are located hydraulically upgradient of former sludge drying bede.	A C (19 only)	Characterize full spectrum of potential contaminants. Support potential site remediation planning activities.
21	IGW	Delineate the northern extent of possible intermediate depth groundwater Contamination. Proposed well is located hydraulically cross-gradient from former sludge drying beds.	A	Characterize full spectrum of potential contaminants.

Table 4-2
Rationale For Proposed RI Monitoring Well Locations And Groundwater Sample Analyses

Location Reference Number(s)	Sample Type(s)	Location Rationale	Analytical Suites	Analytical Rationale
22	DGW	Delineate the presence, nature, and extent of possible deep zone groundwater contamination. Proposed well clustered with a shallow and intermediate well located cross-(shallow) and down-(intermediate) gradient of the former surge pond	A C	Characterize full spectrum of potential contaminants. Support potential site remediation activities.

Source: Ecology and Environment, Inc., 1992.

Notes:
Sample Types
SGW = Groundwater sample from shallow monitoring well;
IGW = Groundwater sample from intermediate monitoring well;
DGW = Groundwater sample from deep monitoring well.

Analytical Suites:
A = TCL VOCs; TCL base-neutral/acid extractable organic compounds (BNAs); TCL pesticides and polychlorinated biphenyls (PCBs); TAL metals (total [i.e., unfiltered] and dissolved [i.e., millipore-filtered], water only); cyanide; gross alpha, beta, and gamma radioactivity; total organic carbon; total Kjeldahl nitrogen (TKN; surface water and sediment only); grain size (sediment only); hardness (water only); and alkalinity (water only).
B = Appendix IX VOCs, BNAs, dioxins, pesticides, PCBs, organophosphorus pesticides, herbicides, total metals, cyanide, and sulfide.
C = Total suspended solids, TKN (groundwater only), ammonia nitrogen, orthophosphate phosphorus, dissolved oxygen (in field), 5-day biological oxygen demand (BOD_5), and chemical oxygen demand (COD).

41 Sampling Objective

The objective of the field sampling plan is to outline a feasible means of sample collection for chemical and physical analysis.

Objective of sampling plan:

- To delineate the extent of soil, sediment, groundwater, surface water, and air contamination.
- To delineate migration pathways of the contaminants.
- To identify potential receptors of the contaminants.

4.2 General Sampling Requirements

The general procedures that field personnel are to follow when collecting soil, sediment, groundwater, and surface water samples are included in this section. Detailed sampling procedures are discussed in Section 4.5 of this SAP. The procedures are designed to prevent cross contamination of the samples.

General sampling procedures:

- All sampling activities will be conducted so that the sampling order will be from the area of least contamination to the area of most contaminated in each medium. If free product or contaminant saturated media are found to exist, grab samples will be collected from that area.

- The preferred order of sample collection will be as follows (on a parameter basis): volatile organic compounds, total organic carbon, extractable organics (including pesticides, herbicides, and polychlorinated biphenyls), total metals, dissolved metals, cyanide, inorganics, and turbidity.

- A clean pair of protective gloves will be donned by the sampler before each sample collection.

- Sample collection will be performed with either disposable sampling devices or decontaminated, stainless steel devices. When required, composite samples will be homogenized in stainless steel bowls. All sampling equipment will be decontaminated in accordance with the procedures outlined in Section 4.6 of this SAP.

- **Disposable sampling equipment shall be constructed of Teflon or PVC. The device shall be decontaminated by the manufacturer prior to shipment to the site. If disposable sampling devices are used, an equipment rinsate blank will be collected before use.**

- Samples collected for volatile organic compound analysis will not be homogenized.

- Sample containers for samples thought to be highly contaminated will be separated from the samples containing trace amounts of contaminants.

- Sample containers will be precleaned and provided by the analytical laboratory, except for stainless steel sleeves used for soil sampling which will be decontaminated onsite.

- All disposable sampling equipment will be double bagged and placed in a trash receptacle for disposal.

4.3 Sample Processing

After collection, samples will be preserved, labeled, packed and prepared for shipment in the following manner:

- Some of the analyses that will be performed on selected samples require that the samples be preserved as soon as they are collected in the field to maintain their integrity. Each sample chemically preserved will be clearly identified as such on the sample label. Specific sample preservations are detailed in Section 4.7.1 and Appendix C of this SAP. All samples will be preserved to 4° C immediately after they are collected and during shipment to the laboratory. A 40 milliliter (ml) vial of tap water will be used as a temperature blank to document sample receipt temperature.

- Samples will be handled as infrequently as possible. Extreme care will be taken to ensure that samples are not cross contaminated. Sealable plastic bags will be used to protect samples from cross contamination. A trip blank, prepared by the laboratory and shipped with the sample containers, will be held and shipped with each set of samples to be analyzed for VOCs. Headspace or bubbles will be avoided in all VOC samples:

- All samples will be identified and fully documented in the field records, on the chain-of-custody forms, and the sample labels. Specific instruction on the completion of the sample labels and chain-of-custody forms are provided in Sections 4.7 and 4.8 of this SAP. Examples of the labels and chain-of-custody forms are contained in Appendix D. Any samples that are thought to be potentially hazardous will be identified as such in the field records, on the chain-of-custody records, and on the sample labels. Documentation will be performed in accordance with Section 7.0 of this SAP.

- Chain-of-custody procedures will be followed to assure that the samples are maintained in a safe and reliable manner and to assure that each step in transportation to the laboratory is documented. This process will be implemented in the field and followed throughout the duration of the sampling process. An example of the chain-of-custody form is provided in Appendix D of this SAP. Chain-of-custody documentation will be perfomed in accordance with the procedures described in Section 4.8 of this SAP.

- All samples will be shipped overnight after collection via express air courier. Any samples that are considered to contain hazardous materials will be shipped in accordance with Title 49, CFR. Specific procedures for sample shipment are provided in Section 4.7.2. Air bill numbers will be recorded on the chain-of-custody forms.

- VOC samples that effervesce due to dissolved gases or high carbonate or bicarbonate content, will not be preserved with HCl if proper notification is given to the laboratory.

4.4 Collection of Auxiliary Data

All auxiliary data relative to a particular sampling location will be collected in conjunction with the sampling activities. Auxiliary data may include field measurements of pH, specific conductivity, temperature, salinity and flow rate. Photographs of sampling events will be collected to keep records of all site activities. Field information will be recorded in the field logbook. All other pertinent information (e.g, water levels, boring logs) will be recorded on the appropriate field forms or in the field logbook.

4.5 Specific Sampling Procedures

The following section describes the sample procedures that will be followed by field personnel during the site investigation. All sampling procedures to be used at NAS Pensacola will comply with EPA approved practices outlined in the SOP/QAM.

4.5.1 Surface Water Sampling

Surface water sampling will be conducted in accordance with Section 4.8 of the SOP/QAM manual. To assess the surface water pathway for contaminants to migrate offsite, the variables to be accounted for include:

- Weather
- Tide
- Flow Rate (Q = VA) where:

Q	=	flow
V	=	velocity
A	=	cross sectional area

- Bottom type
- Vegetation/Biota

The first step in the surface water sampling is to study the hydrodynamics of the drainage ditch to determine the sample locations. The hydrodynamics of the drainage ditch will identify areas of collection, flow, discharge and the influence of the tide. Water flow will be measured twice daily at both high and low tide during sampling. The measurements will be recorded in the field logbook. The sample collection points will be determined and samples will be collected in accordance with the procedures outlined in Appendix E. The samples will be collected at the selected locations during three events. These events will be conducted at high, intermediate, and low tides. A rain gauge will also be installed at or near the sample locations to document the rainfall influence. At each sample point, field measurements of pH, specific conductance, temperature, and salinity will be collected.

4.5.2 Sediment Sampling

Sediment samples will be collected using a stainless steel hand auger at the same sample locations as the surface water samples. Sediment sampling will be conducted in accordance with Section 4.8 of the SOP/QAM manual. Specific procedures to be followed for sediment sample collection are provided in Appendix F.

4.5.3 soil sampling

Soil sampling locations will be based on the results of the soil gas survey. The soil gas survey will indicate areas of elevated soil gas. These areas shall be further investigated by redefining the survey grid to a smaller interval. The intervals shall be decreased to 10 feet

and additional soil gas readings shall be collected. The survey shall be decreased to smaller intervals as needed. Soil borings shall then be advanced at the hot spot locations. Additional soil borings shall be advanced in areas of low readings to obtain random coverage of the subject property. A decision tree showing the criteria for selecting the soil boring locations is shown on Figure 4-1. Soil samples will be collected as outlined below.

Surface Soil Sampling

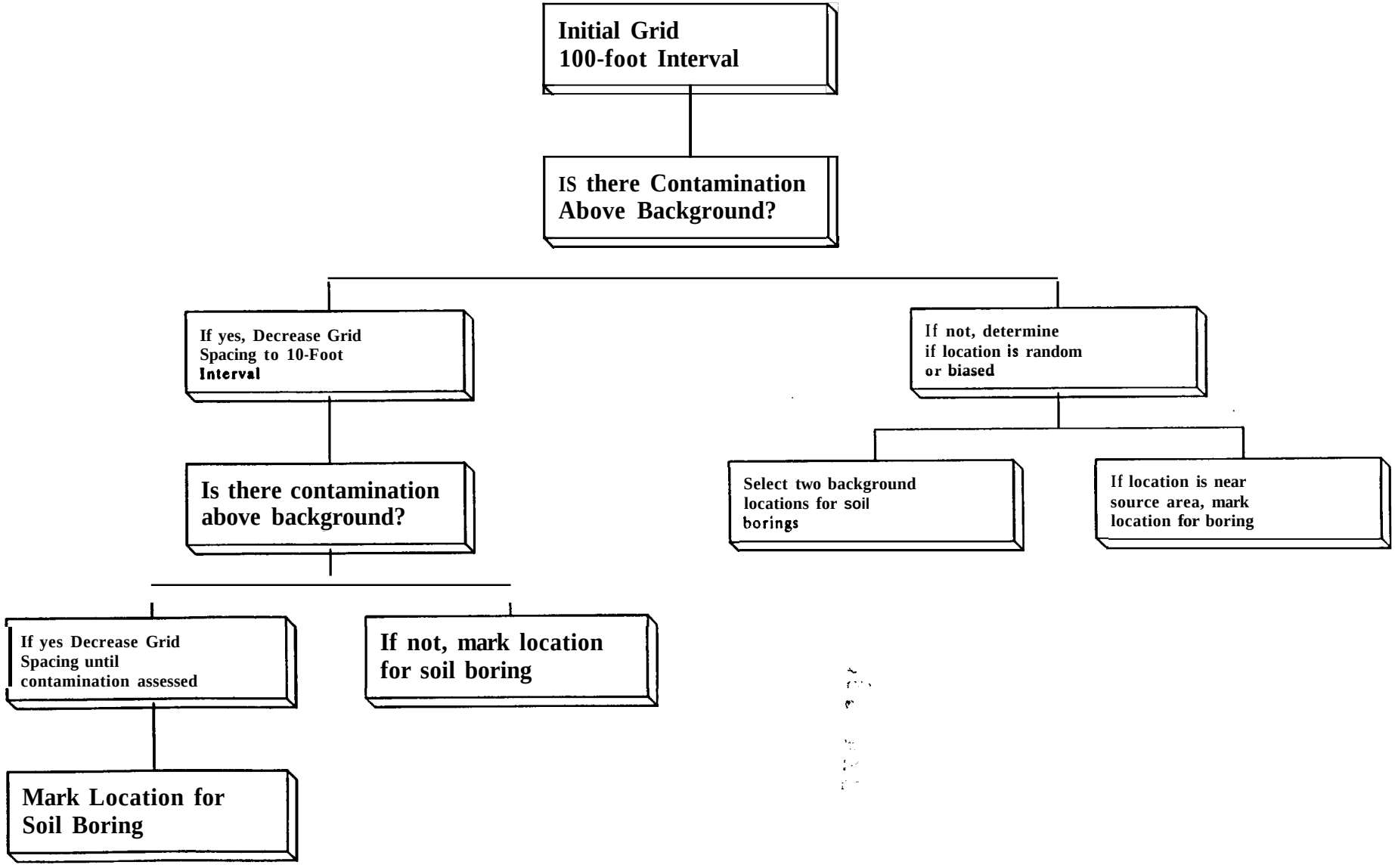
Two surface soil samples will be collected from areas of elevated OVA readings, based an the results of the preliminary air survey. Three additional surface soil samples will be collected from locations with nonelevated readings to determine background levels. Soil sampling will be conducted in accordance with Section 4.11 of the SOP/QAM manual. Surface samples are defined as 0-1' exclusive of rocks, twigs, leaves, and vegetation.

Subsurface Soil Sampling

Drilling Methods — Soil borings will be advanced using hollow stem auger drilling methods in accordance with the procedures outlined in Appendix E.2.1 of the SOP/QAM manual. The augers will be advanced without a bottom plug to facilitate split barrel sample collection. Locations of the soil borings will be determined based on the results of the soil gas survey. After soil samples are obtained to the proper depth, the soil borings will be abandoned with neat cement grout.

Soil Sampling Procedures — Soil samples will be collected continuously to the water table using a 2-inch inside diameter split barrel sampler with stainless steel liners. The split barrel sampler will be 2 feet long and will contain four 6-inch liners. The split barrel sampler will be pushed until the full length of the sampler has been driven or until refusal. Upon sampler retrieval, soil samples will be screened for ionizable organic vapors using a photoionization detector (PID) or a flame ionization detector (FID). The two liners with the highest readings will be covered with Teflon film at each end and capped. If additional samples are needed for

Figure 4-1
Pensacola Operable Unit 10
Decision Tree for Soil Boring Locations



quality control samples (e.g., duplicates, matrix spike samples, matrix spike duplicates) the other two liners will be covered and capped. The soil samples will be labeled and **preserved to 4° C.** The specific sampling procedures *are* provided in Appendix G.

During drilling of the proposed deep monitoring wells, two 2-foot long Shelby tube samples of the low permeability zone will be attempted. The Shelby tubes will be collected in accordance with the procedures in Appendix H. The samples will be submitted for analysis of permeability and grain size.

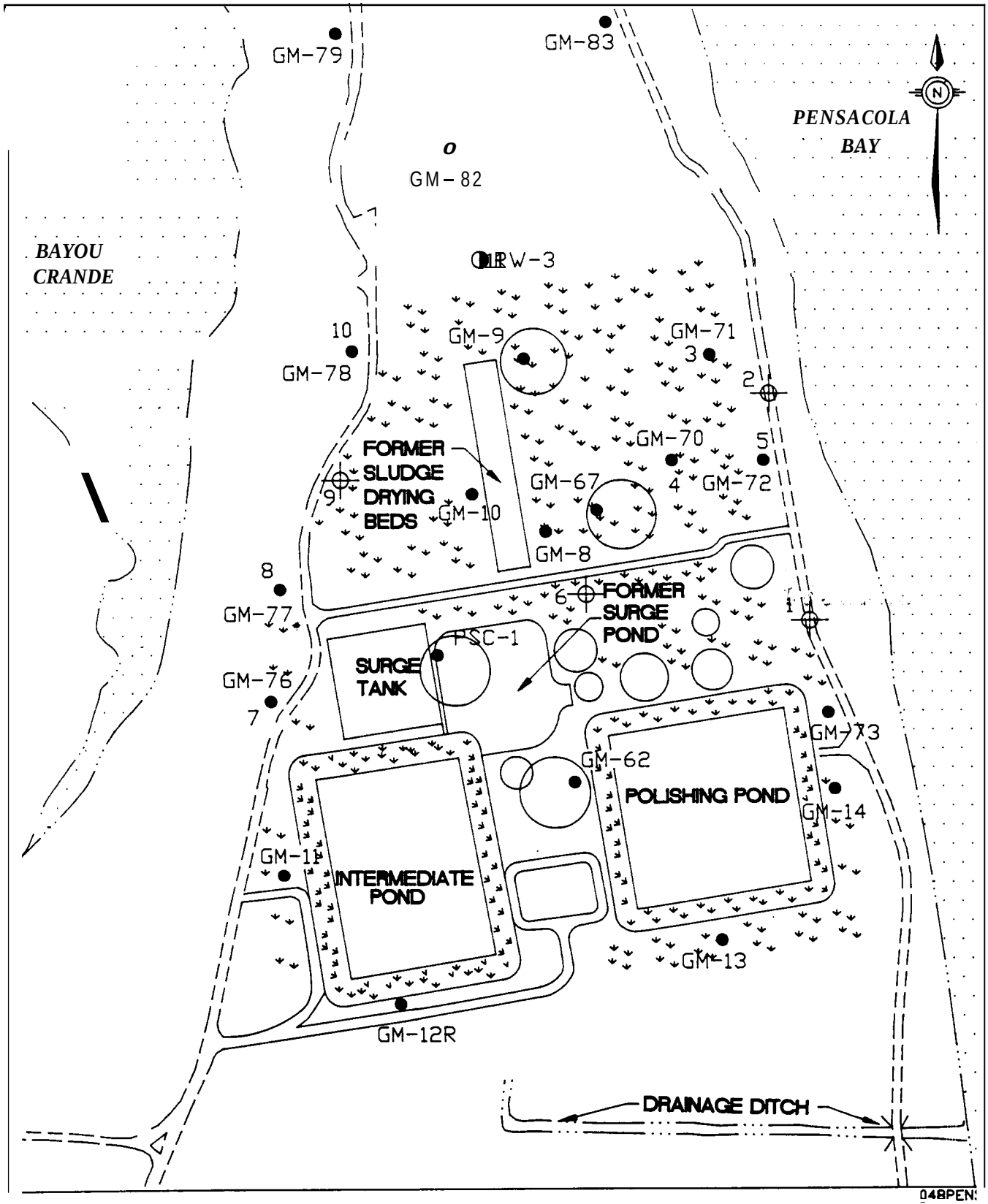
4.5.4 Groundwater Sampling

Drilling Methods

The soil borings to be advanced for monitoring well installation will be advanced using hollow stem auger drilling methods with a PVC plug in accordance with the procedures outlined in Appendix E.2.1 of the EPA SOP/QAM manual. The plug is necessary to keep the augers clear while drilling in saturated soils.

Monitoring Well Construction

Seventeen monitoring wells will be installed at the Group O sites as part of this investigation. The proposed locations of the monitoring wells are shown in Figures 4-2 and 4-3. Six of the monitoring wells will be shallow, approximately 20 feet depth. Ten of the monitoring wells will be completed at an intermediate depth of approximately 40 feet, and one deep monitoring well will be completed to approximately 65 feet. The intermediate and deep monitoring wells will be nested with shallow monitoring wells for calculation of vertical hydraulic gradient. All of the monitoring wells will be constructed in accordance with the procedures outlined in Appendix



SOURCE: GERAGHTY & MILLER INC., 1988

SCALE 0 300 600

● SHALLOV MONITORING WELL (12-15 FEET BLS)

CLUSTERED WELL SITES

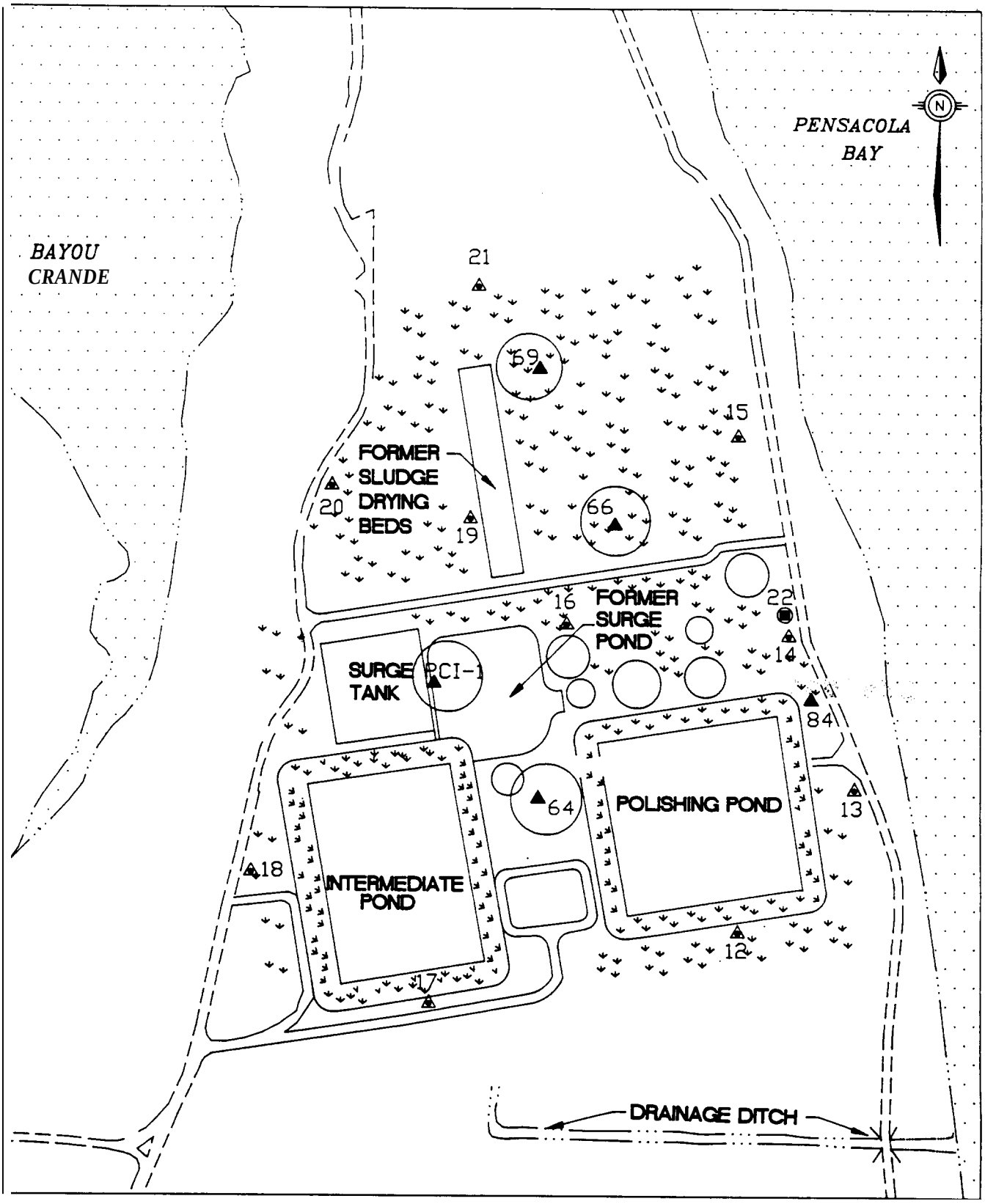
GRASS

2 LOCATION REFERENCE NUMBER

NOTE: TWO ADDITIONAL SHALLOW WELLS WILL BE INSTALLED IN LOCATIONS BASED ON THE SOIL GAS SURVEY RESULTS

TENTATIVE MONITORING WELL LOCATION (SEE NOTE)

FIGURE 4-2 SHALLOW MONITORING WELL AND SAMPLING LOCATIONS NAS PENSACOLA SITES 32,33 AND 35



SOURCE:GERAGHTY & MILLER INC.,1988

048FI4-2

▲ INTERMEDIATE MONITORING WELL (40-40 FEET BLS)

CLUSTERED WELL SITES

GRASS

TENTATIVE INTERMEDIATE MONITORING WELL LOCATION

12 LOCATION REFERENCE NUMBER

0 300 600

SCALE

TENTATIVE DEEP MONITORING WELL LOCATION

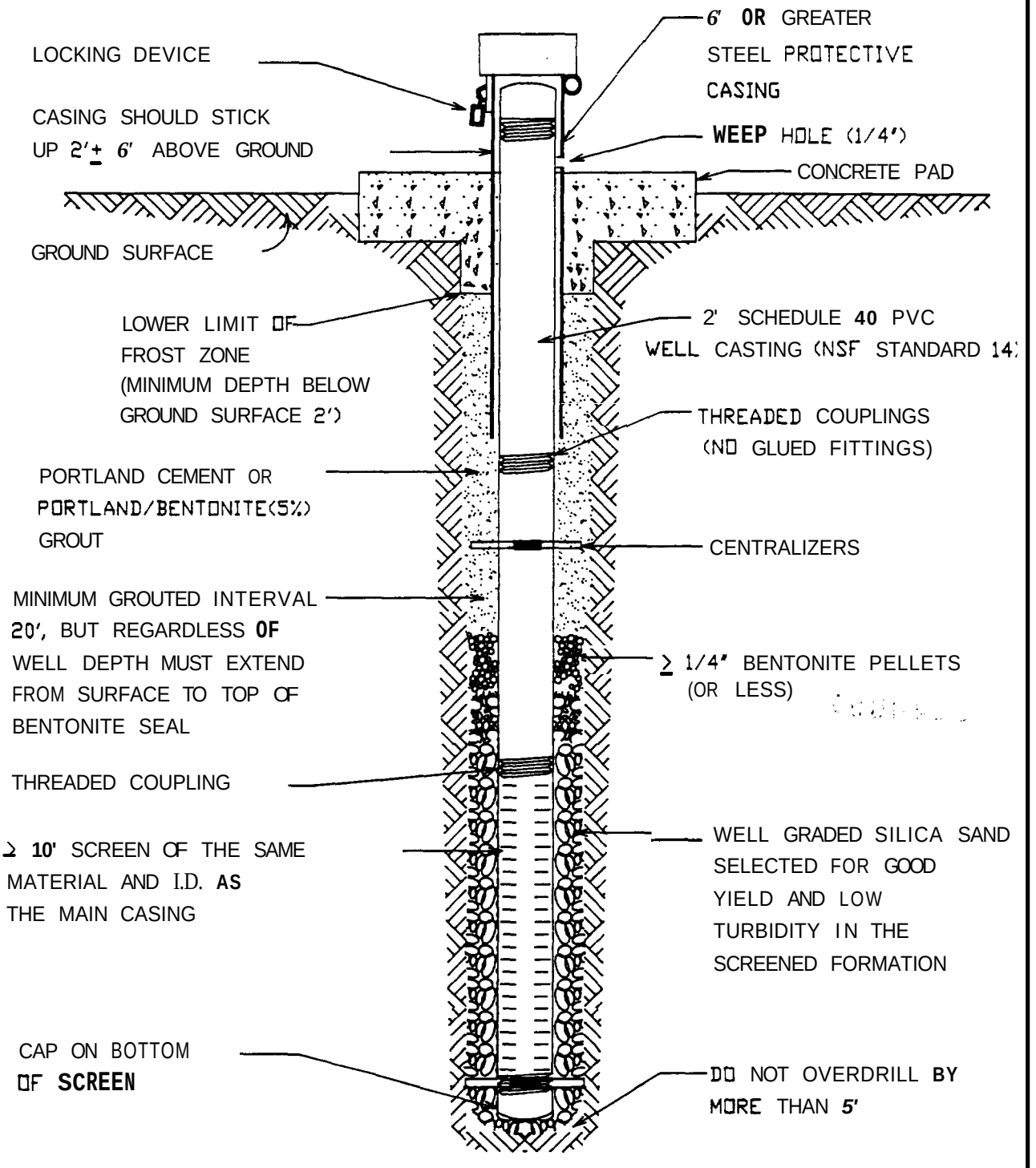
FIGURE 4-3 INTERMEDIATE *AND* DEEP MONITORING WELL SAMPLING LOCATIONS NAS PENSACOLA SITES 32,33 AND 35

E.4.1 and E.4.2 of the SOP/QAM guidance manual with the following modifications. A typical monitoring well construction diagram is provided in Figure 4-4. In **accordance with the approved work plan for Operable Unit 10, all of the monitoring wells will be constructed of 2-inch inside diameter PVC materials. The PVC well construction materials shall be NSF Standard 14 in addition to ASTM Schedule 40. The PVC materials shall not be solvent** rinsed **or cleaned with hot water.** The intermediate and deep monitoring wells will be installed into the impermeable zone of the surficial aquifer using a surface casing. The surface casing will be grouted in place and allowed to set for 24 hours before the monitoring well drilling continues. **The intermediate and deep PVC monitoring wells will be grouted with bentonite grout.** After monitoring well installation, a point will be selected on the top of the inner casing and marked. This point will be the reference mark for elevation measurement. To reduce the risk of cross-contamination, the outer protective casings of the monitoring wells will not be painted. Detailed monitoring well construction procedures are provided in Appendix I.

Monitoring Well Development

Monitoring wells will be developed using a combination of surging and pumping in accordance with Appendix E.7 of the SOP/QAM manual. The monitoring wells will be developed until the groundwater withdrawn is below 0.5 milliliters per liter (ml/l) of settleable solids in 15 minutes using an Imhoff Cone or as free of turbidity as possible given the subsurface conditions. The groundwater withdrawn will also be monitored for specific conductance, pH, and temperature to stabilize to within ± 5%, ± 0.5, and ± 1° C respectively or to within the equipment manufacturer's specifications. All equipment will be calibrated according to the manufacturer's specifications before use onsite. Monitoring well development procedures are described in Appendix J.

TYPICAL ILLUSTRATION OF MINIMUM SPECIFICATIONS FOR SHALLOW MONITORING WELL CONSTRUCTION



SAMPLING AND ANALYSIS
NAS-PENSACULA
PENSACOLA,FLORIDA

FIGURE 4-4
TYPE II
MONITORING WELL DESIGN

DATE: 10/10/92 | DWG NAME: 048TYPE2

Monitoring Well Sampling

Following the installation of the new monitoring wells, the monitoring wells will be purged of static water and groundwater samples will be collected from the newly installed monitoring wells and the existing monitoring wells GM-70, GM-71, GM-72, GM-76, GM-77, and GM-78, and the existing recovery well RW-3. Groundwater samples will be collected in accordance with Section 4.9 of the SOP/QAM guidance manual with the following additions and modifications.

Purging Static Water — Prior to purging, the depth to water and the total well depth will be measured with an oil/water interface probe. The interface probe will be used to check for floating or sinking immiscible hydrocarbons. The interface probe will be decontaminated prior to use onsite and between monitoring wells. The static volume will be calculated using the. formula presented in Section 4.9.7.2 of the SOP/QAM guidance manual. Well headspace will be monitored by PID or FID and recorded in field logbook. The monitoring wells will be purged using a pump or Teflon bailer. At least three well volumes will be removed before sample collection. Purging will continue until three readings of specific conductance, temperature, and pH stabilize to within ± 5%, ± 1°C, and ± 0.5 standard units respectively. Information concerning well purging will be recorded in the field logbook and on the groundwater sampling log. *An* example of the groundwater sampling log is provided in Appendix D, and specific procedures for purging are provided in Appendix K.

Sample Collection — Groundwater samples will be collected in accordance with the procedures in Section 4.9 of the SOP/QAM manual with the following modifications. Groundwater samples will be collected as soon as recharge of the monitoring well allows. Samples will be collected using Teflon bailers to reduce the loss of volatile constituents. Temperature, pH, and specific conductance will be measured and recorded for each sample collected. Samples will be chemically preserved as **specified** in **Appendix C.** Sample collected for metals analysis will not be filtered. After chemical preservation and labeling, the samples will be **preserved to 4° C.**

Weather conditions at the time of sample collection will be noted in the field logbook. Specific procedures for groundwater sample collection are provided in Appendix L.

4.5.5 Air Contaminant Quantification Survey

The preliminary air survey will determine the need for an air contaminant survey. A contaminant quantification survey, if necessary after the preliminary air survey, will be performed with stainless steel Summa canisters for whole air sample collection. Samples will be collected in the breathing zone unless conditions warrant special placement of the canisters. Operation of the canisters will be in accordance with the procedures provided in Section 4.13.2 of the SOP/QAM guidance manual and in accordance with the procedures provided in the EPA document *Compendium Method To-14: The Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Summa Passivated Canister Sampling and Gas Chromatographic Analysis.* The document is provided in Appendix M. The sampling system will be calibrated before and after the sampling period according to manufacturer's specifications.

Prior to sample collection, the vacuum of the canisters will be checked and recorded in the field logbook. The vacuum should equal the vacuum recorded in the laboratory prior to Canister shipment. If the canister is to be evacuated onsite, the pressure should be < 0.05 mm of mercury. A determination must be made regarding the required sample volume. Passive canisters which contain approximately 6 gallons of sample allow filling to approximately two-thirds their volume. If greater volume is needed, a pressurized sample collection system is needed.

In addition, a determination must also be made of the flow rate of the canister before sample collection. Canisters should not be filled to less than 88.1 kPa or greater than 202 kPa (for pressurized), over the sample period.

The laboratory supplying the canisters will provide documentation of canister cleaning and that the canisters are in proper working order. If the samples collected are believed to be contaminated, the canisters will be marked. This will also be noted on the chain-of-custody to allow the laboratory to determine the proper decontamination procedures.

The following information will be documented in the field logbook for each of the samples collected:

- Project Location
- Date
- Sample # and Location
- Temperature
- Barometric Pressure
- Relative Humidity
- Time at beginning and end of sample collection (using a 24-hour clock)
- Total lapsed time of sample collection

Other meteorological conditions such as wind direction and speed, rainfall amounts, cloud cover, etc., will be recorded in the field logbook.

4.5.6 Hydrologic Assessment

The objective of the hydrologic assessment is to better delineate the subsurface materials underlying the Group O sites. The hydrologic assessment of the Group O sites will include a elevation survey of the newly installed monitoring wells; water level measurements in all of the onsite monitoring wells, and/or adjacent surface water bodies; pumping tests of one existing recovery well (RW-5) and a proposed intermediate monitoring well (16); and slugs tests on the newly installed monitoring wells.

All newly installed monitoring wells will be surveyed during the engineering survey. The wells will be referenced to a USGS benchmark. In addition, staff gauges will be placed in the surrounding surface water bodies and surveyed relative to the same benchmark.

Static water levels will be measured at all monitoring wells located on the Group O site complex. Static water level measurements will be collected during four events. One set of water levels will be collected during the sampling of the newly installed monitoring wells, another set of water levels will be collected two to four weeks after the sampling, and the third and fourth sets will be collected during high and low tide. The third and fourth sets will be collected on the same day to determine tidal influences on groundwater elevations. 'The water levels will be used to determine the groundwater elevations for determination of groundwater flow direction and calculation of the hydraulic gradient.

Multi-well pump tests will be conducted on one existing recovery well (RW-5) and one proposed intermediate monitoring well (well location no. 16). The pump test will involved withdrawing groundwater at a constant, known rate from the designated well until the water level stabilizes. Water levels will be simultaneously measured in designated adjacent monitoring wells completed in the same zone. In addition, water levels in adjacent monitoring wells completed to different depth zones will also be measured. The tests will be conducted for at least 8 hours, or until observable drawdown has occurred in adjacent wells. The recovery of the water levels in the production and monitoring wells will be monitored after the pump is turned off, until the water level reaches the initial water level. Drawdown and recovery data from each pump test will be plotted to produce time versus drawdown graphs. The graphs will be used to calculate hydraulic conductivity, transmissivity, and groundwater flow velocity for each zone. Possible methods to be used for determining these parameters are presented in Theis (1935), Lohman (1972) and Bouwer and Rice (1976).

For all of the newly installed monitoring wells, slug tests will be conducted. Falling head and rising head slug tests will be conducted in accordance with the procedures provided in Appendix N. Aquifer parameters to be obtained from the tests include hydraulic conductivity (K), transmissivity (T) and groundwater flow velocity (V). Hydraulic conductivity will be determined using equations provided in Bouwer and Rice (1976) or Cooper e? *al.* (1967). Groundwater velocity will be calculated using a derivation of Darcy's Law:

$$V = \frac{Ki}{n_e}$$

K	=	hydraulic conductivity (ft/day)
i	=	hydraulic gradient (ft/ft)
n_e	=	effectiveporosity

4.5.7 Engineering Survey

When monitoring well installation is complete, a comprehensive cadastral survey will be conducted. All sampling locations will be accurately located horizontally and vertically. The vertical elevations will be surveyed to an accuracy of f0.01 foot. The elevations will be referenced to a spot elevation benchmark located in the northern portion of the site complex, or to a USGS benchmark location on Chevalier Field which is located south of the Group O sites. The horizontal locations will be supplied in latitude and longitude, in accordance with NAD 83. In addition, all utilities, above ground structures, surface improvements (e.g., pavement) will be located.

4.6 Decontamination

Decontamination procedures will be performed in accordance with Appendix B, Section B-8 of the SOP/QAM manual with the following exceptions. The detergent for use on this project will be Liquinox which contains powerful chelating agents to bind and remove trace metals from sampling equipment. When available, hot water will be used for field decontamination. PVC well construction materials will not be solvent rinsed or washed with hot water. Field reagent grade water will meet the specifications of ASTM type III water (D 1193-77 reapproved 1983, Federal test method number: 7916), which provides deionized, filtered, and organic free water for field use. A summary of procedures for decontamination are provided in Appendix O.

4.7 Sample Management

4.7.1 Containers, Preservatives, Holding Times

The volumes, types of containers and preservatives required for the sampling activities are listed in Appendix C. Sample containers and preservatives will be selected in accordance with Appendix A of the SOP/QAM manual. All of the glass containers will have Teflon-lined caps. Soil samples will be collected using stainless steel sampling sleeves.

The sample containers will be obtained from an approved laboratory. The laboratory will prepare and ship the appropriate number of trip blank samples with the sample containers. Stainless steel sampling sleeves to be used for soil sample collection shall be obtained from the manufacturer and decontaminated in the field before use.

4.7.2 Packaging for Shipment

All samples will be packaged for shipment to avoid breakage and prevent cross contamination. General procedures for packaging are provided below.

Packaging Procedures:

- All sample lids will stay with the original containers.

- To determine if any leakage has occurred during shipment, the laboratory will assume that all bottles were filled completely. If the sample volume is lower than the shoulder of the container, the top of the volume will be marked with a permanent marker or grease pencil.

- Ice in sealable plastic bags will be used to cool samples to 4° C.

- Samples will be placed in the shipping containers so that the bottles do not touch each other.

- Any remaining space will be filled with inert packing material. Vermiculite or cellulose insulation are generally used.

- A chain-of-custody record will be placed in a plastic bag and sealed inside each shipping container detailing its contents.

- The container shall be sealed with tape and custody seals in such a manner that the container cannot be opened without breaking the seal. The drain on the cooler will be taped shut both inside and outside to prevent leakage.

- The words "This End Up" or "This Side Up" will be clearly printed on the top of the outer container. Upward pointing arrows should be placed on the sides of the package.

4.7.3 Labeling

Samples will be identified in accordance with Section 3.0 of the SOP/QAM manual with the modifications listed below. A standard sample label is presented in Appendix D. The label will be attached to the sample container. The following information shall appear on the sample label:

A Joint Venture for Professional Services
EnSafe/Allen & Hoshall
(901) 383–9115

Sample Number:
Preservative:
Analysis:
Date: _____
Time: _____
Sampler:

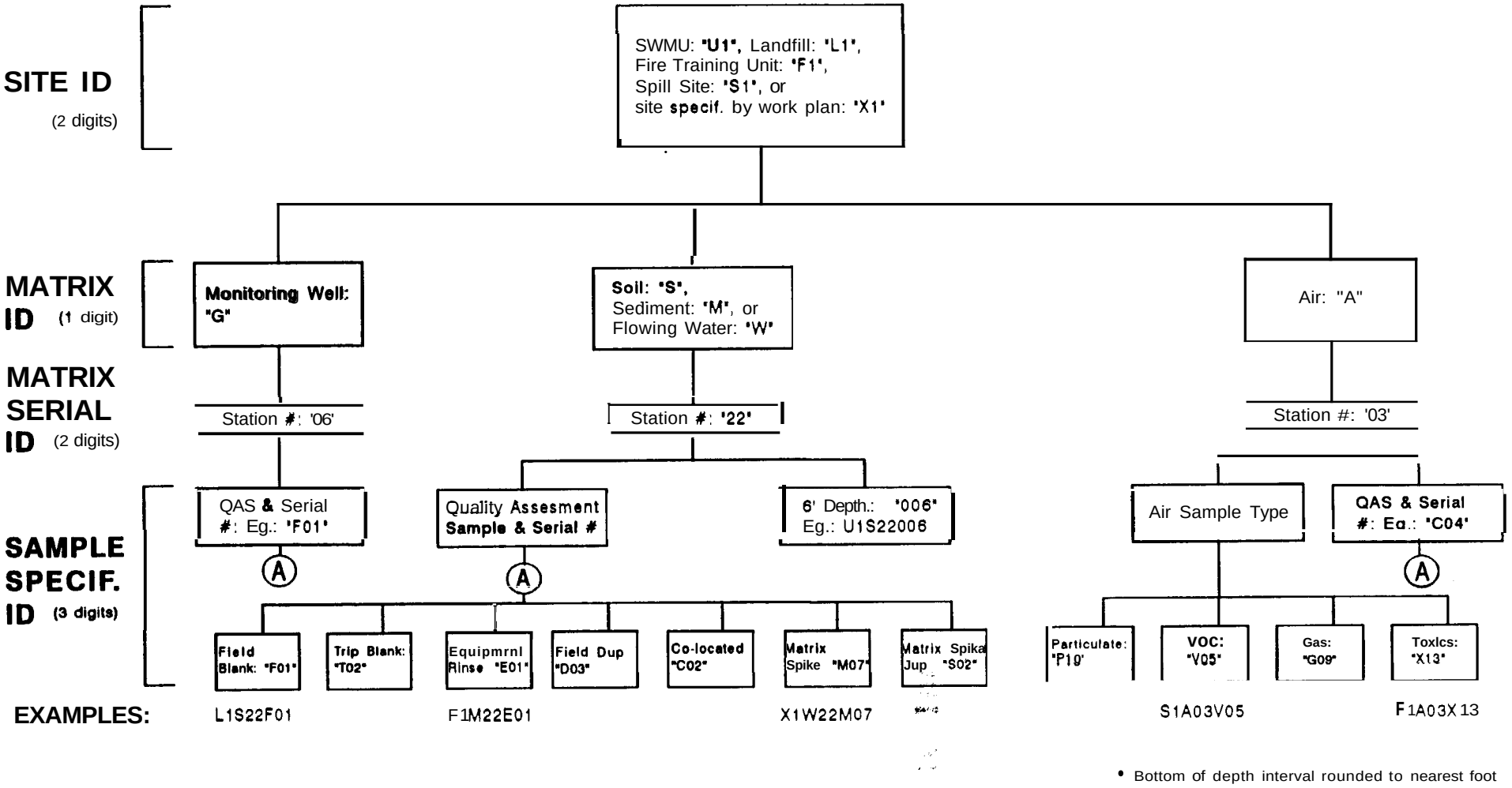
Any errors made on the sample label will be corrected by placing a single line through the error. The error will then be initialed by the sample collector and the correct information will be entered on the sample label. All of the information except for the date, time, and sample collector's initial will be preprinted on the labels. The remaining information will be placed on the label in non-erasable, waterproof, black ink.

Sampling Identification Number

The sample identification number will be assigned using the system provided in Figure 4-5 and shall not exceed eight digits. The site ID number will be numeric and will be the first two digits of the sample identification number. Because of the close proximity of sites 32, 33 and 35, the site ID shall be "33".

Figure 4-5
NAS Pensacola Operable Unit 10

SAMPLE NUMBERING SYSTEM FOR COMMON MATRICES



• Bottom of depth interval rounded to nearest foot

The sample matrix will be represented by the third digit. Some common sample matrices are given below:

G — groundwater
S — soil (includes surface soil sample and soil borings)
M — sediment (defined as fluid borne solid that has settled)
W — surface water
A — air

Other codes may be utilized when the above codes are not applicable. However, the sample matrix will be limited to one digit to allow for other information. The matrix serial ID will be the next two digits and will be the sample collection ID number. For example, if a soil sample were collected at boring B-12 at Site 33 the identification would be "331312."

The next three digits of the sample identification number will be a sample specific identitication number. The deepest sample depth rounded to the nearest foot will be used for sample identification. For example, if a soil sample is collected from 18 to 20-feet depth at soil boring B-4 at Site 33, the sample number would be "33S04020."

The three digits will also be used to designate quality assurance samples. The first digit will designate the type of quality assurance sample. Common designations *are* presented below:

F — field blank
T — trip blank
E — equipment rinsate samples
D — duplicate samples
C — co-located samples
M — matrix spike samples
S — matrix spike duplicate samples

The last two digits will be used as serial numbers to differentiate between quality assurance samples. For example if two field blank samples were collected while collecting soil samples

at boring 22 at Site 32, they would be designated "33S22F01" and "33S22F02." All of the sample identification number shall also be placed on the chain-of-custody form as outlined in Section 4.8 of this SAP and in the field logbook.

4.8 Sample Custody

Custody of samples shall be performed in accordance with Section 3.3 of the SOP/QAM manual with the following modifications:

4.8.1 Field Custody

When samples are collected, they will be labeled and sealed with a custody seal. An example of the custody seal is provided in Appendix D. The seal shall be affixed so that samples cannot be opened without breaking the seal. The seal shall be signed and dated by the sample collector.

4.8.2 Chain-of-Custody Record

The field chain-of-custody form shall be used to record the custody of the samples. An example of the chain-of-custody to be used is provided in Appendix D.

The following information shall be provided in the appropriate spaces on the chain-of-custody form:

- Client which will be completed as E/A&H
- Address of E/A&H
- Project Name/Number (Pensacola/CTO Number)
- Project Manager's Name
- Signature of Sampler(s)
- The field sample number; date and time of collection (24-hour clock); type of sample; type and size of sample containers; preservation temperature and chemical; number of sample containers, and the type of laboratory analysis required.

- Any comments regarding the samples such as strong odor will be completed in the remarks section.
- When sample custody is transferred, the transfer must be documented on the appropriate Section of the chain-of-custody form. The person relinquishing custody shall print his or her name, company name, the reason for the custody transfer (e.g., shipment to the laboratory), date and time and sign the chain-of-custody record. The laboratory person receiving the samples shall also print his or her name, company name, date and time and sign the appropriate section of the chain-of-custody record.
- The method of shipment and the air-bill number of the shipment will be noted on the chain-of-custody form.
- During shipment, the chain-of-custody form shall be sealed in a plastic bag and secured inside the shipping container.
- Any special instructions (i.e. priority turnaround) or comments will be noted in the comments section of the chain-of-custody.
- Sample disposal or storage instructions after the completion of the laboratory analysis will be noted in the appropriate section of the chain-of-custody record.

4.8.3 Transfer of Custody

Transfer of custody and shipment of samples will be performed in accordance with Section 3.3.5 of the SOP/QAM manual. Shipping containers will be secured with tape and a custody seal shall be placed on the outside of the container to prevent opening without breaking the seal. Samples will be shipped overnight to the selected laboratory. The air-bill number will be noted in the appropriate section on the chain-of-custody record and in the field notebook. The chain-of-custody form shall be sealed in a plastic bag and placed inside the shipping container prior to closure.

Field personnel will make arrangements for transportation of samples to the analytical laboratory. When custody is relinquished to a shipper, field personnel shall telephone the

analytical laboratory regarding the expected arrival time of the shipment and any time constraints on sample analysis. The laboratory should be notified as early in the week as possible, and in no case later than 15:00 (Eastern time) on Thursday, regarding samples intended for Saturday delivery.

4.8.4 Laboratory Custody Procedures

A sample custodian accepts custody of the shipped samples from the carrier and enters preliminary information about the package into a receipt log, including the initials of the person delivering the package and the status of the custody seals on the coolers (i.e., broken or unbroken). The laboratory sample custodian is responsible for sample log-in and will open the shipping coolers, check the contents, and verify that the information on the chain-of-custody agrees with samples received. Pertinent information such as shipment, pickup, and courier must be entered into the "Remarks" section of the chain-of-custody record. The custodian will also document the temperature of the cooler (by checking the temperature blank) and the general condition of the sample containers.

Sample preservation shall be verified by the analyst before extraction, digestion or analysis and the pH recorded. If samples are found improperly preserved, the Laboratory QA coordinator will document the improper preservation along with the sample identification and other pertinent information. All other QA/QC discrepancies are followed in a similar manner and must be documented as an out-of-control event with the corrective action taken.

4.9 Investigation Derived Wastes

All investigation-derived waste (IDW) solids and liquids are to be handled in a manner consistent with all applicable local, state, and federal waste disposal guidelines. The disposal method will be selected by the Navy based on the classification of the waste as RCRA hazardous or RCRA non-hazardous. IDW will then be disposed in accordance with the applicable guidelines for that waste category.

The following guidelines will be utilized for IDW disposal:

soils

Soils will be considered unclassified wastes until receipt of the analytical data. Investigation derived soils will be drummed, labeled and transported to a waste storage facility by the Navy. Each label will include the site number, the date of collection, and the waste category. Upon receipt of the analytical results, the soils will be properly disposed. Only DOT-approved lid type drums will be used.

Water

All investigation derived water (e.g., decontamination, sample purging) will be drummed and labeled. The drums will be transported by Navy personnel to the IWTP for disposal. Only DOT-approved bung type drums will be used.

Isopropyl Alcohol

All investigation derived isopropyl alcohol shall be drummed separately and labeled. The drums will be transported by Navy personnel to a waste storage facility. The waste will be properly disposed. Only DOT-approved bung type drums will be used.

Personal Protective Equipment (PPE)

All decontaminated PPE and disposable equipment will be placed in 55-gallon drums for disposal by the Navy.

Documentation

A drum tracking log will be kept for the convenience of the Navy separate from field logbooks. This log will detail number of drums, site number, water type, and date filled.

4.10 Quality Assurance/Quality Control

All surface water, sediment, soil and groundwater samples will be collected in accordance with NEESA Level D (EPA Level IV) protocol. Field QA/QC samples will be collected in accordance with the Section 4.6 of the SOP/QAM manual and with the NEESA *Sampling and Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program*, June 1988 with the following modifications. A discussion of blanks is provided in Section 6 of this *SAP*. A summary of the number of blanks and duplicate samples to be collected was previously provided in Table 4-1. A preservative blank will not be collected. The laboratory will provide the appropriate preservatives with instructions for preservation. Because field blanks are preserved with the Same acid as a preservative blank, and the preservative blank is diluted with the sample reagent grade water as a field blank, no new information would be gained by collecting both a preservative and field blank. Distinction between contamination in the acid preservative or reagent grade water is impossible. The only way to test for preservative contamination under these circumstances would be to ship pure acid to the laboratory undiluted and to ship pure field reagent grade water to the labomtory unpreserved; neither of which solves the distinction problem. The laboratory will analyze each lot of acid used in the field for preservation.

A solid media field blank for VOC analysis will not be collected. A water temperature blank will be included in each cooler, and the reagent grade water will be sampled in the field once per week. **Equipment/materials blanks (i.e., filter sand, drilling mud, grout, bentonite, potable water) will be collected from all of the materials used in well construction. The samples will be archived until needed to verify the material quality.**

5.0 ANALYSIS

5.1 Field Measurements

QA objectives for parameters to be measured in the field by E/A&H personnel are presented in Table 5-1. Field measurements will include pH, temperature, specific conductance, salinity, groundwater level, well head survey, and organic vapor detection.

Table 5-1
Fidd Measurements QA Targets

Measurements Parameter	Reference	Matrix	Precision	Accuracy	Completeness
pH	EPA 150.1[a]	Water	± 0.5 S.U.	± 0.2 S.U.	90%
Temperature	EPA 170.1[a]	Water	± 01.° C	± 0.2° C	90%
Specific Conductivity	EPA 120.1[a]	Water	± 10%	± 1% of full scale	90%
Static Water Level	SOP[b]	Water	± 0.01 ft.	± 0.05 ft.	90%
Salinity	SOP[e]	Water	± 02. ppt	± 1.0 ppt	
PID/FID	SOP[c]	Air	± 10 ppm	± 2 ppm	90%
Well Head Points	SOP[d]	Spatial	± 5%	± 0.1 feet	90%
	SOP[d]	Vertical	± 0.05 feet	± 0.01 feet	90%

Notes:

- [a] — Methods for Chemical Analysis of Water and Wastes, EPA-600/4/79-020, Revised March 1903.
- [b] — Manufacturer's SOP for static water level measurement.
- [c] — Manufacturer's SOP for operation of Photovac Micro TIP.
- [d] — Standard Land Surveying Methods as employed by a State of Florida Registered Land Surveyor in accordance with NAD 83.
- [e] — Manufacturer's SOP for salinity measurement.

5.1.1 Equipment Calibration/Standardization

Instrumentation used to collect field measurements will be calibrated each day of operation according to manufacturer's specifications prior to use onsite. Instrumentation may also be checked for calibration during the day as it is deemed necessary by field personnel. The source of the field standards, date of standard receipt, lot number, and expiration date will be entered in the field logbook. The traceability of each standard to National Institute of Standards and Technology will be based upon this information and product-specific information provided by the standard manufacturer/supplier. Calibration/standardization procedures for instruments with specific applications are presented in Appendix P.

5.1.2 QA Targets for Precision & Accuracy

Precision and accuracy targets for pH, temperature, and specific conductivity measurements are those specified in Section 6 of the SOP/QAM. QA targets for other measurements are based on manufacturer's information pertaining to the precision and accuracy of the associated instruments.

5.2 Laboratory Analysis

5.2.1 Parameters and Methods of Analysis

The parameters of laboratory analysis are listed on Table 4-1. Analytical methods, extraction methods, sample containers, sample preservatives, and holding times are presented in Appendix C. Samples will be analyzed using the full CLP TCL/TAL analyses. Analysis will be performed in accordance with the applicable CLP SOW. The SOW for this investigation is CLP Organic and Inorganic Analysis, Statement of Work, March 1990. Additional references for parameters not included in the CLP analysis are provided in:

- Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, 40 CFR Appendix A to Part 136, July 1987;
- Test Methods for Evaluation Solid Waste (Physical/Chemical Methods), SW-846, Third Edition, September 1986; and

- **Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983.**

Samples will be analyzed for the selected parameters following the criteria established in Section 4 of this SAP.

5.2.2 QA Targets for Precision and Accuracy

Project QA objectives for analytical parameters for soil and groundwater will be stipulated in the respective analytical methods, and as determined by the analytical laboratory's historical data quality evaluation for these methods. The NEESA laboratory approval process will ensure that laboratory method QA/QC standards are appropriate to meet goals for intended data uses. Anticipated general QA goals for these methods are presented in Tables 5-2 and 5-3. The contract labomtory for this activity is CompuChem Laboratory of Research Triangle Park, North Carolina. The laboratory's NEESA-approved QAP will be retained for reference in the E/A&H library and onsite.

Table 5-2
Metals Analyses Data QA Objectives Matrix Spikes/Duplicate Analyses

Compound (Metal)	Medium/High Sample Precision[a] (%RPD)	Low Sample Precision[b] (%RPD)	Accuracy (% Spike Recovery)
Aluminum	± 20	± CRDL[c]	75-125
Antimony	± 20	± CRDL	75-125
Arsenic	± 20	± CRDL	75-125
Barium	± 20	± CRDL	75-125
Beryllium	± 20	± CRDL	75-125
Cadmium	± 20	± CRDL	75-125
Calcium	± 20	± CRDL	75-125
Chromium	± 20	± CRDL	75-125
Cobalt	± 20	± CRDL	75-125
Copper	± 20	± CRDL	75-125
Iron	± 20	± CRDL	75-125
Lead	± 20	± CRDL	75-125
Magnesium	± 20	± CRDL	75-125
Manganese	± 20	± CRDL	75-125
Mercury	± 20	± CRDL	75-125
Nickel	± 20	± CRDL	75-125
Potassium	± 20	± CRDL	75-125
Selenium	± 20	± CRDL	75-125
Silver	± 20	± CRDL	75-125
Sodium	± 20	± CRDL	75-125
Thallium	± 20	± CRDL	75-125
Tin	± 20	± CROL	75-125
Vanadium	± 20	± CRDL	76-125
Zinc	± 20	± CRDL	75-125
Cyanide	± 20	± CRDL	75-125

Notes: QA/QC Requirements excerpted from CLP SOW ILMO 1.0

a — Medium/High Samples = compound or metal present at 6 times (or more) the contract required detection limit (CRDL)[c]

b — Low Samples = compound or metal present at less than 5 timer the CRDL

Fraction	Compound	Soil Precision (%RPD)	Soil Accuracy (%Recovery)	Water Precision (%RPD)	Water Accuracy (%Recovery)
VOA	**Matrix Spikes**				
	1,1-Dichloroethene	22	59-172	14	61-145
	Trichloroethane	24	62-137	14	71-120
	Chlorobenzene	21	60-133	13	75-130
	Toluene	21	59-139	13	76-125
	Benzene	21	66-142	11	76-127
VOA	**Surrogate Spikes**				
	Toluene-d_8	—	50-160	—	86-119
	4-Bromofluorobenzene	—	50-160	—	85-121
	1,2-Dichloroethane-d_4	—	50-160	—	77-120
BNA	**Matrix Spikes**				
BN	1,2,4-Trichlorobenzene	23	38-107	28	39-98
	Acenaphthene	19	31-137	31	46-118
	2,4-Dinitrotoluene	47	28-89	38	24-96
	Di-n-butyl Phthalate	47	29-135	40	11-117
	Pyrene	36	35-142	31	26-127
	N-Nitroso-Di-n-Propylamine	36	41-126	38	41-116
	1,4-Dichlorobenzene	27	26-104	26	36-97
A	Pentachlorophenol	47	17-109	50	9-103
	Phenol	35	26-90	42	12-89
	2-Chlorophenol	50	25-102	40	27-123
	4-Chloro-3-Methylphenol	33	26-103	42	23-97
	4-Nitrophenol	50	11-114	50	10-80
BNA	**Surrogate Spikes**				
	Nitrobenzene-d_5	—	20-140	—	41-120
	2-Fluorobiphenyl	—	20-140	—	44-119
	p-Terphenyl-d_{14}	—	20-140	—	33-128
	Phenol-d_5	—	20-140	—	15-103
	2-Fluorophenol	—	20-140	—	23-121

Table 5-3
Data Quality Objectives
Matrix Spikes/Matrix Spike Duplicates/Surrogate Spikes

Fraction	Compound	Soil Precision (%RPD)	Soil Accuracy (%Recovery)	Water Precision (%RPD)	Water Accuracy (%Recovery)
Pesticides	**Matrix** Spikes				
	Lindane	50	46-127	15	56-123
	Heptachlor	31	35-130	20	40-131
	Aldrin	43	34-132	22	40-120
	Dieldrin	38	31-134	18	52-126
	Endrin	45	42-139	21	56-121
	4,4'-DDT	50	23-134	27	38-127
Pesticides					
	Dibutylchlorendate	—	48-136'	—	20-150'
Herbicides (8150)	Matrix Spikes				
	Silvex	40	40-150	30	40-130
Herbicides (8150)	**Surrogate Spikes**				
	Dicamba	—	50-140	—	50-130

Notes: QA/QC Requirements for CLP analyses excerpted from CLP SOW 3/90 (OLM01.0-OLM01.7); Other QA/QC requirements were derived from the analytical method references (USEPA, SW-846).

VOA = Volatile Organics
BN = Base/Neutral Extractable
A = Acid Extractable
= Advisory Limits
— = Not Applicable

6.0 QUALITY ASSURANCE PLAN

6.1 introduction

This section of the report presents policies, project organization, objectives and functional activities, along with QA/QC measures intended to achieve the quality assurance goals of the facility investigation to be performed by E/A&H at NAS Pensacola, Florida.

This document is intended to fulfill requirements for ensuring that all work be conducted in accordance with quality assurance/quality control protocols and field procedural protocols for environmental monitoring and measurement data as established in the following documents.

- Environmental Protection Agency, Athens, Georgia, February 1991. *Standard Operation Procedures and Quality Assurance Manual.*

- Naval Energy and Environmental Support Activity, Port Hueneme, *California,* June 1988 (NEESA 20.2-047B). *Sampling and Chemical Analysis Quality Assurance Requirements for* the *Navy Installation Restoration Program.*

- Naval Energy and Environmental Support Activity, Port Hueneme, *California,* February 1985 (NEESA 20.2-031A) *Groundwater Monitoring Guide.*

- Southern Division Engineering Command, Revision 4, March 1989. SOUTHDIV *Guidelines for Groundwater Monitoring Well Installation.*

Before field investigations begin, a site meeting will be conducted at the facility. Those people to attend the meeting are to include the task order manager, all field personnel, a representative of the site management and any subcontractors who will be working on the site. The SOUTHDIV EIC and representatives from the State of Florida and EPA Region IV will also be invited to attend. The purpose of this meeting will be to familiarize personnel and

subcontractors with the site, discuss the logistics with management, and discuss health and safety procedures. When authorization is given from the SOUTHDIV EIC, implementation of the investigation will begin by E/A&H.

6.2 Quality Control/Quality Assurance Objectives

In general, quality assurance objectives of E/A&H projects conducted as part of the Navy technical services contract are to assess and document the precision, accuracy, representativeness, completeness, and comparability of all sampling and analysis performed. Quality criteria are outlined here to assure suitability for intended use of data obtained during projects, and to meet goals established by NEESA and in the USEPA document, *Data Quality Objectives for Remedial Response Activities Development Process*, March 1987, Office of Solid Waste and Emergency Response (OSWER) Directive 9355.0-7B. NEESA DQO Level B protocol (EPA Level II) will be utilized for the physical surveys. Soil, surface water, sediment, groundwater and contaminant quantification investigations will be conducted at NEESA DQO Level D protocol (EPA LEVEL IV). The following discussion presents the project specific level of effort for quality assurance and data quality criteria. Quality control sample collection will follow the frequency presented in Table 6-1.

Precision

Precision is a measure or estimate of the reproducibility of measurements and methods and is defined for qualitative data as the variability of a group of values compared with their average value. For purposes of assessing precision of the measurement systems (sample collection and analysis) to be used in this project, duplicates will be obtained and analyzed with the samples collected. Precision, which will be used as a criterion for data classification, is calculated as a relative percent difference in analytical outcome between a given sample and corresponding duplicate.

Table 6-1
Quality Control Sample Collection Frequencies

Quality Control Sample	Frequency of Collection
Trip Blank (volatiles only)	One per sample shipping cooler
Rinsate Blank	One per day per media (to be analyzed every other day unless contaminants are identified); One per 20 soil gas samples collected
Field Blank	One per groundwater sampling event per day
Duplicates	One per 10 water and soil samples collected; One per 20 soil gas samples collected
Matrix Spike/Matrix Spike Duplicate Samples	One per 20 water and soil samples collected; matrix is to be the same sample used for duplicate analysis
Equipment/Material Blanks	One sample each of filter pack sand, drilling mud, grout, bentonite pellets or powder and potable water, used in well construction
Reagent Grade Water (ASTM Type III)	One per week

The type of duplicates used will depend on the part of the measurement system to be evaluated for precision. Field-duplicated samples analyzed by the same laboratory will yield information about sampling method precision and matrix homogeneity. Laboratory duplicated samples give an indication of sample preparation and analytical method precision.

A field duplicate is a sample collected in the field from the exact location as another sample and sent to the laboratory for analysis with another sample. A laboratory duplicate is prepared in the laboratory and consists of a split from a sample sent from the field to the laboratory.

Accuracy

The accuracy of an entire measurement system, which is an indication of any bias that exists, is difficult to measure in environmental measurement systems. Sources of error are the sampling

process, field and laboratory contamination, preservation, handling, sample matrix, and analysis. The accuracy of a method is an estimate of the difference between the true value and the determined mean value. In the field, methods used for detecting false positive results include the preparation of trip blanks, field blanks, and equipment rinsate blanks. In the laboratory, matrix and surrogate spike samples are used to detect positive and negative bias.

A field blank consists of sample containers filled in the field with organic-free, deionized or distilled water prepared, preserved and stored in the same manner as the samples. The field blank is analyzed along with the field samples for the constituents of interest to check for contamination imparted to the samples by the sample containers or other exogenous sources.

Trip blanks will be used for volatile constituent measurements. A trip blank consists of a volatile organic analysis sample container filled with organic-free water in the laboratory that travels unopened with the sample bottles to the field. The blank is then shipped back to the laboratory with water samples collected in the field. It is opened in the laboratory and analyzed along with the field sample for the volatile constituents of interest. Trip blanks will be submitted to the laboratory with water samples that are submitted for volatile analysis at a frequency of one per shipping container.

An equipment rinsate blank is made by taking organic-free deionized or distilled water and placing it in contact with the field sampling apparatus (e.g., bailer, pump, split barrel sampler) following the completion of equipment decontamination. The water will be collected in the same type of containers as the other samples, preserved in the same manner, and analyzed for the same parameters of interest. One equipment Msate blank will be collected each day, but will only be analyzed every other day.

Matrix spike samples prepared by the laboratory are useful in assessing the accuracy of the analytical methods, and can detect matrix effects in which other sample components interfere

with the analysis of the contaminant of concern. The method of measuring analytical accuracy is percent recovery.

Analytical matrix spikes will be performed by the laboratory, typically at a frequency of one per 20 samples per matrix. Matrix spike recoveries for inorganic analyses are definitive; therefore, if matrix spike recoveries are outside of control limits, the data will be flagged as quantitatively suspect or estimated. Control limits for laboratory matrix spike recovery are published by EPA or determined by historical laboratory results. Surrogate spikes, typically prepared from deuterated isomers of selected target compounds, provide an added measure of method accuracy. General precision and accuracy goals for laboratory analytical procedures (NEESA Level D) are as previously provided in Table 5-2. To provide the additional volume needed by the lab for matrix spike and matrix spike duplicate analysis, the first sample of each 20 samples taken will be doubled in volume. This will be done for both soil, water and sediment.

An equipment/materials blank shall be collected from the materials and liquids to be used in monitoring well construction. The samples shall be collected before drilling activities. The equipment/materials blanks shall be archived until needed to verify quality of well construction materials.

A reagent grade water blank will be collected from the ASTM type III water source. The samples shall be prepared, preserved, and stored in the same manner as the other samples. The reagent grade water blank shall be analyzed in the laboratory with the field samples for the constituents of interest to check for contamination imparted to the field samples from the water source.

Representativeness

The goal of this inspection is to delineate the extent of any soil and groundwater contamination, and to determine the most appropriate remedial option. By properly collecting soil and

groundwater samples and measuring monitoring well parameters in accordance with NEESA protocol, samples collected during the investigation should be representative of the areas of concern.

Completeness

Completeness goals for this project are set at 100 percent for laboratory analysis. Data completeness will be expressed both as the percentage of total tests conducted that are deemed valid and as the percentage of the total tests required in the scope of work that are deemed valid.

Completeness goals of field measurements reflect the ability to re-sample immediately, before declaring well stability, and obtaining samples for laboratory analysis. The completeness goals take into consideration unavoidable non-attainment of QA goals which may occur during the course of an investigation. Efforts will be made, however, to maintain soil and groundwater data completeness levels above the 90 percent level for field measurements ifpossible.

Comparability

comparability is assured through the use of established methods of field sampling by experienced field personnel and laboratory analysis as specified by NEESA regulations, as well as other accepted methods.

6.3 Experience Requirements

Experienced field personnel shall perform all site activities at NAS Pensacola. Rèsumès for E/A&H personnel are included in Appendix Q. Subcontractor rèsumès will be available onsite.

6.4 Organization and Responsibilities

Overall responsibility for projects conducted in accordance with NEESA regulations will be vested in NEESA (or its approved representatives). Therefore, project coordination responsibilities lie with the SOUTHDIV EIC. The components of the project chain-of-command

will be performed as established in NEESA 20.2-047B. Project oversight will be organized along the following lines of authority.

6.4.1 Navy Energy and Environmental Support Activity

NEESA is responsible for ensuring that the quality of laboratory analyses performed during the various phases of CLEAN is acceptable. NEESA is also responsible for managing the NEESA contract representative (NCR).

6.4.2 Engineer in Charge

The EIC at the engineering field division provides the site information and history, provides logistical assistance, specifies the sites requiring investigation and reviews results and recommendations. Ms. Linda Martin, SOUTHDIV, Naval Facilities Engineering Command, Charleston, South Carolina, serves as the EIC for this project.

The EIC is responsible for coordinating procurement, finance, and report; for ensuring that all documents are reviewed by the NCR; for communication comments from the NCR and other technical review to the subcontractors; and for ensuring that the subcontractors address all the comments submitted and take appropriate corrective actions.

6.4.3 NEESA Contract Representative

The NCR is responsible for ensuring that each project has appropriate overall QA. The NCR reviews laboratory QA plans and work plans, submits performance sample data, provides field and laboratory audits, and reviews data from the site. The questions from subcontractors and the EIC regarding specific field and laboratory QC practices are directed to the NCR. The NCR also evaluates referee samples.

6.4.4 State or Local Oversight

This Sampling and Analysis Plan will be submitted to the FDER and the EPA Region IV for review and approval. Field activities and meeting will be coordinated with these agencies as required.

6.4.5 Investigation Performance

The following individuals or firms will be responsible for the implementation of all work activities.

Engineering Subcontractor

E/A&H will serve as the engineering subcontractor for this project. As the engineering subcontractor, E/A&H is responsible for designing and implementing the RI/FS activities.

Analytical Laboratory

The analytical laboratory will be employed by E/A&H and must adhere to the laboratory requirements in NEESA 20.2-047B. The laboratory is required to prepare and submit a laboratory QA plan, to analyze and submit the results of proficiency testing, to submit to an onsite inspection, and to correct any deficiencies cited during the inspection by the NCR. The laboratories are required to identify a laboratory QA coordinator (LQAC) responsible for overall quality assurance. The LQAC must not be responsible for schedule, costs, or personnel other than QA assistants. It is preferred that the LQAC report to the laboratory director. The LQAC must have the authority to stop work on projects if QC problems arise which affect the quality of the data produced.

In addition to conforming to all NEESA regulations, all work shall be performed in a manner consistent with:

- The Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) as amended.

- The National Oil and Hazardous Substances Pollution Contingency Plan (NCP), Title 40 Code of Federal Regulations CFR), Part 300, as amended.
- Other appropriate federal, state, and local guidelines, rules, regulations, and criteria (where applicable).

7.0 DATA MANAGEMENT PLAN

The objective of this section of the Sampling and Analysis Plan is to describe the methods E/A&H will use throughout the project to manage collected data.

7.1 General Field Documentation Procedures

The field project manager will be thoroughly familiar with the appropriate documentation procedures. This person will perform or directly oversee the completion of the documents which accompany this investigation. Documentation tasks will be performed on a sample-by-sample or item-by-item basis throughout the day. However, items such as shipping containers, sample container labels, and chain-of-custody forms will be prepared as completely as possible in advance.

All documentation must be completed in waterproof black ink. Corrections will be marked with a single line, date, and initialed. Serialized documents are not to be destroyed or discarded even if illegible or inaccurate. Voided entries must be maintained within project files.

Field documentation shall consist of one master site logbook, one or more site-specific logbooks, field forms, photographs, sample labels and chain-of-custody records. This format allows for detailed recording of information in various field logbooks and forms that are referenced in the site logbook.

Logbooks

Site and field logbooks provide a daily handwritten record of all field activities at an investigation site. All logbooks must be permanently bound and have a hard cover. Field logbooks must be waterproof. The site logbook is a master record of all site activities, and entries are usually made at the end of each work day. Field logbooks are detailed daily records that are kept in real time. A field logbook will be assigned to each of the operable units at NAS Pensacola.

Site Logbooks — The site logbook chronicles all field investigation activities, but does not have the same level of detail as the field logbook. It delineates conditions and activities occurring on a given day and references the appropriate field logbooks and forms for specific information. The site logbook will also record all field changes with supporting rationale.

The field project manager will be responsible for completing the site logbook. This person will sign and date the logbook at the end of each page. Pages will not be removed from the document. All partially used pages will be lined out to prevent data entry at a later date. The front of the site logbook will list the project name and number, name of the subcontractor, service client, contract number, and dates of use.

Site Logbook Contents:

- A list of all field logbooks and a brief outline of their required contents.
- Daily temperature, weather conditions, and names and titles of personnel .present.
- Levels of personnel protection, and changes if required.
- Name, title, organization, and purpose of any site visitors.
- A brief outline of site activities and references to the appropriate logbook.
- Specific comments on any problems encountered, their resolution, and any impact on the field investigation.
- Any changes and the supporting rationale for the change.
- A brief record of all telephone calls and how they affected the investigation.
- A record of samples collected and shipped, the name of the laboratory, the air-bill numbers of the containers, details of the chain-of-custody, and any other pertinent information on sample status.
- A record of instrument calibration, including the name of the personnel who performed the calibration.
- A record of decontamination procedures.

Field Logbooks — All information required on the cover of the site logbook must also be provided on the cover of each field logbook. In addition, the field logbook must provide the specific area to which it was assigned. Entries in the field logbook must be made using a 24-hour clock and will be signed by the responsible person at the bottom of each page. Unused pages or portions of pages not used must be lined out to prevent entry of additional infomation. Copies of the field logbooks will be made on a regular basis to act as a backup for the logbooks.

Field Logbook Contents:

- Date and time task started and completed; weather conditions, and the names, titles, and organizations of personnel performing the tasks.
- A description of the level of personal protective equipment and any changes.
- A description of the site activities in specific detail or an indication of which forms were used for that activity.
- Description of field tests perfomed and results of the tests.
- Detailed description of samples collected, and any quality control samples collected. The time and name of the person to whom the sample custody was transferred or a reference to the appropriate documentation should be noted.
- A list of the time, equipment type and procedures followed for decontamination.
- A record of instrument calibration and any failures that occur with a brief description of repairs and/or replacements.

Field Data Record Forms — Forms are an extremely useful tool in the field and they help ensure that all necessary information is recorded. Blank spaces should not appear on completed forms. If information does not apply, the space should be marked "N/A." All forms will be completed in the field and placed in the project files. The forms will be copied on a regular basis to provide backup for the originals. Forms that may be used during this investigation include subsurface boring logs, monitoring well construction diagrams, monitoring well

development forms, sample records and additional data as appropriate. Field forms to be used during this investigation are provided in Appendix D.

Sample Labels — Sample labels will be completed then attached to each sample container after collection. Sample labels will be completed as outlined in Section 4.7.3 of the Sampling and Analysis Plan. Sample label information will be recorded in the field logbook as a cross-reference at the time of collection.

Chain-of-Custody Records — The chain-of-custody record will contain a summary of the contents of the shipment, dates, times, sample numbers, number and volume of containers, and documentation of the transfer of custody of the samples. Chain-of-custody forms will be completed in accordance with Section 4.8.2 of this *SAP*.

Subsurface Boring Logs — Soil boring logs will be maintained by a qualified E/A&H geologist. The lithology will be described from the split barrel samples using the Unified Soil Classification System (USCS). A summary of the USCS is provided in Appendix R.

The subsurface boring logs will be completed as the boring is advanced. Items to be recorded include materials encountered, depth to water, obvious contamination, and any other necessary information. A general log will be recorded in the field logbook as a cross-reference. An example of a boring log is provided in Appendix D.

Monitoring Well Construction Diagrams — A monitoring well construction diagram will summarize the monitoring well construction. Information to be completed includes location, date drilled, drilling method, well depth, screen location, and general construction data. A general log will also be recorded in the field logbook as a cross-reference. Monitoring well schematics are found in Section 4 of this *SAP*.

Photographs

Photographs will be taken of all pertinent field activities as directed by the Field Project Manager.

The following will be recorded in the field logbooks:

- Date, time, location of photograph
- Photographer
- Weather conditions
- Description of photograph and direction facing
- Sequential number of the photograph and the film roll number

When the photographs are developed, the information above will be transferred to the back of the photographs and signed by the photographer.

7.2 Analytical Data Validation

The CLP data shall be validated using procedures in 'National Functional Guidelines for Organic Data Review,' June 1991 and 'National Functional Guidelines for Inorganic Data Review," July 1988. Data from other analytical methods shall be validated by E/A & H. Independent data validation shall be conducted on 10% of the samples by Advance Science, Inc. of Oak *Ridge*, Tennessee.

7.3 Other Related Data

Other related data will include illustrations, graphs, meeting summaries, audit reports, and laboratory results. This information will be compiled and reviewed for report presentation.

Meeting Summaries, Telephone Conversations, and Notes

These listed items will be recorded in the field logbooks along with the dates, time, and names of persons involved. This information will be available for photocopying if requested.

Meetings and conversations with a substantial impact on the project will be described in a memorandum to the project manager.

Illustrations, Computations, and Engineering Data

Original illustrations and graphics will be initialed and dated by the person originating the document. A second person will check these documents for completeness and accuracy. All maps, calculations and data will be reported or prepared to normally accepted standards and confidence levels.

7.4 Reports

Progress Report

These reports will be prepared monthly by the project manager and will include: the number of samples collected, sites investigated, monitoring wells installed, any deviations from the approved field or laboratory procedures, and other appropriate information. These report will be directed to the EIC.

Final Report

The final remedial investigation report will be written following sampling and validation of all laboratory analysis. The report will consolidate and summarize the collected data and document the unit evaluation. An initial draft report will be submitted for comment by the Navy, USEPA, and FDER. Where appropriate, the comments will be incorporated into the final document.

Interim reports will be completed as necessary to describe significant divergence of the site conditions from those anticipated, to secure concurrence on the need for emergency or interim corrective measures, or to gain regulatory input on unanticipated issues.

Data obtained from sampling and analysis procedures will be summarized and presented in a tabular form. These tables will be supported by the raw laboratory reports submitted under separate cover to the EIC.

Graphical presentation of the sampling results will be in several formats. Isoconcentration maps will be developed for the soil and groundwater parameters. In addition, maps showing the sample locations labeled with the sampling results will also be completed. Groundwater surface contours along with flow direction and gradient will also be displayed on the site base maps. Cross-sectional plots may be used if it is determined that they would enhance the understanding of the site. Forms completed during the investigation will be included in the appendices of the report.

The interpretation of all of the accumulated data and analytical results will be performed as a project team effort. The expertise of each project team member will be used to develop proper conclusions and recommendations. The final decision on interpretation of the data for the report will lie with the task order manager.

8.0 REFERENCES

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9.0 FLORIDA PROFESSIONAL GEOLOGIST SEAL

I hereby affix my seal to the Sampling and Analysis Plan for Operable Unit 10, in accordance with Chapter 492 of the Florida Statutes and applicable rules and regulations developed pursuant thereto indicating that the related methods and procedures included in this plan are in accordance with standard, currently accepted geological practices.

Name:	Brian E. Caldwell
License Number:	1330
State:	Florida
Expiration Date:	July 31, 1994

Brian E. Caldwell

Brian E. Caldwell

10/13/92

Date

APPENDIX A

SUMMARY OF DRINKING WATER STANDARD EXCEEDANCES

Table 1
Summary of Detected Concentrations of Sodium in Groundwater
NAS Pensacola Operable Unit 10
(All results are in mg/l)

Monitoring Well	Sampling Event (Month/Year)									
	8/84	10/84	9/85	1/87	12/87	2/88	6/88	8/88	11/88	3/89
GM-14	NR	NR	NR	NS	NR	NR	NR	NR	NR	200
GM-63	NS	NS	NA	NS	470	420	789	448	NR	515
GM-64	NS	NS	NA	NS	330	200	86	142	NR	55
GM-65	NS	NS	NA	NS	120	110	110	76	NR	100
GM-66	NS	NS	NA	NS	640	670	527	465	NR	662
GM-68	NS	NS	NA	88	61	67	72	79	NR	57
GM-69	NS	NS	NA	NS	510	500	561	598	NR	669
GM-71	NS	NS	NA	18	9.8	5.7	4	7	NR	3,201
GM-72	NS	NS	NA	NS	79	42	44	73	NR	2,793
GM-76	NS	NS	NA	NS	210	190	207	501	NR	22
GM-77	NS	NS	NA	NS	53	43	40	303	NR	117
GM-84	NS	NS	NR	NS	NA	NA	NA	NA	NR	4,450
PCS-1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
PCI-1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
PCD-1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

Table 1
Summary of Detected Concentrations of Sodium in Groundwater
NAS Pensacola Operable Unit 10
(All results are in mg/l)

Monitoring Well	Sampling Event (Month/Year)									
	5/89	8/89	11/89	3/90	5/90	8/90	11/90	3/91	7/91	10/91
GM-14	**184**	**180**	**164**	140	120	NS	130	56	25	31
GM-63	**604**	**449**	**479**	**560**	**510**	**550**	**560**	**480**	**500**	**480**
GM-64	3.8	105	**241**	**320**	**210**	**310**	**290**	**320**	**320**	**380**
OM-66	1.78	1,070	88.9	120	180	180	92	93	96	92
OM-66	6.83	160	842	820	670	680	730	670	680	**640**
OM-68	**69**	600	893	77	81	90	84	81	89	84
OM-69	879	618	693	690	600	620	670	680	610	670
OM-71	268	NS	110	6.7	12	NS	150	6.2	3.2	4.6
OM-72	288	NS	27.4	17	13	NS	60	83	19	7.2
OM-76	39.1	NS	136	100	64	NS	110	110	110	110
OM-77	1.48	NS	46.5	44	28	NS	1,100	200	150	72
OM-84	10,200	4,470	6,640	4,300	4.400	NS	4,700	14.000	NS	NS
PCS-1	NS	NS	NS	NS	19	7.4	5.2	220	6.7	15
PCI-1	NS	NS	NS	NS	120	310	370	360	270	260
PCD-1	NS	NS	NS	NS	130	160	190	180	180	180
FPDWS	160	160	160	160	160	160	160	160	160	160

KEY:

NS – Monitoring well did not exist, or existed and was not sampled.
NA – Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reported.
Standard exceedances are in BOLD.

Source: Ecology and Environment, Inc., 1991.

Table 2
Summary of Detected Concentrations of Cadmium
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Sampling Event (Month/Year)	Monitoring Well	
	GM-66	**FPDWS**
7/84	NS	10
10184	NS	10
9/85	NA	10
1/87	NS	10
2/87	NA	10
5/87	NA	10
8/87	NA	10
12/87	NA	10
2/88	NA	10
6/88	NA	10
8/88	NA	10
11/88	NR	10
3/89	ND	10
5/89	NA	10
8/89	NA	10
11/89	NA	10
3/90	**43**	10
5/90	NA	10
8/90	**31**	10
11/90	NA	10
3/91	57	10
7/91	NA	10
10/91	NA	10

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reported.
ND = Parameter not detected at or above the method detection limit.
Standard exceedances are bold.

Source: Ecology and Environment, Inc., 1991.

Table 3
Summary of Detected Concentrations of Chromium
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Sampling Event (Month/Year)	Monitoring Well	
	GM-66	FPDWS
7/84	NS	50
10/84	NS	50
9/85	NA	50
1/87	NS	50
2/87	NA	50
5/87	NA	50
8/87	NA	50
12/87	NA	50
2/88	NA	50
6/88	NA	50
8/88	NA	50
11/88	NR	50
3/89	NA	50
5/89	NA	50
8/89	NA	50
11/89	NA	50
3/90	**210**	50
5/90	NA	50
8/90	**240**	50
11/90	NA	50
3/91	**360**	50
7/91	NA	50
10/91	NA	50

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reported.
Standard exceedances are bold.

Source: Ecology and Environment, Inc., 1991.

Table 4
Summary of Detected Concentrations of Lead
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Sampling Event (Month/Year)	Monitoring Well	
	GM-66	FPDWS
7/84	NS	50
10/84	NS	*50*
9/85	NA	50
1/87	NS	*50*
2/87	NA	50
5/87	NA	50
8/87	NA	50
12/87	NA	50
2/88	NA	50
6/88	NA	50
8/88	NA	50
11/88	NR	50
3/89	NA	50
5/89	NA	50
8/89	NA	50
11/89	NA	50
3/90	2.6	50
5/90	NA	50
8/90	NA	50
11/90	NA	50
3/91	**400**	50
7/91	NA	50
10/91	NA	50

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reported.
Standard exceedances are bold..

Source: Ecology and Environment, Inc., 1991.

Table 5
Summary of **Detected Concentrations** of Arsenic
NAS Pensacola Operable Unit 10
(All results are in $\mu g/l$)

Sampling Event (Month/Year)	Monitoring Well	
	GM-66	FPDWS
7/84	NA	50
10/84	NA	50
9/85	NS	50
1/87	ND	50
12/87	NS	50
2/88	NA	50
6/88	NA	50
8/88	NA	50
11/88	NA	50
3/89	ND	50
5/89	ND	50
8/89	ND	50
11/89	ND	50
3/90	ND	50
5/90	**51**	50
8/90	ND	50
11/90	2.6	50
3/91	ND	50
7/91	ND	50
10/91	NA	50

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
ND = Parameter not detected at or above the method detection limit.
Standard exceedances are bold.

Source: Ecology and Environment, Inc., 1991.

Table 6
Summary of Detected Concentrations of Mercury
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Sampling Event (Month/Year)	Monitoring Well	
	GM-66	FPDWS
7/84	NS	2
10/84	NS	2
9/85	NS	2
1/87	NS	2
12/87	NS	2
2/88	NS	2
6/88	NS	2
8/88	NS	2
11/88	NS	2
3/89	ND	2
5/89	ND	2
8/89	0.22	2
11/89	**4.4**	2
3/90	ND	2
5/90	ND	2
8/90	NS	2
11/90	ND	2
3/91	ND	2
7/91	ND	2
10/91	NA	2

KEY:

NA = Monitoring well sampled but sample not analyzed for this parameter.
NS = Monitoring well did not exist, or existed and was not sampled.
ND = Parameter not detected at or above the method detection limit.
Standard exceedances are bold.

Source: Ecology and Environment, Inc., 1991.

Table 7
Summary of Detected Concentrations of Vinyl Chloride
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Monitoring Well	Sampling Event (Month/Year)													
	2/84	7/84	10/84	9/85	4/86	1/87	2/87	5/87	8/87	12/87	2/88	4/88	6/88	8/88
DG-1	NR	10	32	NS	NR	NS	NR	NR	NR	NS	ND	NS	NS	NS
DG-2	NR	NS	NS	1	NR	NS	NR	NR	NR	2(J)	ND	NS	NS	NS
DG-4	NR	20	16	NS	ND	ND	NR	NR	ND	2(J)	250(E)	ND	NS	ND
DG-5	NR	ND	9	NS	NR	NS	NR	NR	NR	NS	7(J)	NR	ND	ND
DG-6	ND	ND	13	NS	NR	ND	NR	NR	NR	NS	ND	NS	NS	NS
OM-8	NR	NR	NR	NA	NR	NS	NR	NR	NR	NS	NS	NR	NR	NR
OM-9	NR	NS	21	NA	NR	NS	NR	NR	NR	ND	ND	NR	NR	NR
GM-10	NR	NS	7	NA	NR	NS	NR	NR	NR	NS	NS	NR	NR	NR
OM-64	NS	NS	NS	ND	NR	NS	NR	NR	NR	2(J)	ND	NR	NR	NR
GM-66	NS	NS	NS	27	NR	NS	146	ND	ND	270	170	NR	NR	NR
OM-69	NS	NS	NS	3	NR	NS	17	ND	ND	22	13	NR	NR	NR
GM-75	NS	NS	NS	1	NR	NS	NR	NR	NR	3(J)	2(J)	NS	NR	NR
FPDWS	1	1	1	1	1	1	1	1	1	1	1	1	1	1

Table 7 (continued)
Summary of Detected Concentrations of Vinyl Chloride
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Monitoring Well	Sampling Event (Month/Year)											
	11/88	3/89	5/89	8/89	11/89	3/90	5/90	8/90	11/90	3/91	7/91	10/91
DG-1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DG-2	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DG-4	ND	NR	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DG-5	ND	NR	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DG-6	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
GM-8	NR	**7**	ND	ND	ND	**180**	(L)	**74**	ND	ND	ND	ND
GM-9	NR	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
GM-10	NR	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
GM-64	NR	**4**	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
OM-66	NR	**380**	ND	ND	**180(J)**	**240(D)**	ND	**180**	ND	ND	ND	ND
GM-69	NR	**14**	ND	**9(J)**	**11**	**12**	ND	(L)	(L)	(L)	ND	(L)
GM-75	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
FPDWS	1	1	1	1	1	1	1	1	1	1	1	1

KEY:

NS	=	Monitoring well did not exist, or existed and was not sampled.
NA	=	Monitoring well sampled but sample not analyzed for this parameter.
NR	=	No data reported.
ND	=	Parameter not detected at or ebove the method detection limit.
(L)	=	Present below the detection limit.
(J)	=	Estimated value; compound present below the detection limit.
(E)	=	Concentration exceeded calibrated range of instrument.
(D)	=	Compound analyzed et e secondary dilution factor.

Standard exceedances are **bold**.

Source: Ecology and Environment, Inc., **1991.**

Table 8
Summary of Detected Concentrations of 1,1- Dichloroethene
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Well	Sampling Event (Month/Year)												
	2/84	5/84	7/84	10/84	9/85	1/87	2/87	5/877	8/87	12/87	2/88	6/88	8/88
DG-1	NS	NS	ND	800	NS	NS	NR	NR	NR	NS	ND	NA	NA
DO-6	8	< 10	ND	26	NS	ND	NR	NR	NR	NS	ND	NS	NS
OM-8	NS	NS	ND	ND	NA	NS	NR	NR	NR	NR	NR	NR	NR
OM-9	NS	NS	126	(L)	NA	NS	11	NR	NR	ND	ND	NR	NR
GM-64	NS	NS	NS	NS	ND	NS	NR	NR	NR	ND	ND	9	NR
GM-66	NS	NS	NS	NS	160	NS	446	NR	NR	390	320(E)	600	NR
GM-67	NS	NS	NS	NS	ND	NS	ND	NR	NR	ND	ND	NR	NR
OM-09	NS	NS	NS	NS	ND	NS	NR	NR	NR	2(J)	ND	NR	NR
FPDWS	7	7	7	7	7	7	7	7	7	7	7	7	7

Monitoring Well	11/88	3/89	6/89	8/89	11/89	3190	6190	8/90	11/90	3/91	7/91	10/91
DG-1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DO-6	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
GM-8	NR	**125**	**36**	ND	ND	ND	ND	ND	ND	ND	ND	ND
GM-9	NR	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
OM-64	NR	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
GM-66	NR	**310**	1,100	**320(J)**	**320(J)**	**350(E)**	(L)	**260**	(L)	(L)	ND	(L)
GM-67	NR	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
GM-69	NR	ND	ND	ND	ND	ND	ND	98	ND	ND	ND	ND
FPDWS	7	7	7	7	7	7	7	7	7	7	7	7

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reported.
ND = Parameter not detected at or above the method detection limit.
(L) = Present below the detection limit.
(J) = Estimated value; compound present below the detection limit.
(E) = Concentration exceeded calibrated range of instrument.
Standard exceadances are **bold**.

Source: Ecology and Environment, Inc., 1991.

Table 9
Summary of Detected Concentrations of 1,2-Dichloroethane
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Sampling Event (Month/Year)	Monitoring Well		
	GM-9	GM-64	FPDWS
7/84	**9,600**	NS	3
10/84	<3	NS	3
9/85	NA	ND	3
1/87	NS	NS	3
2/87	NA	NR	3
5/87	ND	NR	3
8/87	ND	NR	3
12/87	ND	ND	3
2/88	ND	ND	3
6/88	ND	**4**	3
8/88	ND	ND	3
11/88	NR	NR	3
3/89	ND	ND	3
5/89	ND	ND	3
8/89	ND	ND	3
11/89	ND	ND	3
3/90	ND	ND	3
5/90	ND	ND	3
8/90	ND	ND	3
11/90	ND	ND	3
3/91	ND	ND	3
7/91	ND	ND	3
10/91	ND	ND	3

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reported.
ND = Parameter not detected at or above the method detection limit.
Standard exceedances are bold.

Source: Ecology and Environment, Inc., 1991.

Table 10
Summary of Detected Concentrations of Chloroform
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Sampling Event (Month/Year)	Monitoring Well	
	DG-1	FPDWS
7/84	NR	100
10184	320	100
9/85	NS	100
1/87	NS	100
12/87	NS	100
2/88	ND	100
6/88	NS	100
8/88	NS	100
11/88	NS	100
3/89	NS	100
5/89	NS	100
8/89	NS	100
11/89	NS	100
3/90	NS	100
5/90	NS	100
8/90	NS	100
11/90	NS	100
3/91	NS	100
7/91	NS	100
10/91	NS	100

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NR = No data reported.
ND = Parameter not detected at or above the method detection limit.
Standard exceedances are bold.

Source: Ecology and Environment, Inc., 1991.

Table 11
Summary of Detected Concentrations of Carbon Tetrachloride
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Sampling Event (Month/Year)	Monitoring Well		
	DG-1	DG-6	FPDWS
2/84	NA	NR	3
7/84	NR	NR	3
10/84	**425**	**9**	3
9/85	NS	NS	3
1/87	NS	ND	3
12/87	NS	NS	3
2/88	ND	ND	3
6/88	NS	NS	3
8/88	NS	NS	3
11/88	NS	NS	3
3/89	NS	NS	3
5/89	NS	NS	3
8/89	NS	NS	3
11/89	NS	NS	3
3/90	NS	NS	3
5/90	NS	NS	3
8/90	NS	NS	3
11/90	NS	NS	3
3/91	NS	NS	3
7/91	NS	NS	3
10/91	NS	NS	3

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reported.
ND = Parameter not detected at or above the method detection limit.
Standard exceedances are bold.

Source: Ecology and Environment, Inc., 1991.

Table 12
Summary of Detected Concentrations of Trichloroethene
NAS **Pensacola Operable** Unit 10
(All results are in µg/l)

Monitoring Well	Sampling Event (Month/Year)											
	2/84	5/84	7/84	10/84	9/85	1/87	2/87	5/87	8/87	12/87	2/88	6/88
UG-1	NR	NR	ND	NR	NS	ND	NR	NR	NR	NS	ND	ND
DG-1	NR	NR	ND	20	NS	NS	NR	NR	NR	NS	ND	NS
DG-2	NR	NR	NS	NS	6	NS	NR	NR	NR	ND	ND	NS
DG-4	NR	NR	ND	13	NS	ND	NR	NR	NR	ND	1(J)	NS
DG-6	58	21	20	32	NS	ND	NR	NR	NR	NR	ND	NS
GM-8	NR	NR	ND	ND	NA	NS	NR	NR	NR	NS	NS	NR
GM-9	NR	NR	60	(L)	NA	NS	7	1	NR	ND	ND	ND
GM-12R	NR	NR	NR	NR	NA	ND	NR	NR	NR	NS	NR	NR
GM-65	NS	NS	NS	NS	4	NS	NR	NR	NR	50	ND	ND
GM-66	NS	NS	NS	NS	2,600	NS	15,907	24,750	13,600	10,000	4,200(E)	6,300
GM-67	NS	NS	NS	NS	50	NS	78	8	NR	ND	ND	ND
GM-68	NS	NS	NS	NS	10	ND	NR	NR	NR	ND	ND	ND
OM-69	NS	NS	NS	NS	3	NS	8	6	NR	ND	ND	ND
GM-70	NS	NS	NS	NS	7	NS	NS	NS	NS	NS	NS	NS
FPDWS	3	3 1	3 1	3	3	3	3	3	3	3	3 1	3

Table 12 (continued)
Summary of Detected Concentrations of Trichloroethene
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Monitoring Well	Sampling Event (Month/Year)												
	8/88	11/88	3/89	5/89	8/89	11/89	3/90	5/90	8/90	11/90	3/91	7/91	10191
UG-1	ND	ND	ND	ND	ND	**6(B)**	ND	ND	ND	ND	ND	ND	ND
DO-1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DO-2	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DG-4	ND	ND	ND	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DG-6	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
OM-8	NS	NS	66	**1(J)**	ND	**1(J)**	ND	ND	ND	ND	ND	ND	ND
OM-9	ND	NS	ND	ND	ND	**4(J,B)**	ND	ND	ND	ND	ND	ND	ND
OM-12R	NR	NS	NR	ND	ND	**6(B)**	ND	ND	NA	ND	ND	ND	ND
OM-65	ND	NS	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
GM-66	6.000	NA	**8.900**	***6,600***	**11,000**	**8,400(E)**	ND	ND	**7,300(E)**	**140**	8,200	**12.000**	**4,600**
OM-67	ND	NS	ND	ND	ND	**2(J,B)**	ND	ND	ND	ND	ND	ND	ND
GM-68	ND	NS	ND	ND	ND	**4(J,B)**	ND	ND	ND	ND	ND	ND	ND
OM-69	ND	NS	ND	ND	ND	**1(J)**	ND	ND	ND	ND	ND	ND	ND
GM-70	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
FPDWS	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**	**3**

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reported.
ND = Parameter not detected.
(L) = Present below the detection limit.
(J) = Estimated value; compound present below the detection limit.
(B) = Compound also present in method blank.
(E) = Exceeds calibration limit.
Standard exceedances are **bold**.

Source: Ecology and Environment, Inc., 1991.

Table 13
Summary of Detected Concentrations of 1,1,1-Trichloroethane
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Sampling Event (Month/Year)	Monitoring Well				
	DG-1	DG-6	GM-8	GM-9	FPDWS
7/84	110	**330**	ND	**11,500**	200
10/84	**5,900**	103	ND	(L)	200
9/85	NS	NS	NA	NA	200
1/87	NS	ND	NS	NS	200
2/87	NR	NR	NR	**356**	200
5/87	NR	NR	NR	NR	200
8/87	NR	NR	NR	NR	200
12/87	NS	NS	NS	ND	200
2/88	ND	ND	NS	ND	200
6/88	NS	NS	NR	ND	200
8/88	NS	NS	NR	ND	200
11/88	NS	NS	NR	NR	200
3/89	NS	NS	**1,200**	ND	200
5/89	NS	NS	6	ND	200
8/89	NS	NS	ND	ND	200
11/89	NS	NS	ND	ND	200
3/90	NS	NS	ND	ND	200
5/90	NS	NS	ND	ND	200
8/90	NS	NS	ND	ND	200
11/90	NS	NS	ND	ND	200
3/91	NS	NS	ND	ND	200
7/91	NS	NS	ND	ND	200
10/91	NS	NS	ND	ND	200

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reported.
ND = Parameter not detected at or above the method detection limit.
(L) = Present below the detection limit.
Standard exceedances are bold.

Source: Ecology and Environment, Inc., 1991.

Table 14
Summary of Detected Concentrations of Tetrachloroethene
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Monitoring Well	Sampling Event (Month/Year)												
	2/84	5/84	7/84	10/84	9/85	1/87	2/87	5/87	8/87	12/87	2/88	6/88	8/88
DG-1	NS	NS	ND	500	NS	NS	NR	NR	NR	NS	ND	NS	NS
DG-2	NS	NS	NS	NS	17	NS	NR	NR	NR	ND	ND	NS	NS
DG-3	NS	NS	NS	NS	10	NS	NS	NS	NS	NS	NS	NS	NS
DG-4	NS	NS	ND	11	NS	ND	NR	NR	NR	ND	ND	ND	ND
DG-6	6	<10	ND	38	NS	ND	NR	NR	NR	NS	ND	NS	NS
GM-8	NS	NS	ND	NR	NA	NS	NR	NR	NR	NS	NS	NR	NR
GM-9	NS	NS	676	4	NA	NS	42	23	NR	ND	ND	ND	ND
GM-62	NS	NS	NS	NS	6	NS	NR	NR	NR	ND	ND	ND	ND
GM-63	NS	NS	NS	NS	6	NS	NR	NR	NR	ND	ND	ND	ND
OM-64		NS	NS	NS	9	NS	NR	NR	NR	ND	ND	ND	ND
GM-66		NS	NS	NS	6	NS	8	ND	ND	4(J)	4)J)	ND	ND
OM-69		NS	NS	NS	ND	NS	NR	NR	NR	ND	ND	ND	ND
OM-81		NS	NS	NS	78	NS	NS	NS	NS	NS	NS	NS	NS
OM-82		NS	NS	NS	19	NS	NS	NS	NS	NS	NS	NS	NS
OM-83	NS	NS	NS	NS	26	NS	NR	NR	NR	NA	NA	NA	NA

Table 14 (continued)
Summary of **Detected Concentrations** of **Tetrachloroethene**
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Monitoring Well	Sampling Event (Month/Year)											
	11/88	3/89	5/89	8/89	11/89	3/90	5/90	8/900	11/90	3/91	7/91	10/91
DG-1	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**
DG-2	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**	**NS**
DG-4	NR	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DO-6	NS	**NS**	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
OM-8	NR	**138**	**22**	9	**1(J)**	ND	ND	**ND**	ND	**(L)**	ND	ND
OM-9	NR	ND	ND	ND	ND	ND	**ND**	ND	**ND**	ND	ND	ND
GM-62	**NR**	**ND**	**ND**	**ND**	**ND**	**ND**	**ND**	**ND**	**ND**	**ND**	**ND**	**ND**
GM-63	NR	ND	ND	ND	ND	ND	**ND**	ND	ND	ND	ND	ND
GM-64	NR	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
OM-66	NR	ND	ND	ND	ND	**4(J)**	ND	ND	ND	ND	ND	ND
GM-69	NR	**4**	**6**	7	**7**	**(L)**	**(L)**	ND	ND	**ND**	ND	ND
OM-81	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
OM-82	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
OM-83	NR	NS	NA	NS	NA	NA	NA	NS	NA	NA	NA	NA
FPDWS	3	3	3	3	3	3	3	3	3	3	3	3

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reported.
ND = Parameter not detected at or above the method detection limit.
(L) = Present below the detection limit.
(J) = Estimated value; compound present below the detection limit.
Standard exceedances are **bold**.

Source: Ecology and Environment, Inc., **1991.**

Table 15
Summary of Detected Concentrations of Benzene
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Sampling Event (Month/Year)	Monitoring Well							
	DG-4	DG-5	DG-6	GM-8	GM-66	GM-67	GM-69	FPDWS
2/84	NS	NS	**3.5**	ND	NS	NS	NS	1
5/84	NS	NS	**<10**	ND	NS	NS	NS	1
7/84	NS	NS	ND	ND	NS	NS	NS	1
10/84	**2**	**2**	**2**	ND	NS'	NS	NS	1
9/85	NS	NS	NS	NA	ND	NR	**3**	1
1/87	ND	NS	ND	NS	NS	NS	NS	1
2/87	NR	NR	NR	ND	ND	NR	**9**	1
5/87	NR	NR	NR	ND	ND	NR	**3**	1
8/87	NR	NR	NR	ND	ND	**2**	NR	1
12/87	**3(J)**	NS	NS	ND	ND	ND	**6**	1
2/88	0.4(J)	ND	ND	ND	ND	ND	**5**	1
6/88	NS	NR	NS	ND	ND	ND	ND	1
8/88	NR	NR	NS	ND	ND	ND	ND	1
11/88	NS	NR	NS	ND	NR	NA	NA	1
3/89	NS	NR	NS	ND	ND	ND	**3**	1
5/89	NS	NS	NS	ND	ND	ND	ND	1
8/89	NS	NS	NS	ND	ND	ND	**3(J)**	1
11/89	NS	NS	NS	ND	ND	ND	ND	1
3/90	NS	NS	(L)	**2(J)**	ND	ND	ND	1
5/90	NS	NS	NS	ND	ND	ND	ND	1
8/90	NS	NS	NS	(L)	ND	ND	(L)	1
11/90	NS	NS	NS	ND	ND	ND	ND	1
3/91	NS	NS	NS	ND	ND	ND	ND	1
7/91	NS	NS	NS	**750**	ND	ND	ND	1
10/91	NS	NS	NS	**350**	ND	ND	ND	1

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
NR = No data reponed.
ND = Parameter not detected at or above the method detection limit.
(L) = Present below the detection limit.
(J) = Estimated value; compound present below the detection limit.
Standard exceedances are **bold**.

Source: Ecology and Environment, Inc., 1991.

Table 16
Summary of Detected Concentrations of 1,4-Dichlorobenzene
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Monitoring Well	Sampling Event (Month/Year)													
	2/84	5/84	7/84	10/84	9/85	1/87	2/87	5/87	8/87	12/87	2/88	4/88	6/88	8/88
GM-69	NS	NS	NS	NS	1,350	NS	165	NA	473	880	110	NS	588	800
GM-8	NS	NS	NA	NA	30	NS	NS	NS	NS	NS	NS	NS	NS	NS
DG-1	NS	NS	200	387	NS	NS	NS	NS	NS	NS	15	NS	NS	NS
DO-6	37.6	77	120	182	NS	40	NS	NS	NS	NS	ND	NS	NS	NS
DG-4	NS	NS	ND	98	NS	30	NS	NS	NS	63	45	88	NS	ND
DG-5	NS	NS	6(J)	170	NS	NS	NS	NS	NS	NS	24	NS	ND	ND
DG-2	NS	NS	NS	NS	170	NS	NS	NS	NS	66	28	NS	NS	NS
GM-9	NS	NS	NA	NA	4,000	NS	127	NA	ND	ND	ND	NS	ND	ND
OM-10	NS	NS	NA	NA	120	NS	NS	NS	NS	NS	NS	NS	NS	NS
OM-67	NS	NS	NS	NS	200	NS	128	NA	45	ND	ND	NS	ND	ND
FPDWS	75	75	75	75	76	75	75	75	75	75	75	75	75	75

Table 16
Summary of Detected Concentrations of 1,4-Dichlorobenzene
NAS Pensacola Operable Unit 10
(All results are in µg/l)

Monitoring Well	Sampling Event (Month/Year)											
	11/88	3189	6189	6/89	11/89	3/90	6190	8/90	11/90	3/91	7/91	10191
OM-69	NS	1,510	410	660	250	140	660	330(E)	200	390	390	300(E)
GM-8	NS	424	130	66	2s	2,800	2,600	860	190	3,800(E)	820	250
DG-1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DO-6	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DO-4	ND	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DG-5	ND	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
DO-2	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
GM-9	NS	7	4(J)	ND	ND	ND	ND	ND	(L)	(L)	(L)	(L)
GM-10	NS	13	11	10	5(J)	(L)	(L)	(L)	(L)	(L)	(L)	ND
GM-67	NS	11	8(J)	4(J)	8(J)	(L)	(L)	(L)	(L)	(L)	12	10
FPDWS	75	76	75	75	75	76	75	75	75	75	75	75

KEY:

NS = Monitoring well did not exist, or existed and was not sampled.
NA = Monitoring well sampled but sample not analyzed for this parameter.
ND = Parameter not detected at or above the method detection limit.
(L) = Present below the detection limit.
(J) = Estimated value; compound present below the detection limit.
(E) = Concentration exceeded calibrated range of instrument.
Standard exceedances are **bold**.

Source: Ecology and Environment, Inc., **1991.**

APPENDIX B

SOIL GAS SUBCONTRACTOR *QAP*

QUALITY ASSURANCE PLAN
for
SOIL GAS SERVICES

Pensacola Naval Air Station
Florida

QUALITY ASSURANCE PIAN
for
SOIL GAS SERVICES
at
Pensacola Naval Air Station
Florida

Prepared for
Ensafe - Allen & Hoshall
5720 Summer Trees Drive, #8
Memphis, TN 38134

Prepared by
TARGET ENVIRONMENTAL SERVICES, INC.
9180 Rumsey Road
Columbia, MD 21045
(410) *992-6622*

August 4, 1992

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1.0 Project Description

The specific objective of this survey is to assist E/A&H with the implementation of a Remedial Investigation for Group O Sites at the Pensacola NAS. The soil gas survey will be completed at the Industrial Wastewater Treatment Plant.

E/A&H will provide general base maps of the Group O Sites and determine approximately 275 sampling location, prior to mobilization of TARGET's field team. E/A&H's on-site representative and TARGET's field team will discuss the proposed plan and make necessary modifications in the field. A sampling depth of 2-5 feet is planned.

Samples will be analyzed on a research grade gas chromatograph equipped with capillary columns, thermal oven, and with a data processor and associated hardware. Each instrument is appropriately calibrated at the beginning of the project, and as needed for the duration of the project using an instrument-response curve and injection of standards of known concentrations. Calibration checks will be performed at a minimum of twice a day. Retention times of the compounds in the standards are used to identify the unknown compounds in field samples, and their response factors are used in calculating actual concentrations. Duplicate analyses will be performed on at least every tenth field sample (see QA/QC section).

This level of gas chromatography not only provides reliable identification and quantification of standard analytes, it also produces a chromatogram with enough resolution to match sample signatures with known contaminant signatures and determine (in many cases) the nature of the original product, and its relative age.

TARGET uses both FID and ECD detectors when the analytes of interest include both non-halogenated and halogenated compounds. When assessing petroleum hydrocarbons and other non-halogenated compounds, TARGET has found that a full signature analysis on a FID provides much more information than the results from a PID or any other detector. Since older spills are often degraded, analyzing for only a limited number of compounds may result in an occurrence being missed whereas a full analytical signature will allow successful identification and mapping of the spill.

Some compounds will coelute with other compounds. When this happens, the results will be reported as a coelutant pair. If further resolution is desired, a representative sample from any given area of interest will be selected for analysis by GC/MS. All GC/MS analyses will be performed by Maryland Spectral Services, Inc., Baltimore, Maryland.

The results of these analyses and associated data will be interpreted (including chromatogram signature analysis for characterizing likely original products) and reported by TARGET in the form of a written report including a summary of background information, descriptions of sampling and analytical procedures, tabulated analytical results (including QA/QC), a scaled base map with labelled sample locations, contoured maps (as appropriate) of individual component concentrations, and a discussion and interpretation of the findings.

20 Personnel

2.2 Management Personnel

The officers of **TARGET** Environmental Services, Inc. **(TARGET)** have actively participated in the environmental industry for the past **20-30** years and have combined their expertise at **TARGET** to produce quality service to both public and private clients.

J. EDWARD TILLMAN is the founder and President of The **TARGET** Companies, **Inc.** and its subsidiary, **TARGET** Environmental Services, Inc. **(TARGET). Mr.** Tillman is responsible for the overall direction of corporate activities, **as** well **as** the quality of corporate services. He plays an active role in management, research and development, and client interaction. Geochemistry and environmental project management are Mr. Tillman's areas of expertise. The 18 years of experience in these fields has afforded him the opportunity to develop and refine various forms of surface and subsurface sampling technologies which have been instrumental in the development of **TARGET's** full range of soil gas surveying methods. Prior to founding **TARGET** in 1981, Mr. Tillman's professional and management experience included sewing as senior consultant for several major geotechnical consulting forms on numerous projects in the Middle East, Canada and most parts of the United States. Mr. Tillman has published and presented numerous papers on the investigation of contaminants in the subsurface and has conducted seminars on environmental issues. He has chaired a **committee** on ground water problems for the State of Maryland and holds certifications from the American Association of Petroleum Geologists (#3159), the American Institute of Professional Geologists (#6245), and is a registered Professional Geologist in the state of Delaware.

KENNETH B. **RANLET** is Vice-President of Operations of **TARGET.** Mr. Ranlet has been active in geotechnical project management with the petroleum and environmental industries for 18 years. Most of his geological experience has concentrated in the Gulf Coast, Southwest, Midwest and Eastern regions of the United States. At **TARGET,** he is responsible for the management and direction of all soil gas, water and soil sampling projects, the overall development and refinement of technical procedures, and the planning and design of project and field activities. Mr. Ranlet is directly involved in the interpretation and reporting *of* project results and maintains close client interaction regarding matters pertaining to each project. Mr. Ranlet is a member of the Association of Engineering Geologists, is certified by the American Association of Professional Geologists (#2778), and is a registered geologist in the state of South Carolina.

21. Laboratory Personnel

ELIZABETH J. TIERNEY is Laboratory Director for TARGET. She directs all in-house laboratory operations. She also oversees the maintenance and calibration of the various gas chromatographs. Her duties include method development and implementation of new chromatographic technologies. Before coming to work for TARGET, Dr. Tierney was employed in the GC-MS Laboratory for the State of Maryland Department of Health and Mental Hygiene. She was responsible for the analysis of water, sediment, and hazardous waste samples utilizing EPA methodologies to insure compliance with state and federal regulations. She earned a PhD in chemistry from the University of Maryland.

RUSSELL C. URICH III serves as Manager in TARGET's environmental mobile laboratory. His responsibilities include overall coordination of mobile laboratory scheduling, maintenance and servicing of GC's, troubleshooting, R & D and supervision of mobile laboratory personnel. Mr. Urich was employed by Shimadzu Scientific Instruments for eight years as a Quality Assurance Engineer and supervised the assembly and QA of Gas Chromatographs. In this position, he implemented QA and manufacturing procedures for the Transformer Gas Analysis System which is in use worldwide for the analysis of dissolved gases in transformer oil. He also designed, fabricated and tested the "Weasel" gas manifold system for delivery of carrier gas. His degree is in Marine Engineering from Florida Institute of Technology.

SCOT E REYNOLDS is the GC Supervisor in the environmental laboratory. His responsibilities are sample scheduling, operation of GC equipment, standards preparation, writing SOP's, calibrating instruments, data processing and maintenance of GC's. Mr. Reynolds majored in Chemistry at Rochester Institute of Technology. Before joining TARGET, he worked for three years as a Chemical Technician for Eastman Kodak Co.

MICHAEL MARRALE is a In-Field Analysis Coordinator for TARGET's Columbia office, the California Regional office, and the St. Louis/Dallas Regional office. He is responsible for the daily operations of the mobile laboratories as well as maintaining standard operating procedures and quality assurance/quality control procedures for field analysis. Mr. Marrale has a B.A. in Environmental Science from the University of Virginia.

GREGORY A. BEAL is a Field Chemist/Environmental Field Technician for TARGET's Columbia Office. His experience includes air monitoring using photoionization detectors, organic vapor analyzers, colorimetric devices, explosimeters, and oxygen meters. He has extensive experience in the operation of gas chromatographs. His duties encompass calibration, maintenance, and trouble-shooting of GC equipment used for on-site analysis, in addition to sample collection.

3.0 Laboratory Facilities

The primary operation of the laboratory is to analyze soil gas/vapor/air samples. TARGET operates ten Shimadzu gas chromatographs, equipped with auto samplers, flame-ionization detector (FID), electron capture detector (ECD), and with computerized data systems which are dedicated to the 24-hour analysis of vapor samples. Additional capactty is available if needed through the use of identical analytical equipment in TARGET's mobile labs. Each GC can routinely analyze 50 soil gas samples/day under standard run parameters. The fixed-base laboratory operates seven days/week. Providing allowances for equipment maintenance and additional processing for highly contaminated samples, the laboratory can analyze approximately 1,200 samples/week. The laboratory is also equipped to analyze soil and water samples by purge and trap utilizing GC/PID (photo-ionization detector) and Hall detector.

4.0 Quality Assurance Objectives

4.1 Precision

Precision will be assessed by the comparison of replicate samples. A replicate analysis will be obtained for every tenth sample.

4.2 Accuracy

Accuracy will be determined by the analysis of field blanks, lab blanks and check standards. Accuracy will be estimated by comparisons of measured check standard concentrations of each analyte with known concentrations in the stock gas, and comparison of replicate analyses. The data quality objective with respect to field and lab blanks is to achieve analytical concentrations below the quantification limit for all analytes.

When contamination is determined to be present in a lab or field blank, it will be acknowledged in the report. An explanation will be given as to the cause of the contamination. An assessment as to the effect of the contamination on the validity of the data from any field sample locations will be made.

4.3 Representativeness

Representativeness of data collection will addressed by careful preparation of the sampling program which specifies sufficient and proper numbers, and frequency and location of samples, so as to assure that sample data accurately and precisely represent selected characteristics of the soil gas.

5.0 Sampling Procedures

Upon arrival at the site, TARGETs 2-man team will check the site map for accuracy, measure out the sampling grid, and mark each location with a pin flag or paint. An electric hammer-drill will be used to penetrate pavement where necessary. TARGET personnel will repair such holes upon completion of sampling.

51 Field Sampling Procedures

Soil gas samples are to be collected at predetermined locations with on-site additions or deletions as directed by the client or prevailing conditions. The samples will be taken from a depth greater than 2 feet to avoid surface interferences and less than 5 feet to avoid ground water. The actual sampling depth will depend on local conditions (variations in the depth of collection may be necessary due to shallow ground water or bedrock, for example).

Prior to sampling, the entire sampling system is purged with a volume of ambient air (filtered through a charcoal canister) or nitrogen no less than 20 times the volume of the sampling apparatus void space. To collect the soil gas samples a 1/2 inch hole is produced to the sampling depth by using a manual slide hammer or drive rod. After the slide hammer is removed from the hole, a specially designed stainless steel probe is inserted to the full depth of the hole and packed off to isolate the probe's perforations from the atmosphere. A sample of in-situ soil gas is withdrawn through the probe and used to displace the atmospheric air or nitrogen from the sampling system. This volume of soil gas is then vented to the atmosphere. A second sample of soil gas is withdrawn through the probe and encapsulated in a pre-evacuated glass vial at two atmospheres of pressure (15 psig).

This sampling system is designed to minimize variables and obtain the most representative sample available from each sample location. A large (approximately 1/2 liter), gas tight syringe is used instead of an electric pump, coupled with small diameter sampling probes and lines. This allows for the total volume of the extracted sample to be reduced, and provides samples which more accurately represent contaminant concentrations in the subsurface. A small, measured, uniform sample volume insures consistently representative samples throughout the entire survey.

52 Documentation

Field documentation at each sample location includes the sample identification, depth, general area observations, available surface and subsurface material observations, observed relative moisture levels, qualification of soil vapor availability, equipment used to collect the sample, general remarks, and a general description of the sample location.

53 Control

Field Control Samples are collected by drawing prepurified nitrogen or ambient air (filteredthrough an MSA organic cartridge filter) through the sampling apparatus and probe prior to each day's sampling activities, after every twentieth sample, and at the conclusion of each sampling day. These field control samples are labeled and analyzed in the same manner as the actual field samples and are visually indistinguishable from them.

60 Sample Custody

Samples are logged on a Chain of Custody form which accompanies the samples during custody transfers, or transport to the analytical laboratory.

At the end of each sampling day, samples are recounted and chain-of-custody forms are completed for all samples. All information on the chain-of-custody form and the sample labels is checked against the sample field log book entries before leaving the sampling site.

The field sampling personnel are responsible for the care and custody of the collected samples until properly dispatched to the receiving laboratory or turned over to an assigned custodian or overnight carrier. Upon transfer of custody, the chain-of-custody form is signed by one member of the sampling team, including the date and time.

After the samples are analyzed on-site, the samples will be hand delivered to target's fixed-base laboratory or may be shipped by overnight carrier. Overnight carriers (i.e. Federal Express, UPS, etc.) do not sign chain-of-custody forms; therefore, the chain-of-custody records will be sealed within each container of samples. The receipt or airbill for the shipment will be retained in the project file as part of the chain-of-custody documentation.

All chain-of-custody forms received by the laboratory are signed and dated by the laboratory sample custodian and returned to TARGET project management department for the project file.

70 Calibration

FID analysis is conducted at Range One, while ECD analysis is conducted at Range 0. Three-point least squares linear regression calibration curves are generated for each detector as needed and the correlation coefficients are examined for each standardized analyte. Correlation coefficients must be greater than 0.99. The curve is then used to, quantify the concentration of analytes in samples. Following the initial three-point calibration, check standards are analyzed at the beginning and end of each day to ensure retention time and response stability. Windows for retention times will be set using the narrowest time band possible (usually 0.05 - 0.1 minutes) without including non-standardized peaks.

The FID and ECD stock standards are replaced as needed from pre-prepared (Scott Specialty Gases) cylinders that contain mixtures of our standard analytes. A volume of gas is extracted from the main pressurized cylinder. The line is purged to eliminate contamination or dilution and then a one litre Tedlar bag with a teflon lined septa is filled. Each Tedlar bag is used only once.

Aliquots from the Tedlar bags are withdrawn using Hamilton gas tight glass syringes and are then injected into 30 ml pre-capped, pre-cleaned (EPA protocol B Level II) evacuated glass vials. Each standard has its own designated syringe which is cleaned with ultra-pure nitrogen before the standards are prepared.

80 Analytical Procedures

8.1 Modified EPA Methods 601 and 602

A gas chromatograph equipped with an electron capture detector (GC/ECD), following modified EPA 601 methodology, is used to identify and quantify chlorinated compounds typically found in industrial solvents, while a flame ionization detector (GC/FID), following a modified EPA 602 methodology, is used for petroleum and other non-haiogenated compounds.

8.2 Reported Quantification Limits

The "Reported Quantification Limit" is a concentration level at which the degree of confidence in the actual presence of a compound becomes meaningful. A reported quantification limit should not be confused with the concentration represented by the smallest detectable chromatogram peak area. The importance of reported quantification limits should also be weighed in the context of acceptable exposure levels and the general levels of contamination on a site. The reported quantification levels for compounds to be identified on the GC/FID (petroleum hydrocarbons) will be 1.0 µg/l. The reported quantification levels for compounds identified on the GC/ECD are 1.0 µg/l.

The following tables list the minimum concentrations (the reported quantification limits) of selected compounds reported using TARGET's soil gas analyses (additional analytes are available).

VOLATILE ORGANIC COMPOUNDS	REPORTED QUANTIFICATION LIMITS
benzene	1.0
ethylbenzene	1.0
toluene	1.0
total xylenes (ortho, meta, para- isomers)	1.0
1,1-dichloroethene (1,1-DCE)	1.0
c-1,2-DCE	1.0
t-1,2-DCE	1.0
dichloroethane (1,1-DCA)	1.0
methylene chloride	1.0
chloroform	1.0
trichloroethene (TCE)	1.0
1,1,1-trichloroethane (1,1,1-TCA)	1.0
1,1,2-TCA	1.0
carbon tetrachloride	1.0
tetrachloroethene (PCE)	1.0

9.0 Data Validation and Reporting

The data is reviewed to ensure all analytes are identified and their concentrations are within acceptable criteria. The data and chromatograms are then submitted to the Interpretaion and Reporting group.

10.0 Audits

The performance audit consists of laboratory blanks (every 10 samples), replicate analysis of every tenth sample, check standards (at the end of every job or after sample 40), and field blanks (at the beginning and end of each day's sampling and after every 20 samples).

Data from quality control samples (laboratory blanks, field blanks, check standards), standards and headspace samples, including sample replicates, are reviewed by a mobile laboratory operator, a QA assistant and project scientist in TARGET's Interpretation and Reporting Department.

11.0 Preventive Maintenance

Maintenance is conducted on a daily basis and all information is recorded in the system maintenance book. Daily checks include: purge and flow rates, gas pressure readings to the GC, the FID is checked to ensure it is lit, and the sufficient pressure warning light is checked on the autosampler. The columns are conditioned daily at 200°C. Injection septa are replaced on a daily basis. The alignment of the syringe body with the injection ports of the GC and the autosampler are also checked on a daily basis.

The carrier gases are checked twice daily, on arrival in the morning and on departure in the evening. They are replaced when necessary or if there is a problem with the analysis on the GCs (e.g., if there is any baseline drift which is recurring and increasing in intensity, the carrier gas cylinder is replaced automatically and the supplier of the carrier gas is notified).

Other maintenance checks include: 1) teflon plunger tips on the syringes are replaced once per month, or more if necessary; 2) the carousel wheel and its mechanisms are adjusted when necessary; 3) columns are replaced as required based on loss of resolving capability or decrease in retention times (all other parameters constant) of more than five minutes.

All tubing is replaced when necessary. TARGET uses Supelco conditioned stainless steel premium grade 304 (1/8"OD x 21 mm ID).

APPENDIX C

ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

TABLE 1
ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Parameter	Analytical Method	Extraction Method	Sample Matrix	Container Size / Material	Sample Preservation*	Holding Times
Hardness	EPA 130.1, 130.2	N/A	Water	500 ml HDPE/Glass Jar	Chill, 4° C, HNO_3 or H_2SO_4 pH < 2	Six months until analysis
pH	EPA150.1, 150.2	N/A	Water	250 ml HDPE/Glass Jar	None required	Analyze imrnediately
Total Suspended Solids, non-filterable	EPA 160.2	N/A	Water	250 ml HDPE Jar	Chill, 4° C	7 days until analysis
Alkalinity	EPA310.1, 310.2	N/A	Water	250 ml HDPE/Glass Jar	Chill, 4° C	14 days until analysis
Nitrogen, as Ammonia	EPA 350.1, 350.2	N/A	Water	500 ml HDPE/Glass Jar	Chill, 4° C, H_2SO_4 pH < 2	28 days until analysis
Kjeldahl Nitrogen	EPA351.2, 351.3	N/A	Water	1 liter HDPE/Glass Jar	Chill, 4° C, H_2SO_4 pH c 2	28 days until analysis
		N/A	Soil	1 liter HDPE/Glass Jar	Chill, 4° C, H_2SO_4 pH < 2	28 days until analysis
Phosphorus, Orthophosphate	EPA 365.1, 365.2	N/A	Water	1 liter HDPE/Glass Jar	Filter immediately, Chill, 4° C	Analyze within 48 hours
Sulfide	EPA 376.1	N/A	Water	1 liter polyethylene	Chill, 4° C 0.04% zinc acetate NaOH pH>9	7 days until analysis
		N/A	Soil	1 liter polyethylene	Chill, 4° C 0.04% zinc acetate NaOH pH>9	7 days until analysis
Biological Oxygen Demand (BOD)	€PA 405.1	N/A	Water	One liter HDPE/Glass Jar	Chill, 4° C	Analyze within 48 hours

TABLE 1
ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Parameter	Analytical Method	Extraction Method	Sample Matrix	Container Size / Material	Sample Preservation'	Holding Timer
Chemical Oxygen Demand (COD)	EPA 410.1	N/A	Water	500 ml HDPE/Glass Jar	Chill, 4° C, H_2SO_4 pH < 2	28 days until analysis
Total Organic Carbon (TOC)	EPA 415.1, 415.2	N/A	Water	250 ml Glass Jar	Chill, 4° C, H_2SO_4 pH c 2	28 days until analysis
		N/A	Soil	250 ml Glass Jar	Chill, 4° C, H_2SO_4 pH < 2	28 days until analysis
Alpha emission, total, and beta emission, total (pCi/L)	EPA 900.0	N/A	Water	1 gallon HDPE/Glass Jar	HNO_3 pH < 2	6 months until analysis
		N/A	Soil	1 gallon HDPE/Glass Jar	HNO_3 pH c 2	6 months until analysis
Ignitability	EPA 1010	N/A	Water	250 ml Glass Jar	N/A	N/A
		N/A	Soil	Shelby tube	N/A	N/A
Metals + Cyanide	TAL	CLP	Water	One liter HDPE Jar + 16 ounce HDPE Jar for Cyanide	Chill, 4° C Metals - HNO_3 pH < 2 Cyanide - NaOH pH > 12	Metals - 6 months Mercury - 26 days Cyanide - 12 days
		CLP EPA 5030	Soil	(2) 8 ounce Glass Jar	Chill, 4° C	12 days until analysis
Volatile Organic Compounds	EPA 8010	EPA 5030	Water	(2) 40 ml. VOA Vials with Teflon-lined septa	Chill, 4° C, HCl[c]	14 days until analysis
		EPA 5030	Soil	(2) 8 ounce Glass Jars with Teflon-lined septa	Chill, 4° C, HCl[c]	14 days until analysis

TABLE 1
ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Parameter	Analytical Method	Extraction Method	Sample Matrix	Container Size / Material	Sample Preservation[a]	Holding Times
Semivolatile Organic Compounds (Base-Neutral/Acid Extractables)	EPA 8015	EPA 5030	Water	(2) 40 ml. VOA Vials with Teflon-lined septa	Chill, 4° C, HCl[c]	14 days until analysis
		EPA 5030	Soil	(2) 8 ounce Glass Jars with Teflon-lined septa	Chill, 4° C, HCl[c]	14 days until analysis
Organophosphorus Pesticides	EPA 8140	EPA 3510 or 3520	Water	250 ml Amber Glass Jar with Teflon-lined septum	Chill, 4° C[c]	Extract within 7 days, analyze within 40 days
		EPA 3540 or 3550	Soil	8 ounce Glass Jar	Chili, 4° C"	Extract within 14 days, analyze within 40 days
Chlorinated Herbicides	EPA 8150	NIA	Water	2.5 Liter Amber Glass Jar	Chill, 4° C[c]	Extract within 7 days, analyze within 40 days
		N/A	Soil	8 ounce Glass Jar	Chill, 4° C[c]	Extract within 14 days, analyze within 40 days
Dioxins	EPA 8280	NIA	Water	2.0 Liter Amber Glass	Chill, 4° C[c]	Extract within 7 days, analyze within 40 days
		NIA	Soil	8 ounce Glass Jar with Teflon-lined septum	Chill, 4° C	Extract within 14 days, analyze with 40 days

TABLE 1
ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Parameter	Analytical Method	Extraction Method	Sample Matrix	Container Size / Material	Sample Preservation*	Holding Times
Halogens, Total Organic (TOX)	EPA 9020, 9021	NIA	Water	250 ml HDPE/Glass Jar	Chill, 4° C, H_2SO_4 pH < 2	28 days until analysis
		NIA	Soil	250 ml HDPE/Glass Jar	Chill, 4° C, H_2SO_4 pH < 2	28 days until analysis
Cation Exchange Capacity (CEC)	EPA 9081	NIA	Soil	Shelby tube	N/A	N/A
Volatile Organic Compounds (VOC)	CLP	CLP	Water	(2) 40 ml VOA Vials with Teflon-lined septa	Chill, 4° C, HCl pH < 2, store in dark	10 days until analysis'
		CLP	Soil	(2) 8 ounce Glass Jars with Teflon-lined septa	Chill, 4° C, store in dark	10 days until analysis'
Semivolatile Organic Compounds (Base-Neutral/Acid Extractables)	CLP	CLP	Water	(2) One liter Amber Glass Jars	Chill, 4° C°	Extract within 5 days, analyze within 40 days'
		CLP	Soil	8 ounce Glass Jar with Teflon-lined septum	Chill, 4° C	Extract within 10 days, analyze within 40 days'
Pesticides/PCBs	CLP	CLP	Water	(2) One liter Amber Glass Jar	Chill, 4°C, 5 ≤ pH ≤ 9, store in dark	Extract within 5 days, analyze within 40 daysi'
		CLP	Soil	8 ounce Glass Jar	Chill, 4° C, store in dark	Extract within 10 days, analyze within 40 days'
Percent Moisture	ASTM D-2216-80		Soil	Shelby tube	NIA	NIA

TABLE 1
ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Parameter	Analytical Method	Extraction Method	Sample Matrix	Container Size / Material	Sample Preservation[a]	Holding Times
BTU Content	ASTM D-2015-77	N/A	Soil	Shelby tube	N/A	N/A
Ash Content	ASTM D-482	N/A	(Soil	Shelby tube	N/A	N/A
Sulfur	ASTM D-129-64	N/A	Soil	Shelby tube	N/A	N/A
Grain Size	ASTM D-422-63	N/A	Soil	Shelby tube	N/A	N/A

Notes:

CLP analyses performed in accordance with Statement of Work (SOW)3/90.

Soil samples collected with split barrel samplers shall be submitted to the laboratory in stainless steel sampling sleeves.

'Any preserving agents used must be ACS Certified Reagent Grade.

'Ascorbic acid should be added only in the presence of residual chlorine. Maximum holding time is 24 h w n when sulfide is preset. Optionally, all samples may be teated with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The umple ir filtered a d then NaOH is rdded to pH > 12.

'Sodium thiosulfate ($Na_2S_2O_3$), 0.008 %, should be added in the presence of free or combined chlorine.

'Samples receiving no pH adjustment must be analyzed within seven dryr of sampling.

The pH adjustment is *not* required if acrolein will *not* be measured. Sampler for acrolein receiving no pH adjustment m a t be analyzed within 3 dryr of sampling.

"hepH of the umple must be rdjuated to 7 to 10 if diphenylnitrosamine is to be determined.

[a]If umple will be extracted within 72 hours, pH adjustment is not required.

[b]If residual chlorine is present, sodium thiosulfate preservative (see note 3) is added to another container that is then filled.

[c]CLP holding timer are from Validated Time of Sample Receipt (VTSR).

N/A = *not* applicable. Extraction is not required or is part of the analytical method.

APPENDIX D

STANDARD FIELD FORMS

EN SAFE® / ALLEN + HOSHALL

Environmental and Safety Designs, Inc.

LOCATION OF BORING

JOB NO.	CLIENT	LOCATION

DRILLING METHOD:	BORING NO.
	SHEET
SAMPLING METHOD:	OF
	DRILLING
	START / FINISH

WATER LEVEL					TIME	TIME
TIME						
DATE					DATE	DATE
CASING DEPTH						

DATUM ELEVATION

SURFACE CONDITIONS:

SAMPLER TYPE	INCHES DRIVEN / INCHES RECOVERED	DEPTH OF CASING	SLEEVE NO. / SAMPLE DEPTH	BLOWS/6in SAMPLER	VAPOR CONCENTRATIONS (PPM)	DEPTH IN FEET	SOIL GRAPH
						0.5	
						1.0	
						1.5	
						2.0	
						2.5	
						3.0	
						3.5	
						4.0	
						4.5	
						5.0	
						5.5	
						6.0	
						6.5	
						7.0	
						7.5	
						8.0	
						8.5	
						9.0	
						9.5	
						0.0	

DRILLING _____

BY _____ CHK _____

DATE _____

CLNBORLG

SAMPLE LABEL

A Joint Venture for Professional Services

EnSafe/Allen & Hoshall

(901) 383-9115

Site:

Sample Number:

Presentative:

Analysis:

Date: _____

Time: _____

Sampler:

SECURITY SEAL

EnSafe ™ *ENVIRONMENTAL AND SAFETY DESIGNS, INC.* *5724 Summer Tree Dr.* *Memphis, Tennessee 38134* OFFICIAL SAMPLE SEAL	SAMPLE #	DATE:	SEAL BROKEN BY:
	SIGNATURE:		DATE:
	PRINT NAME & TITLE:		

NAVY CLEAN
ENSAFE/ALLEN & HOSHALL
(901) 383-9115

CHAIN OF CUSTODY RECORD

PAGE _____ OF _____

CLIENT _____ PROJECT MANAGER _____

ADDRESS _____ TELEPHONE NO. (901) 372-7962

PROJECT NAME/NUMBER _____ FAX. NO. (901) 372-2454

MEDIA STATUS: (A, B, OR C) _____ SAMPLERS: (SIGNATURE) _____

FIELD SAMPLE NUMBER	DATE	TIME	SAMPLE TYPE	TYPE/SIZE OF CONTAINER	PRESERVATION		NO. OF CONTAINERS	ANALYSIS REQUIRED								REMARKS
					TEMP.	CHEMICAL										

RELINQUISHED BY:	DATE	RECEIVED BY:	DATE	RELINQUISHED BY:	DATE	RECEIVED BY:	DATE
SIGNATURE		SIGNATURE		SIGNATURE		SIGNATURE	
PRINTED		PRINTED	TIME	PRINTED	TIME	PRINTED	TIME
COMPANY		COMPANY		COMPANY		COMPANY	
REASON		REASON		REASON		REASON	

METHOD OF SHIPMENT: _____
SHIPMENT NO. _____
SPECIAL INSTRUCTION: _____

COMMENTS: _____

AFTER ANALYSIS. SAMPLES ARE TO BE:
- ☐ DISPOSED OF (ADDITIONAL FEE)
- O STORED (90 DAYS MAX)
- O STORED OVER 90 DAYS (ADDITIONAL FEE)
- O RETURNED TO CUSTOMER'

036CUST1

GROUNDWATER SAMPLING

Sample IO: _____

PROJECT NAME _____ JOB NO: _____ DATE: _____

WELL NO. _____ LOCATION _____

WEATHER CONDITIONS _____ AMBIENT TEMP: _____

REVIEWED BY: _____ Personnel: _____

PURGING DEVICE

Type Device? _____

How was the device decontaminated? _____

How was the line decontaminated? _____

Which well was previously purged? _____

SAMPLING DEVICE

Type Device? _____

Mow was tho device decontaminated? _____

How was tho line decontaminated? _____

Which well was previously sampled? _____

INITIAL WELL VOLUME

Well diameter (in.) _____

Stickup (ft.) _____

Depth to bottom of well from TOC (ft.) _____

Depth to water surface from TOC (ft.) _____

Length of water (ft.) _____

Volume of water (ft.) _____

(gal.) _____

Amount of sediment at bottom of well (ft.) _____

3 Volumes of water (gal.) _____

PURGING

T i started _____ Finished _____

Volume purged _____

Comments on Well Recovery _____

Depth to water (ft.) _____

Completion _____

Additional Comments _____

Sample Collected: Start _____

Finish _____

IN-SITU TESTING

	1	2	3	4	5	6	7
Date:							
Time:							
Well Volume Purged (gal.)							
Turbidity							
Odor							
OVA (ppm)							
pH (units)							
Conductivity (µmho)							
Water Temperature (°C)							
TDS (mg/l)							
Depth to water (ft.)							

NOTES: 1 ft. length of 4" = 0.067 ft^3 or 0.65 gal. 1 ft. length 2" = 0.022 ft^3 or 0.16 gal.

Turbidity choices: clear, turbid, opaque

Revision Date: 8/5/92

AQUIFER TEST DATA

Owner _____ Location _____ Project No. _____

Date _____ Company Performing Test _____ Measured By _____

Well No. _____ Distance from Pumping Well _____ Type of loot _____ Toot No. _____

Measuring Equipment _____

Time Data

Pump On: Date _____ Time _____ (t)
Pump Off: Date _____ Time _____ (t')
Duration of Aquifer Test:
Pumping _____ Recovery _____

Water Level Data

Static Water Level _____
Measuring Point _____
Elevation of Measuring Point _____

Discharge Data

How Q Measured _____
Depth of Pump/Air Line ____
Previous Pumping?
Yes ___ No___
Duration _____ End _____

Comments on Factors Affecting Test Data

Time	Elapsed Clock Time	Time Since Rate Started t	Time Since Rate Stopped t'	t/t'		Water Level Measurement	Correction or Correction	Water Level	Water Level Change s or s'		Discharge Measurement	Rate	Pump (psf)	

SLUG TEST DATA SHEET

Well No. _____ Project No. _____

Date: _____ Owner: _____

Static Water Level: _____ ft. Location: _____

Depth to Bottom: _____ ft. Field Data by: _____ Calc. by: _____

Volume of Water Added ( ), or Bailed ( ), or Pumped ( ): _____ Gal.

Measurement Device _____ Water Level _____ Transducer _____ Other _____

Clock Time	Elapsed Time (Min.)	Reading	Head, H (Ft.)	Ratio H/Ho	Comments

Screened Interval ( ), Gravel Pack ( ), or Open Hole ( ) Length: L = _____ ft.

Casing Diameter: d = _____ ft., Depth: _____ ft., and Type: _____

Screen ( ), Gravel Pack ( ), or Open Hole Diameter ( ): D = _____ ft.

Calculated Head, Ho-calc. = Water Vol. _____ $ft.^3$ /Casing or Hole Area = _____ $ft.^2$

Ho-calc. = _____ ft. Ho-meas. _____ ft.

Boundary Cam Selected (Fig. 191 _____ Anisotropy: m = _____

Basic Time Lag: T = _____ min.

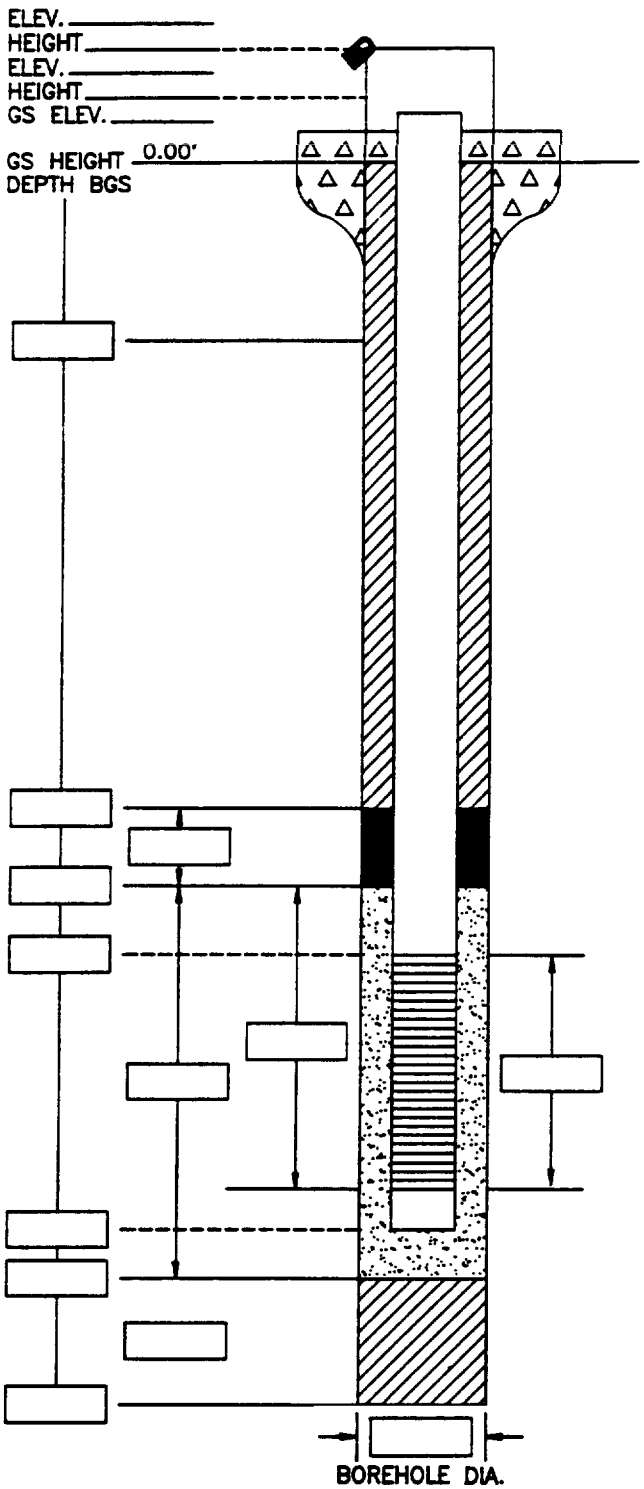
Kh = _____ ft./min. = _____ = _____

MEASUREMENT OF GROUNDWATER LEVELS

PROJECT NO.	DATE:
OWNER:	BY:
LOCATION:	METHOD OF MEASUREMENT:

Well Number	Time	Reference Point	Elevation of Reference	Depth to Product (feet)	Depth to Water (feet)	Water Elevation	Product Thickness	Adjusted Water Elevation	Notes

MONITORING WELL CONSTRUCTION LOG --STANDARD		
WELL NO.:	INSTALLATION:	SITE:
PROJECT NO.:	CLIENT/PROJECT:	
HAZWRAP CONTRACTOR:	DRIG CONTRACTOR:	
COMP. START: (: ____m)	COMP. END: (: ____m)	
BUILT BY:	WELL COORD.:	



PROTECTIVE CSG
MATERIAL/TYPE ____
DIAMETER ____
DEPTH BGS ____ WEEP HOLY(Y/N) ____

GUARD POSTS (Y/N)
NO. ____ TYPE ____

SURFACE PAD
COMPOSITION & SIZE ____

RISER PIPE
TYPE ____
DIAMETER ____
TOTAL LENGTH(TOC to TOS) ____
VENTILATED CAP (Y/N)

GROUT
COMPOSITION & PROPORTIONS ____
TREMIED (Y/N)
INTERVAL BGS ____

CENTRALIZERS (Y/N)
DEPTH(s) ____

SEAL
TYPE ____
SOURCE ____
SETUP/HYDRATION TIME ____
VOL FLUID ADDED ____
TREMIED (Y/N)

FILTER PACK
TYPE ____
AMT. USED ____
TREMIED (Y/N)
SOURCE ____
GR. SIZE DIST. ____

SCREEN
TYPE ____
DIAMETER ____
SLOT SIZE & TYPE ____
INTERVAL BGS ____

SUMP (Y/N)
INTERVAL BSG ____ LENGTH ____
BOTTOM CAP (Y/N)

BACKFILL PLUG
MATERIAL ____
SETUP/HYDRATION TIME ____
TREMIED (Y/N)

048MWCLG

APPENDIX E

SURFACE WATER SAMPLE COLLECTION PROCEDURES

GENERAL SURFACE WATER SAMPLE COLLECTION PROCEDURES

A study will be conducted of the hydrodynamics of the surface water body to determine the sample locations. Flow rates will be measured each day and recorded in the field logbook. A rain gauge will be installed at the sample location to document the rainfall influence.

- The collection point shall be located on a site map and described in the field logbook.

- The samples shall be collected from areas of least contamination to areas of worst contamination and from downstream to upstream.

- Surface water sample locations will be approached from the downstream direction to minimize sediment disturbance.

- The samples will be collected to minimize agitation of the underlying sediments.

- Surface water samples collected in water greater than 1-foot depth will be collected above the bottom with a Kemmerer sampling device. Surface water samples collected in water less than 1-foot depth will be collected at the water surface with the sample container, or Teflon or stainless steel sampling device.

- If surface water and sediment samples are to be collected at the same location, the surface water sample will be collected first.

- Three samples will be collected at each sample location. One sample will be collected at high tide, one sample at low tide and one sample will be collected between high and low tide.

- The sample container to be submitted for laboratory analysis shall be submerged with the mouth facing upstream.

- If use of the laboratory container is not possible, a decontaminated glass bottle, or Teflon or stainless steel sampling device will be used for sample collection.

- Field measurements for pH, specific conductance, salinity, flow rate and temperature of the sample will be collected and recorded.

- Samples will be chemically (as needed) and preserved to 4°C upon collection.

- Samples will be labeled and the chain-of-custody form completed in accordance with the procedures outlined in Sections 4.7 and 4.8 of this report.

The following information for each sample location will be recorded in the field logbook at a minimum: date of collection, time of collection (using the 24-hour clock), weather at the time of sample collection and any observable physical conditions (turbidity, odor, etc.) of the water.

APPENDIX F

SEDIMENT SAMPLE COLLECTION PROCEDURES

SEDIMENT *SAMPLE* COLLECTION GENERAL PROCEDURES

Sediment samples will be collected in accordance with the following guidelines:

- If sediment and surface water samples are to be collected at the same location, the surface water sample shall be collected first.

- The sediment sample collection point shall be located on a site map and referenced in the field logbook.

- Sediment samples shall be collected in a manner that minimizes disturbance.

- Sediment plumes and density currents will be avoided during sediment sample collection.

- A clean, decontaminated sampling device (hand auger) and gloves shall be used for each sediment sample collected.

- As soon as they are collected, the soil sample for **VOC analysis shall be placed** in the appropriate sample containers to minimize volatilization. **The VOC sample shall not be homogenized.**

- **Any other samples collected shall be composited and homogenized before placement in the appropriate sample containers.**

- Samples will be labeled and the chain-of-custody form completed in accordance with the procedures outlined in Sections 4.7 and 4.8 of this report.

- Samples will be preserved to 4° C as soon as they are collected.

The following information for each sample location shall be recorded in the field logbook at a minimum: Date of sample collection, time of sample collection (using the 24-hour clock), weather at the time of sample collection and any observable physical conditions (odor, discoloration) of the sample.

APPENDIX G

SPLIT-BARREL SAMPLE COLLECTION WITH LINERS

SPLIT-BARREL SAMPLE COLLECTION WITH LINERS

Before Sampling:

1. Stake the location(s) to be sampled.
2. Clear vegetation and debris from the ground surface.
3. Prepare the site by placing plastic sheeting around the hole site and over the ground on which the drill rig will be parked.
4. Set up drill rig at sampling location.
5. Set up the decontamination line for the split-barrel samplers and other down hole equipment. The drill rig will be decontaminated in accordance with the procedures outlined in Appendix O of this report.
6. Put on personal protective clothing and equipment as required by the site-specific health and safety plan.
7. Install four decontaminated 6" stainless steel liners in the split-barrel sampler.

During sampling:

8. Install a decontaminated split barrel sampler on the center rod(s) and insert into the hollow-stem auger. Connect the hammer assembly and lightly tap the rods to seat the drive shoe at the ground surface.
9. Mark the center rods in 6-inch increments from the top of the auger flight(s).
10. Drive the sampler using the hammer. Use a full 30-inch drop as specified by ASTM Method D-1586. Record the number of blows required to drive the sampler through each 6-inch increment.
11. Cease driving when the full length of the sampler has been driven or upon refusal. Refusal occurs when little (< 1 in) or no progress is made after 50 blows of the hammer.
12. Pull the sampler free by using upswings of the hammer to loosen the sampler. Pull out center rod and sampler.
13. Unscrew the split barrel assembly from the center rod and place it on the sampling table.
14. Remove the drive shoe and head assembly. If necessary, tap the split-barrel sampler assembly with a hammer to loosen threaded couplings.
15. With the drive shoe and head assembly off, split the sampler and remove the liners without disturbing the sample.

16. Screen sample with PID or FID, and select two samples with highest readings for laboratory analysis.
17. Immediately install a Teflon septa over the ends of the selected liners and cap and seal the ends of the liners with PVC caps.
18. Label the samples in accordance with Section 4.7 of this report. Mark the top and bottom of the sample on the outside of the liner.
19. Preserve to 4°C the samples collected.
20. Attach the hollow-stem auger with the cutting head and center rod(s).
21. Proceed to the next sampling depth.
22. Slightly raise the auger flight(s) to disengage the cutting head and rotate without advancement to clean the bottom of the hole of cuttings.
23. Collect samples as outlined above by inserting the split barrel sampler into the hollow-stem auger.
24. Describe the sample lithology from the auger cuttings and from observation of the bottom end of the sample in the liner on the soil boring logs.

After Sampling:

25. When sampling is complete, remove the drill rig to the heavy equipment decontamination site.
26. Decontaminate split-barrel sampler and other down hole equipment between sampling events.
27. Remove plastic sheeting. Place plastic and other disposables in the appropriate receptacles.
28. Note all relevant information in the field logbook before leaving the site.

APPENDIX H

SHELBY TUBE SAMPLE COLLECTION PROCEDURES

SHELBY TUBE SAMPLE COLLECTION PROCEDURES

The following steps shall be followed when collecting Shelby tube samples.

Before Sampling:

1. Survey and stake the location(s) to be sampled.
2. Clear vegetation and debris from the ground surface.
3. Prepare the site by placing plastic near the borehole.
4. Set up the decontamination line if required.

During Sampling:

5. Advance augers to the desired sample depth.
6. Attach a head assembly to a decontaminated Shelby tube. Attach the Shelby tube to the center rods.
7. Lower the Shelby tube and center rods into the hollow stem augers until seated at the bottom.
8. Use the rig's hydraulics to push the Shelby tube to its full length or until refusal. Attach a hoisting plug to the upper end of the center rod, twist to break off the sample and pull it out of the borehole with the rig winch.
9. Retrieve the Shelby tube to the surface, detach it from the center rod, and remove the head assembly.
10. Seal the ends of the Shelby tube immediately. Mark the top or up end of the tube and transport to the laboratory in an upright position.

After Sampling:

11. Upon completion of the boring, remove the drill rig for decontamination.
12. Dispose of cuttings in accordance with Section 4.9 of this SAP.
13. Remove plastic sheeting and place the plastic and any other disposables in the appropriate trash receptacle.
14. Note all relevant information in the field logbook before leaving the site.

Note: Shelby tube may not retain loose, sandy soils.

APPENDIX I

MONITORING WELL CONSTRUCTION PROCEDURES

MONITORING WELL INSTALLATION IN UNCONFINED AQUIFERS

1. Advance the borehole to the required depth using a bit or auger flight with PVC plug of a diameter sufficient to allow for insertion of the tremie pipe when the casing is centered. It is preferred that the borehole be at least 4 inches in diameter larger than the casing diameter. The hole should be drilled slightly deeper than required for the combined length of casing and screen. The final completion depth should be sounded with a decontaminated, weighted tape before continuance of well placement.
2. Condition the borehole by circulating air (or mud, if used) or by rotating augers without drilling until the hole is cleaned of cuttings. Remove the cuttings from the area round the auger.
3. Prepare the casing and screen for installation. **The casing and screen will be decontaminated in accordance with Appendix O except the PVC casing and screen not be cleaned with hot water or solvent rinsed.**
4. Withdraw the drill rods and bit or the auger flights. (If installing casing through hollow-stem augers, skip this step. Auger flights will be withdrawn after installation of casing.) Check the hole depth with a weighted surveyor's tape.
5. Hang the casing string, screen down, over or in the top of the borehole. Lower the casing string into the well.
6. Install centralizers every 30 to 40 feet as the casing string is lowered into the borehole. As a minimum requirement, one centralizer is required above the screen and bentonite seal. If a sediment trap is used, one centralizer must also be placed on the trap.
7. If a filter pack is required, prepare the dry sand.
8. When the casing string is set to the desired depth, hang the centered casing in place and insert the tremie pipe. There should be 2 to 3 feet of stickup once the well has been lowered to its final position unless the wellhead is flush mounted because of its location.
9. Install the filter pack through the tremie. Six inches or more of filter pack material must be spotted at the bottom of the hole, under the screen. The tremie should be slowly withdrawn so that the filter pack is placed evenly around the screen without bridging. Filter pack will be installed to at least 2 to 3 feet above the top of the screen. A minimum of 2 to 3 feet of bentonite is required above the screen.
10. Check the depth to the top of the filter pack with a weighted tape.

11. Do not remove the tremie pipe unless a larger diameter pipe is needed for installing the bentonite seal and grout.

12. Tremie bentonite pellets (not powder) onto the top of the filter pack. Bentonite pellets must be used if the seal is to be seated below the water table. Granular, flake, or slurried bentonite may be used above the water table. If a tremie pipe is used, slowly withdraw the pipe as the bentonite is added to ensure even placement of the bentonite seal around the annulus. Check the depth with a weighted tape.

13. If the bentonite seal is installed above the water table, hydrate according to the manufacturer's specifications. Allow adequate hydration time (8 hours) before proceeding to Step 15.

14. Mix Portland cement with 2 to 5 percent powdered bentonite and water to make a pumpable slurry. Weigh the bentonite before mixing; adding more than 5 percent bentonite will severely reduce grout strength.

15. Assume, for planning purposes, a shrinkage factor of 10 percent of the grout seal after installation. Grout exiting to the surface should be reweighed to ensure that the appropriate density is maintained and that a complete seal is formed.

16. Tremie the grout into the annulus using a side exiting tremie. Slowly withdraw the tremie pipe as the annulus fills. Grout the well to within 2 to 4 feet of the surface but not higher than the average frost line. Compare actual volume of grout placed with calculated volume. Both should be annotated in the field logbook. If the two values differ, explain the reason for the difference int he field logbook.

17. After installing grout, dismantle and decontaminate tremie equipment.

18. Mix and pour concrete for the wellhead pad. Concrete must extend below the frost line and to the top of grout. It may be convenient to first fill the annulus to the bottom of the pad form and then set the locking well cover or locking protective casing. After the protective casing has been installed, the remaining concrete should be poured into the pad form.

19. Finish the concrete pad so that it slopes away from the wellhead in all directions with a minimum thickness of 6 inches. If weather conditions warrant, cover the concrete until cured. Lock the well cover.

20. If the well design specifies traffic barriers, dig the holes and set the barrier posts in concrete separate from the concrete pad. Posts and concrete must extend to a depth of 2 feet.

21. Record the appropriate construction/completion information in the field logbook and on the appropriate field log forms.

22. If a form was used for the concrete pad, return to the well site after the concrete has cured for at least 24 hours and remove the form. Backfill around the pad with native soil. Drill two 0.25-inch weep holes on opposite sides of the protective casing and just above the concrete pad.

23. The well identification should be stamped in the protective casing. Paint the well posts.

MONITORING WELL INSTALLATION IN CONFINED AQUIFERS

1. Advance an oversized borehole through unconsolidated surface deposits to a depth of 2 to 3 feet into the top of the confining bed. In general, the borehole should be 4 inches in diameter larger than the casing to be installed when a tremie is to be used.

2. Condition the borehole by circulating air (or mud, if used) or by rotating augers without drilling until the hole is cleaned of cuttings.

3. While performing Step 2, prepare necessary length(s) of surface casing.

4. Pressure grout cement-bentonite grout to fill the portion of the borehole in the confining bed. If a bentonite seal is to be set below the static water level, only pellets may be used. The bentonite should be allowed to hydrate in the presence of groundwater, but potable or distilled water may be added if needed.

5. Insert the surface casing into the borehole and push firmly into the confining bed.

6. Mix Portland cement with bentonite powder (in a ratio of 3 to 5 pounds of bentonite to one 94-pound bag of cement) and water to make a pumpable slurry. Weigh the bentonite before mixing.

7. Insert the side exiting tremie pipe into the borehole and begin pumping grout. Slowly withdraw the tremie pipe as the annulus fills to ensure even placement with no bridging.

8. Allow grout to cure for 24 hours or longer before proceeding.

9. After grout has cured, continue drilling with a smaller diameter bit or auger string. Advance the borehole to the desired depth. The hole should be drilled a few feet deeper than necessary to allow for cavings during casing placement. If more than one aquifer will be encountered during drilling, the well must be cased in separate stages to prevent cross-contamination. Steps (1) through (8) of this procedure should be followed for each separate aquifer that must be cased off.

10. Condition the borehole by circulating air (or mud, if used) until the hole is cleaned of cuttings. Pull the drill string out of the borehole when no additional cuttings reach the surface. Check the hole depth with a weighted surveyor's type.

11. Prepare the casing string in manageable sections while conditioning the borehole. **The casing and screen must be decontaminated in accordance with Appendix O except PVC materials must not be washed with hot water or solvent rinsed.** Tighten casing joints to the manufacturer's specifications.

12. Insert the first segment of the casing string and lower to a convenient height for adding the second casing segment. Chock the casing, add the second segment, then release the chock and lower the casing. Repeat this process until the full casing string is hanging in the well. Centralizers, beginning at the top of the screen, should be placed at 30- or 40-foot intervals. The casing string should be allowed to hang in the well rather than set on the bottom. Casing strings with Teflon screens or schedule 40 PVC should never be set on the bottom because the weight of casing will significantly reduce the slot size and may collapse the screen.

13. If the well is to be completed without a filter pack, insert a decontaminated pump tube or submersible pump which will collapse unconsolidated sediments against the screen. Lower a weighted surveyor's tape to make sure that formation sands or gravels have collapsed to a distance of 2 ft. or more above the top of the screen. Continue pumping until sufficient collapse has occurred. If pumping does not cause collapse, then a filter pack should be spotted at the screen as in Step (14) of this procedure. This method is not intended for use with Teflon screens.

14. If a filter pack is required, as is most common, prepare the dry sand or sand slurry while proceeding with Step (12) of this procedure.

15. Insert the tremie to a location near the bottom of the screen and begin running the filter material through the tremie. Slowly withdraw the tremie so that the filter pack is placed

evenly around the screen without bridging. Six inches or more of filter pack material must be spotted at the bottom of the hole, under the screen. The filter pack will be installed to at least 2 to 3 feet above the top of the screens. If the top of the screen is below the bottom of the confining layer, extend the filter pack to the confining layer, if appropriate.

16. If the filter pack was installed dry, do not remove the tremie unless a larger diameter pipe is needed for installing the bentonite seal and grout. Check the depth of the top of the filter pack to ensure that it rises above the top of the screen.

17. Tremie the bentonite pellets onto the top of the filter pack. Bentonite pellets will be used where the seal is installed below the water table. The bentonite seal must extend 2 to 3 feet into the confining layer, if possible. Slowly withdraw the tremie pipe as bentonite is added to ensure even placement around the casing without bridging.

18. Hydrate the bentonite according to the manufacturer's specifications.

19. **Prepare the bentonite grout in accordance with the manufacturer's specifications.**

20. Tremie the grout into the annulus. Slowly withdraw the tremie pipe as the annulus fills to ensure even placement. Grout the well to within 2 to 4 feet of the surface but not above the average frost line.

21. After installing grout, dismantle and decontaminate the tremie equipment.

22. Mix and pour concrete for.the wellhead pad. Concrete must extend below the frost line and to the top of grout. It may be convenient to first fill the annulus to the bottom of the pad form and then set the locking well cover or locking protective casing. After the protective casing has been installed, the remaining concrete should be poured into the pad form.

23. Finish the concrete pad so that it slopes away from the wellhead in all directions, with a minimum thickness of 6 inches. If weather conditions warrant, cover the concrete until cured. Lock the well cover. If the well design specifies traffic barriers, dig the holes and set the barrier posts in concrete. Posts and concrete must extend to a depth of 2 feet.

24. Record the appropriate construction/completion information in the field logbook and on the appropriate field logbook forms.

25. If a form was used for the concrete pad, return to the well site after the concrete has cured for at least 24 hours and remove the form. Backfill around the pad with native

soil. Drill two 0.25-inch weep holes on opposite sides of the protective casing and just above the concrete pad.

26. The well identification should be stamped in the protective casing. Paint the well posts.

APPENDIX J

MONITORING WELL DEVELOPMENT PROCEDURES

MONITORING WELL DEVELOPMENT PROCEDURES

Monitoring wells will be developed using a combination of surging and pumping until readings of specific conductance, pH, and temperature stabilize and the groundwater withdrawn is free of turbidity. The procedures for development and stabilization will be discussed below.

surging

Monitoring wells will be developed initially with a surge block. The surge block will be constructed of polyvinyl chloride and will be lowered in the monitoring well with nylon rope. The surge block will be operated using an up and down motion so that groundwater will be surged in and out of the monitoring well screen. The procedure will be continued for approximately 10 minutes.

Pumping

Upon completion of the surging, the monitoring wells will be pumped using a Bennett pump, a Brainard Kilman pump or equivalent until the water withdrawn is free of turbidity. If the productivity of the monitoring well is low, the monitoring well shall be alternately pumped then left idle to allow for recovery. Groundwater withdrawn from the monitoring wells during development will be placed in 55-gallon drums for future disposal.

Stabilization

Monitoring wells will be developed until the groundwater withdrawn is free of turbidity and readings of pH, specific conductance, and temperature stabilize. Groundwater will be considered free of turbidity when groundwater withdrawn is below 0.5 milliliter per liter (ml/l) of settleable solids using an Imhoff Cone or as clear as possible given the subsurface conditions. Groundwater will be considered stabilized when three readings of specific conductance, pH, and temperature stabilize to within ± 5%, ± 0.1, and ± 1°C respectively or to within manufacturer's limitations. All of the equipment will be calibrated prior to use onsite and operated according to manufacturer's specifications.

APPENDIX K

MONITORING WELL PURGING PROCEDURES

MONITORING WELL PURGING PROCEDURES

- No less than three volumes of water (water column) shall be purged.
- Purging shall be conducted using a teflon bailer.
- Groundwater will be withdrawn until temperature, pH, and conductivity have stabilized before sampling. **A monitoring well is stabbed when three readings of temperature, pH, and specific conductance are within ±1°C, ±0.1, and ± 5% respectively.**
- Upgradient and background wells shall be purged and sampled before downgradient wells to reduce the risk of cross-contamination.
- Information concerning well purging shall be recorded in the field logbook and on the Groundwater Sampling Form.
- Low recharge wells are defined for this project as wells with a recharge rate that does not allow sampling within 12 hours of initial purging. These wells will be documented as dry.

APPENDIX L

GROUNDWATER *SAMPLE* COLLECTION PROCEDURES

PROCEDURES FOR GROUNDWATER *SAMPLING* WITH BAILERS

Before Sampling:

1. Put on protective clothing and equipment as specified in the site-specific health and safety plan.
2. Prepare the site for sample acquisition by covering the ground surface around the wellhead with plastic sheeting. Arrange the required sampling equipment for convenient use. If onsite decontamination is required, arrange the necessary supplies in a nearby but separate location, away from the wellhead.
3. *Open* the well and note the condition of the casing and cap. Check for vapors using a PID or FID equipment. Using a product interface probe, determine thickness of product (if present), the static water level and depth to well bottom. Record this information in the field logbook or on the groundwater sampling form.
4. Purge the well according to the procedures in Appendix K of this report. Allow the water level to recover to a depth at least sufficient for the complete submergence of the bailer without contacting the well bottom.
5. While the well is recovering from purging, decontaminate the Teflon coated stainless steel leader and bailer. If the bailer was decontaminated before arrival at the site, remove the protective wrappings. Securely attach the bailer to Teflon coated stainless steel leader. Attach the leader to the line securely. The end of the line should also be secured.
6. *Arrange* the sample containers in the order of use. Volatile organic analyte (VOA) samples, will be obtained first, followed in order by semivolatiles (SVOA) and other samples.

During sampling:

7. Lower the bailer into the well. The bailer should enter the water slowly to prevent d o n , particularly when VOA and SVOA samples axe being collected. Do not permit the bailer *to* contact the well bottom.
8. Retrieve the filled bailer to the surface. Do not allow the line to contact the ground. Hang the bailer from a bailer stand *or* other support, if available, or have an assistant hold it off the ground. The first bailer of water should be used as a rinse and then

discarded. For VOA samples, the sample bottles should be tilted when filling to prevent aeration. Check the filled vial for bubbles. If sample filtration is required, it should be done as soon as possible upon sample retrieval. If, after collecting VOA and SVOA samples, the total required sample volume is greater than the water remaining in the bailer, discard. Again lower the bailer to collect water for additional sample volume, if needed.

After Sampling:

9. Fill the remaining sample containers to capacity with the water contained within the bailer. Add preservative (if needed), cap, seal, and properly label all containers. Place the filled containers in the cooler(s) immediately.
10. Record sample types and amounts collected, and time and date of collection in the field logbook and on the groundwater sampling form. Prepare chain-of-custody and analytical request documents as required by the project quality assurance plan.
11. Decontaminate sampling equipment.
12. Clean up the area and place disposable materials (plastic sheeting, gloves, Tyvek) in the designated trash receptacle. Close and lock the well cover.

COMPENDIUM METHOD T0-14

THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AMBIENT AIR USING SUMMA @-PASSIVATED CANISTER SAMPLING AND GAS CHROMATOGRAPHIC ANALYSIS

QUALITY ASSURANCE DIVISION
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
U.S. ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NORTH CAROLINA 27711

MAY, 1988

METHOD TO14

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AMBIENT AIR USING SUMMA® PASSIVATED CANISTER SAMPLING AND GAS CHROMATOGRAPHIC ANALYSIS

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MTHOD TO14

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AMBIENT AIR USING SUMMA® PASSIVATED CANISTER SAMPLING AND GAS CHROMATOGRAPHIC ANALYSIS

1. Scope

1.1 This document describes a procedure for sampllng and analysis of volatile organic compounds (VOCs) in ambient air. The method is based on collection of whole air samples in SUMMA® passivated stainless steel canisters. The VOCs are subsqwntly separated by gas chromatography and measured by mass-selective detector or multidetector techniques. This method presents procedures for sampling Into canisters to final pressures both above and below atmospheric pressure (respectively referred to as pressurized and subatmospheric pressure sampllng).

102 This method is applicable to specific VOCs that have been tested and determined to be stable when stored in pressurized and sub atmospheric pressure canisters. Numerous compounds, many of which are chlorinated VOCs, have been successfully tested for storage stability in pressurized canisters (1,2). However, minimal documentation is currently available demonstrating stability of VOCs In subatmospheric pressure canisters.

1.3 The organic compounds that have been successfully collected In pressurized canisters by thfs method are listed in Table 1. These canpounds have been successfully measured at the parts per billion by volume (ppbv) level.

2. Applicable Documents

2.1 ASTM Standards

D1356 - Definition Of Terms Rclrttd to Atmospheric Sampling and Analysis
E260 - Recommended Practice for General Gas Chromatography Procedures
E355 - Practice for Gas Chromatography Terms and Relationships

2.2 Other Documents

U.S. Environmental Protectton Agency Technical Assistance Document (3)
Laboratory rnd Ambient Air Studies (4-17)

3. Summary of Method

3.1 Both subatmospheric pressure rnd pressurized sampling modes use an initially evacuated canister and 8 pump-ventilated sample line during sample collection. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of ambient air is drawn through 8 sampling train comprised of components that regulate the rate and duration of sampling into a pre-evacuated SUMMA® passivated canister.

3.2 After the air sample is collected, the canister valve is closed, an identification tag is attached to the canister. and the canister is transported to a predetermined laboratory for analysis.

3.3 Upon receipt at the laboratory, the canister tag data is recorded rnd the canister is attached to the analytical system. During analysis, water vapor is reduced in the gas stream by 8 Nafion® dryer (If applicable), rnd the VOCs are *then* concentrated by collection in 8 cryogenically-cooled trap. The cryogen is then removed and th temperature of the trap is raised. The VOCs originally collected in the trap are revolatilized, separated on 8 6C column, then detected by one or more detectors for identification and quantitation.

3.4 The analytical strategy for Method TO14 Involver using 8 high-resolution gas chromatograph (6C) coupled to one or more appropriate 6C detectors. Historically, detectors for 8 6C have been divided lnto two groups: non-specific detectors rnd specific detectors. The non-specific detectors include, but are not limited to, the nitrogen-phosphorus detector (NPD), the flame ionization detector (FID), the electron capture detector (ECD) and the photoionization detector (PID). The specific detectors include the mass spectrometer (MS) operating ln either the selected lon monitoring (SIM) mode or the SCAN mode, or the ton trap detector. The use of these detectors or a combination of these detectors as part of m analytical scheme is determiand by the required specificity and sensitivity of the appllcrtlon. While the non-specific detectors are less expensive per analysis and in some cases more sensitive than the specific detector, they vary in specificity and sensitivity for a specific class of compounds. For instance, if multiple halogenated compounds are targeted,

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an ECD is usually chosen; If only compounds containing nitrogen or phosphorus are of interest, a MPD can be used; or, if a variety of hydrocarbon canpounds are sought, the broad response of the FID or PID is appropriate. In each of these cases, however, the specific identification of the canpound within the class It determined only by its retention time, which can be subject to shifts or to interference from other nontargeted compounds. When misidentification occurs, the error is generally a result of a cluttered chromatogram, making peak assignment difficult. In particular, the more volatile organics (chloroethanes, ethyltoluenes, dichlorobenzenes, and various freons) exhibit less well defined chromatographic peaks, leading to mlsidentlflcatlon using non-specific detecton. Quantitative comparisons indicate that the FID is more subject to error than the ECD because the ECD is a much more selective detector for a smaller class of compounds which exhibits a stronger response. Identification errors, however, can be reduced by: (a) employing simultaneous dttcctlon by different detectors or (b) correlating retention times from different GC columns for confirmation. In either case, interferences on the non-specific detectors can still cause error In identifying a canplex sample, The non-specific detector system (GC-MPD-FID-ECD-PID), however, has been used for approximate quantltation of relatively clean samples. The nonspecific detector system can provide a "snapshot" of the constituents in the sample, allowing determination of:

- Extent of misidentification due to overlapping peaks,
- Position of the VOCs within or not within the concentration range of anticipated further analysis by specific detectors (GC-MS-SCAN-SIM) (if not, the sample is further diluted), and
- Existence of unexpected peaks which netd further identification by specific detectors.

On the other hand, the use of specific detectors (MS coupled to a GC) allows positive compound identification, thus lending itself to more specificity than the multidetector GC. Operating in the SIM mode, the MS can readily approach the same sensitivity as the

multidetector system, but its flexibility is limited. For SIM operation, the MS is programmed to acquire data for a limited number of targeted compounds while disregarding other acquired information. In the SCAN lode, however, the MS becomes a universal detector, often detecting compounds which are not detected by the multidetector approach. The GC-MS-SCAN will provide positive identification, while the GC-MS-SIM procedure provides quantitation of 8 restricted "target compound" list of VOCs.

The analyst often must decide whether to use specific or non-specific detectors by considering such factors as project objectives desired detection limits, equipment availability, cost and personnel capability In developing an analytical strategy. A list of some of the advantages and disadvantages associated with non-specific rnd specific detectors may assist the analyst in the decision-making process.

Non-Specific Multidetector Analytical System

Advantages	Disadvantages
o Somewhat lower equipment cost than GC-MS	o Multiple detectors to calibrate
o Less sample volume required for analysis	o Compound identification not positive
o More sensitive - ECD may be 1000 times more sensitive than GC-MS	o Lengthy data interpretation (one hour each for analysis and data reduction)
	o Interference(s) from co-eluting compound(s)
	o Cannot identify unknown compounds - outside range of calibration - without standards
	o Does not differentiate targeted compounds from interfering compounds

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Specific Detector Analytical System

GC-MS-SIM

Advantages	Disadvantages
o positive compound identification	o can't Identify non-specified compounds (ions)
o greater sensitivity than GC-MS-SCAN	o somewhat greater equipment cost than multidetector 6C
o less operator interpretation than for multidetector GC	o greater sample volume required than for multidetector GC
o can resolve co-eluting peaks	o universality of detector sacrificed to achieve enhancement in sensltlvity
o more specific than the multidetector GC	

GC-MS-SCAN

o positive compound identification	o lower sensitivity than GC-MS-SIM
o can identify all compounds.	o greater sample volume requirec than for multidetector GC
o less operator interpretation	o somewhat greater equipment cos than multidetector GC
o can resolve co-eluting perks	

The analytical finish for the measurement chosen by the analyst should provide a definitive identification and a precise quantitatlon of volatile organics. In a large part. the actual approach to these two objectives is subject to equipment availability. Figure 1 Indicates some of the favorite options that are used as an analytical flnlsh. The GC-MS-SCAN option uses a capillary column GC coupled to a MS operated in a scanning lode and supported by spectral library search routines, This option offers the nearest approximation to unambiguous identification and covers 8 wide range of compounds as defined by the completeness of the spectral library. GC-MS-SIM rode is limited to 8 set of target compounds which are user defined rnd Is more sensitive than GC-MS-SCAN by virtue of the longer dwell times at the restricted number of m/z values, Both these techniques, but especially the GC-MS-SIM option, can use a supplemental general non-specific detector to verify/identify the presence of VOCs. Finally, the option labelled GC-multidetector system uses a

combination of retention time and multiple general detector verification to identify compounds. However, interference due to nearly identical retention times can affect system quantitation when using this option,

Due to the low concentrations of VOCs encountered in urban air (typically less than 4 ppbv and the majority below 1 ppbv) along with their complicated chromatograms, Method TO-14 strongly recommends the specific detectors (GC-MS-SCAN-SIM) for positive identification and for primary quantitation to ensure that high-quality ambient data is acquired.

For the experienced analyst whose analytical system is limited to the non-specific detectors, Section 10.3 does provide guidelines and example chromatograms showing typical retention times and calibration response factors, and utilizing the non-specific detectors (GC-FID-ECD-PID) analytical system is the primary quantitative technique.

4. Significance

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4.1 VOCs enter the atmosphere from a variety of sources, Including petroleum refineries, synthetic organic chemical plants, natural gas processing plants, and automobile exhaust. Many of these VOCs are acutely toxic; therefore, their determination in ambient air is necessary to assess human health impacts.

4.2 Conventional methods for VOC determination use solid sorbent sampling techniques. the most widely used solid sorbent is Tenax®. An air sample is drawn through a Tenax®-filled cartridge where certain VOCs are trapped on the polymer. The sample cartridge is transferred to a laboratory and analyzed by GC-MS.

4.3 VOCs can also be successfully collected in stainless steel canister. Collection of ambient air samples in canisters provides (1) convenient integration of ambient samples over a specific time period, (e.g., 24 hours); (2) remote sampling and central analysis; (3) ease of storing and shipping samples; (4) unattended sample collection; (5) analysts of samples from multiple sites with one analytical system; and (6) collection of sufficient sample volume to allow assessment of measurement precision and/or analysis of

samples by several analytical systems. However, care must be exercised In selecting, cleaning, and handling sample canisters and sampllng apparatus to avoid-losses or contamination of the samples. Contamlnatlon is a critical issue wlth canister-based tampllng because the canister Is the last element in the tampllng train.

4.4 Interior surfaces of the canisters are treated by the SUMMA® passivation process, In which a pure chrome-nickel oxide is formed on the surface, This type of vessel has been used In the past for sample collection and has demonstrated sample storage stability of many specific organic compounds.

4.5 This method can be applied to sampllng and analysis of not only VOCs, but also some selected semivolatile organlc canpounds (SVOCs). The term "semivolatile organlc compounds" is used to broadly describe organlc canpounds that are too volatile to be collected by filtration air sampling but not volatile tnough for thermal desorption from solid sorbents. SVOCs can generally be classified as those wlth saturation vapor pressures at 25°C between 10^{-1} and 10^{-7} mm Hg. VOCs are generally classified as those organics having saturated vapor pressures at 25°C greater than 10^{-1} mm Hg.

5. Definitions

Note: Definitions used In this document and in any user-prepared Standard Operating Procedures (SOPs) should be consistent with ASTM Methods 01356, E260, and E355. All abbreviations and symbols within this method are defined at point of use.

5.1 Absolute canister pressure = Pg+Pa, where Pg = gauge pressure In the canister (kPa, psi) and Pa = barometric pressure (see 5.2).

5.2 Absolute pressure = Pressure measured wlth reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPa, mm Hg or psia.

5.3 Cryogen = A refrigerant used to obtaln very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is 1tquld oxygen (bp -183.0°C) or liquid argon (bp -185.7°C).

5.4 Dynamic calibration - Calibration of m analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed md by introducing such standards into the Inlet of the sampling or analytical system in a manner very similar to the normal sampling or analytical process.

5.5 Gauge pressure - Pressure measured above ambient atmospheric pressure (as opposed to absolute pressure), Zero gauge pressure is equal to ambient atmospheric (barometric) pressure.

5.6 MS-SCAN - The GC is coupled to a MS programmed in the SCAN mode to scan all ions repeatedly during the GC run. As used in the current context, this procedure serves as 1qualitative identification and characterization of the sample.

5.7 MS-SIM - The GC is coupled to 8 MS programmed to acquire data for only specified ions and to disregard all others. This is performed using SIM coupled to retention time discriminators. The GC-SIM analysis provides quantitative results for selected constituents of the sample gas as programmed by the user.

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5.8 Megabore® column - Chromatographic column having an internal diameter (I.D.) greater than 0.50 mm. The Megabore® column is A trademark of the J&W Scientific Co. For purposes of this method, Megabore® refers to chromatographic columns with 0.53 mm I.D.

5.9 Pressurized sampling - Collection of an air sample In a canister with a (final) canister pressure above atmospheric pressure, using a sample pump.

5.10 Qualitative accuracy - The ability of an analytical system to correctly identify compounds.

5.11 Quantitative accuracy - The ability of an analytical system to correctly measure the concentration of in identified compound.

5.12 Static calibration - Calibration of an analytical system using standards in a form different than the samples to be analyzed. An example of 8 static calibration would be injecting 8 small volume of 8 high concentration standard directly onto 8 GC column, bypassing the sample extraction and preconcentration portion of the rnrlytlcrl system.

5.13 Subatmospheric sampling - Collection of an air sample in an evacuated canister at a (final) canister pressure below atmospheric pressure, without the assistance of a sampling pump. The canister is filled as the internal canister pressure increases to ambient or near ambient pressure. An auxiliary vacuum pump may be used as part of the sampling system to flush the inlet tubing prior to or during sample collection.

6. Interferences and Limitations

6.1 Interferences can occur in sample analysis if moisture accumulates in the dryer (see Section 10.1.1.2). An autanated cleanup procedure that periodically heats the dryer to about 100°C while purging with zero air eliminates any moisture buildup. This procedure does not degrade sample integrity.

6.2 Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers) should be thoroughly cleaned to ensure that the filling apparatus will not conraminare samples. Instructions for cleaning the canisters and certifying the field sampling system are described in Sections 12.1 and 12.2, respectively.

6.3 Because the GC-MS analytical system employs a Nafion® permeable membrane dryer to remove water vapor selectively from the sample stream, polar organic compounds may permeate concurrent with the moisture molecule. Consequently, the analyst should quantitate his or her system with the specific organic constituents under examination.

7. Apparatus

7.1 Sample Collection

[Note: Subatmospheric pressure and pressurized canister sampling systems arc commercially available and have been used as part of U.S. Environmental Protection Agency's Toxics Air Monitoring Stations (TAM), Urban Air Toxic Pollutant Program (UATP), and the non-methane organic compound (NMOC) sampling and analysis program. 3

7.1.1 Subatmospheric Pressure (See Figure 2 Without Metal Bellows Type Pump)

7.1.1.1 Sampling inlet line - stainless steel tubing to connect the sampler to the sample inlet.

7.1.1.2 Sample canister - leak-free stainless steel pressure vessels of desired volume (e.g., 6 L), with valve and SUMMA® passivated Interior surfaces (Scientific Instrumentation Specialists, Inc., P.O. Box 8941, Moscow, ID 83843, or Anderson Samplers, Inc., 4215-C Wendell Dr., Atlanta, GA, 30336, or equivalent).

7.1.1.3 Stainless steel vacuum/pressure gauge - capable ot measuring vacuum (-100 to 0 kPa or 0 to 30 in Hg) and pressure (0-206 kPa or 0-30 psig) In the sampling system (Matheson, P.O. Box 136, Morrow, GA 30200, Model 63-3704, or equivalent). Gauges should be tested clean and leak tight.

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7114 Electronic mass flow controller - capable of maintaining a constant flow rate (± 10%) over a sampling period of up to 24 hours and under conditions of changing temperature (20-40°C) and humtdity (Tylan Corp., 19220 S. Normandie Ave., Torrance, CA 90502, Model FC-260, or equivalent).

7.1.1.5 Particulate matter filter - 2-um sintered stainless steel in-line filter (Nupro Co., 4800 E 345th St. Willoughby, OH 44094, Model SS-2F-K4-2, or equivalent).

7116 Electronic timer - for unattended sample collection (Paragon Elect. Co., 606 Parkway Blvd., P.O. Box 2[illegible], Twin Rivers, WI 54201, Model 7008-00, or equivalen.).

7.1.1.7 Solenoid valve - electrically-operated, bi-stable solenoid valve (Skinner Magnelatch Valve, New Britain, CT, Model V5RAM49710, or equivalent) with Viton® seat and o-rings.

7.1.1.8 Chromatographic grade stainless steel tubing and fittings - for interconnections (Alltech Associat., 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #8125,

or equivalent). All such materials In contact with sample, analyte, and support gases prior to analysis should be chromatographic grade stainless steel,

7.1.1.9 Thermostatically controlled heater - to maintain temperature inside insulated sampler enclosure above ambient temperature (Watlow Co., Pfafftown, NC, Part 04010080, or equivalent).

7.1.1.10 Heater thermostat - automatically regulates heater temperature (Elmwood Sensors, Inc., 500 Narragansett Park Dr., Pawtucket RI 02861, Model 3455-RC-0100-0222, or equivalent).

7.1.1.11 Fan - for cooling sampling system (EG&G Rotron, Woodstock, NY, Model SUZAI, or quivalent).

7.1.1.12 Fan thermostat - automatically regulates fan operation (Elmwood Sensors, Inc., Pawtucket, RI, Model 3455-RC-0100-0244, or equivalent).

7.1.1.13 Maximum-minimum thermometer - records highest and lowest temperatures durlng sampllng period (Thomas Scientific, Brooklyn Thermometer Co., Inc., P/N 9327H30, or quivalent).

7.1.1.14 Nupro stainless steel shut-off valve - leak free, for vacuum/pressure gauge.

7.1.1.15 Auxiliary vacuum pump - continuously draws ambient air to be sampled through the Inlet manifold at 10 L/min. or higher flow rate. Sample is extracted from the manifold at a lower rate, and excess air is exhausted. [Note: The use of higher Inlet flow rates dilutes any contamination present in the inlet md reduces the possibility of sample contamination as a result of contact with active adsorption sites on inlet walls.]

7.1.1.16 Elapsed time meter - measures duration of sampling (Conrac, Cramer Div., Old Saybrook, CT, Type 6364, PIN 10082, or equivalent).

7.1.1.17 Optional fixed orifice, capillary, or adjustable micrometering valve - may be used In lieu of the electronic flow controller for grab samples or short duration time-integrated samples. Usually appropriate only in situations where screening samples are taken to assess future sampling activity.

7.1.2 Pressurized (Figure 2 Ylth Metal Bellows Type Pump and Figu

7.1.2.1 Sample pump - stainless steel, metal bellows type (Metal Bellows Corp., 1075 Providence Highway, Sharon, MA 02067, Model MB-151, or equivalent), capable of 2 atmospheres *output* pressure, Pump must be free of leaks, clean, and uncontrainrted by oil or organic compounds. [Note: An alternative sampling system has been developed by Dr. R. Rasmussen, The Oregon Graduate Center (18,19) rnd Is illustrated in Figure 3. This flow system uses, in order, a pump, 8 mechanical flow regulator, rnd a mechanical compensating flow restrictive device. In this configuration the pump is purged with 8 large sample flow, thereby eliminating the need for an auxiliary vacuum pump to flush the sample inlet. Interference using this.configuration have been minimal.]

7.1.2.2 Other supporting materials - all other components - the pressurized sampling system (Figure 2 with meta bellows type pump and Figure 3) are similar to compc nents discussed in Sections 7.1.1.1 through 7.1.1.16

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7.2 Sample Analysis

7.2.1 GC-MS-SCAN Analytical System (See Figure 4)

7.2.1.1 The GC-MS-SCAN analytical system must be capable of acquiring and processing drtr in the MS-SCAN mode.

7.2.1.2 Gas chromatograph - capable of sub-ambient temperature programming for the oven, with other generally standard features such as gas flow regulators, automatic control of valves and integrator, etc. Flame ionization detector optional, (Hewlett Packard, Rt. 41, Avondale, PA 19311, Model 5880A, with oven temperature control and Level 4 BASIC programming, or equivalent.)

7.2.1.3 Chromatographic detector - mass-selective detector (Hewlett Packard, 3000-T Hanover St., 9B, Palo Alto CA 94304, Model HP-5970 MS, or equivalent), equipped with computer and appropriate software (Hewlett Packard, 3000-T Hanover St., 9B, Palo Alto, CA 94304

HP-216 Computer, Quicksilver K software, Pascal 3.0, mass storage 9133 HP Winchester with 3.5 inch floppy disk, or equivalent). The GC-MS Is set in the SCAN mode, where the MS screens the sample for identification and quantltatlon of VOC species.

7.2.1.4 Cryogenic trap with temperature control assembly - refer to Section 10.1.1.3 for complete description of trap rnd temperature control assembly (Nutech Corporation, 2142 Geer St., Durham, WC, 27704, Model 320-01, or equivalent).

7.2 .1.5 Electronic mass flow controllers (3) - maintain constant flow (for carrier gas and sample gas) and to provide analog output to monitor flow anomalies (Tylan Model 260, 0-100 cm^3/min, or equivalent).

7.2.1.6 Vacuum pump - general purpose laboratory pump, capable of drrwing the desired sample volume throug the cryogenic trap (Thomas Industries, Inc., Sheboy WI, Model 1078A20, or equivalent).

7.2.1.7 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings - refer to Sectio 7.1.1.8 for description.

7.2.1.8 Chromatographic column - to provide compound separa such as shown in Table 5 (Hewlett Packard, Rt. 41, Avondale, PA 19311, OV-1 capillary column, 0.32 mm 50 m with 0.88 um crosslinked methyl silicone coati or equivalent).

7.2.1.9 Stainless steel vacuum/pressure gauge (optional) - capable of measuring vacuum (-101.3 to 0 kPa) and p sure (0-206 kPa) In the sampling system (Matheson, Box 136, Morrow, GA 30200, Model 63-3704, or equiva lent). Gauges should be tested clean and leak tigl

7.2.1.10 Stainless steel cylinder pressure regulators - sta two-stage cylinder regulators with pressure gauges helium, zero air rnd hydrogen gas cylinders.

7.2.1.11 Cas purifiers (3) - used to remove organic impurit and moisture from gas streams (Hewlett Packard, Rt Avondale, PA, 19311, PW 19362 - 60500, or equivale

7.2.1.12 Low dead-volume tee (optional) - used to split the exit flow from the GC column (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #5839, or equivalent).

7.2.1.13 Nafion® dryer - consisting of Nafion tubing coaxially mounted within larger tubing (Perma Pure Products, 8 Executive Drive. Toms River, NJ, 08753 Model MD-125-48, or equivalent). Refer to Section 10.1.1.2 for description.

7.2.1.14 Six-port gas chromatographic valve - (Seismograph Service Corp, Tulsa, OK, Seiscor Model VIII, or equivalent).

7.2.1.15 Chart recorder (optional) - compatible with the detector output signals to record optional FID detector response to the sample.

7.2.1.16 Electronic integrator (optional) - compatible with the detector output signal of the FID and capable of integrating the area of one or more response peaks and calculating peak areas corrected for baseline drift.

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7.2.2 GC-MS-SIM Analytical System (See Figure 4)

7.2.2.1 The GC-MS-SIM analytical system must be capable of acquiring and processing data in the MS-SIM mode.

7.2.2.2 All components of the GC-MS-SIM system are identical to Sections 7.2.1.2 through 7.2.1.16.

7.2.3 GC-Multidetector Analytical System (See Figure 5 and Figure 6)

7.2.3.1 Gas chromatograph with flame ionization and electron capture detectors (photoionization detector optional) - capable of sub-ambient temperature programming for the OVM and simultaneous operation of all detectors, and with other generally standard features such as gas flow regulators, automatic control of valves and Integrator, etc. (Hewlett Packard, Rt. 41, Avondale, PA 19311, Model 5880A, with oven temperature control and Level 4 BASIC programming, or equivalent).

7.2 03.2 Chart recorders - compatible with the detector output signals to record detector response to the sample.

7.2.3.3 Electronic Integrator - compatible wlth the detector output signals and capable of integrating the area of one or more response peaks and calculating peak areas corrected for baseline drift,

7.2.3.4 Slx-port gas chromatographic valve - (Seismograph Service Corp, Tulsa, OK, Seiscor Model VIII, or equivalent)

7.2.3.5 Cryogenic trap wlth temperature control assembly - refer to Section 10.1.1.3 for complete descrlptlon of trap and temperature control assembly (Nutech Corporation, 2142 Geer St., Durham, NC 27704, Model 320-01, or equivalent).

7.2.3.6 Electronic mass flow controllers (3) - maintain constant flow (for carrier gas, nitrogen make-up gas and sample gas) and to provide analog output to monitor flow anomalies (Tylan Model 260, 0-100 cm^3/min, or equivalent).

723 .7 Vacuum pump - general purpose laboratory pump, capable of drawing the desired sample volume through the cryogenic trap (see 7.2.1.6 for source and description)

7.2.3 .8 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings - refer to Section 7.1. for description.

7.2.3.9 Chranatographic column - to provide compound separation such as shown in Table 7. (Hewlett Packard, Rt. 41, Avondale, PA 19311, OV-1 capillary column, 0.32 mm x 50 m wlth 0.88 um crosslinked methyl silicone coating, or equivalent). [Note: Other columns (e.g., OS-624) can be used as long as the system meets user needs, The wider Megabore® column (i.e., 0.53 mm I.D.) it less susceptible to plugging as a result of trapped water, thus eliminating the need for a Nafion® dryer in the analytical system. The Megabore® column has sample capacity approaching that of a packed column, while retaining much of the peak resolution traits of narrower columns (i.e., 0.32 mm I.D.).

7.2.3.10 Vacuum/pressure gauges (3) - refer to Section 7.2.1.9 for description.

7.2.3.11 Cylinder pressure stainless steel regulators - standard, two-stage cylinder regulators with pressure gauges for helium, zero air, nitrogen, rnd hydrogen gas cylinders.

7.2.3.12 Gas purifiers (4) - used to remove organic impurities and moisture from gas streams (Hewlett-Packard, Rt. 41, Avondale, PA, 19311, PW 19362 - 60500, or equivalent).

7.2.3.13 Low dead-volume tee - used to split (50/50) tht exit flow from the GC column (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #5839, or equivalent).

7.3 Canister Cleanlng System (See Figure 7)

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7.3.1 Vacuum pump - capable of evacuating sample canister(s) to an absolute pressure of <0.05 mm Hg.

7.3.2 Manifold - stainless steel manifold with connections for simultaneously cleaning several canisters.

7.3.3 Shut-off valve(s) - seven (7) on-off toggle valves.

7.3.4 Stainless steel vacuum gauge - capable of measuring vacuum in tk manifold to an absolute pressure of 0.05 mm Hg or lest.

7.3.5 Cryogenic trap (2 required) - stainless steel U-shaped open tubular trap cooled with liquid oxygen or argon to prevent contamination from back diffusion of oil from vacuum pump rnd to provide clean, zero air to sample canister(s).

7.3.6 Stainless steel pressure gauges (2) - 0-345 kPa (0-50 psig) to monitor zero air pressure.

7.3.7 Stainless steel flow control valve - to regulate flow of ze+o—.fr into canister(s).

7.300 Humidifier - pressurizable water bubbler containing high performance llquid chromatography (HPLC) grade deionized water or other system capable of providing moisture to tr zero rlr supply.

7.3.9 Isothermal oven (optional) for heating canisters (Fisher Scientific, Pittsburgh, PA, Model 349, or equivalent).

7.4 Calibration System and Manifold (See Flgure 8)

7.4.1 Calibration manifold.- glass manifold, (1.25 cm 1.0. x 66 cm) with sampling ports and Internal baffles for flow disturbance to ensure proper mixing.

7.4.2 Humidifier - 500-mL impinger flask contalnlng HPLC grade deionized water.

7.4.3 Electronic mass flow controllers - one 0 to 5 L/min and one 0 to 50 cm^3/min (Tylan Corporation, 23301-TS Wilmington Ave., Canon. CA. 90745, Model 2160, or quivrlent).

7.4.4 Teflon. filter(s) - 47-mm Teflon® filter for particulate control. kst source.

8. Reagents and Materials

8.1 Gas cylinders of helium, hydrogen, nitrogen, and zero air - ultrahigh purlty grade, best source.

8.2 Gas calibration standards - cylinder(s) containing approximately 10 ppmv of each of the following compounds of interest:

vinyl chloride
vlnylidene chloride
1,1,2-trichloro-1,2,2-trifluoroethane
chloroform
1,2-dichloroethane
benzene
toluene
Freon 12
methyl chloride
1,2-dichloro-1,1,2,2-tetrafluoroethane
methyl bromide
ethyl chloride
Freon 11
dichloromethane
1,1-dichloroethane
cis-1,2-dichloroethylene
1.2-dichl oropropane
1,1,2-trichloroethane
1,2-dibromoethane
tetrachloroethylene
chlorobenzene
benzyl chloride
hexachloro-1,3-butadiene
methyl chloroform
carbon tetrachloride
trichloroethylene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
ethyl benzene
o-xylene
m-xylene
p-xylene
styrene
1.1,2,2-tetrachloroethane
1.3,5-trimethyl benzene
1,2,4-trimethylbenzene
m-dichlorobenzene
o-dichlorobenzene
p-dichlorobenzene
1,2,4-trichlorobenzene

The cylinder(s) should be traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to a NBS/EPA approved Certified Reference Material (CRM). The components may be purchased In *OM* cylinder or may be separated into different cylinders. Refer to manufacturer's specification for guidance on purchasing and mixing VOCs in gas cylinders. Those compounds purchased should match one's own target list.

8.3 Cryogen - liquid oxygen (bp -183.0°C), or liquid argon (bp -185.7°C), best source.

8.4 Gas purifiers - connected in-line between hydrogen, nitrogen, and zero air gas cylinders rnd system inlet line, to remove moisture and organic impurities from gas streams (Alltech Associates, 2051 Waukegan Road, Deerfield, IL, 60015, or equivalent).

8.5 Deionized water - high performance liquid chromatography (HPLC) grade, ultrahigh purity (for humidifier), best source.

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8.6 4-bromofluorobenzene - used for tuning GC-MS, *best* source.

8.7 Hexane - for cleaning sampling system components, reagent grade, best source.

8.8 Methanol - for cleaning sampling system components, reagent grade, best source.

9, Sampling System

9.1 System Description

9.1.1 Subatmospheric Pressure Sampling [See Figure 2 (Without Met 1 Bellows Type Pump)]

9.1.1.1 In preparation for subatmospheric sample collection in a canister, the canister is evacuated to 0.05 ann Hg. When opened to the atmosphere containing the VOCs do k sampled, the differential pressure causes the sample *to* flow into the canister. This technique may be used to collect grab samples (duration of 10 to 30 seconds) or time-integrated samples (duration of 12 to 24 hours) taken through a flow-restrictive inlet (e.g., mass flow controller. critical orifice).

9.1.1.2 With a critical orifice flow restrictor, there will be a decrease In the flow rate as the pressure approaches atmospheric. However, with a mass flow controller, the subatmospheric sampling system can maintain a constant. flow rate from full vacuum to within about 7 kPa (1.0 psi) or less below ambient pressure.

9.1.2 Pressurized Sampling [See Figure 2 (With Metal Bellows Type Pun

9.1.2.1 Pressurized sampling Is used when longer-term integrated samples or higher volume samples are required. The sample is collected in a canister using a pump and flow control arrangement to achieve a typical 103-206 kPa (15-30 psig) final canister pressure. For example, a 6-liter evacuated canlster can be filled at 10 cm^3/min for 24 hours to achieve a final pressure of about 144 kPa (21 psig).

9.1.2.2 In pressurized canister sampling, a metal bellows typ pump draws in ambient air from the sampling manifold to fill and pressurize the sample canlster.

9.1.3 All Samplers

9.1.3.1 A flow control device is chosen to maintain a constan flow into the canister over the desired sample period This flow rate Is determined so the canister is fille (to about 88.1 kPa for subatmospheric pressure sampling or to about one atmosphere above ambient pressure for pressurized sampling) over the desired sample period. The flow rate can be calculated by

$$F = \frac{P \times V}{T \times 60}$$

where:

F = flow rate (cm^3/min).

P = final canister pressure, atmospheres absolute. P It approximately equal to

$$\frac{\text{kPa gauge}}{101.2} + 1$$

V = volume of the canister (cm^3),

T = sample period (hours).

For example, if a 6-L canister is to be filled to 202 kPa (2 atmospheres) absolute pressure In 24 hours, the flow rate can be calculated by

$$F = \frac{2 \times 6000}{24 \times 60} = 83 \ cm^3/min$$

9.1.3.2 For automatic operation, the timer Is wired to start and stop the pump at appropriate times for the deir sample period. The timer must also control the s le nold valve, to open the valve when starting the pump and close the valve when stopping the pump.

9.1.3.3 The use of the Skinner Magnelatch valve avoids any substantial temperature rise that would occur wit a conventional, normally closed solenoid valve that would have to be energized during the entire sam period. The temperature rise in the valve coil cause outgassing of organic compounds from the Vi valve seat material. The Skinner Magnelatch valve requires only a brief electrical pulse to open or close rt the appropriate start and stop times and therefore experiences no temperature increase. The pulses may be obtained either rlth rn electronic timer that can be programmed for short (5 to 60 seconds) ON periods, or with I conventional mechanical timer and 8 special pulse circuit. A simple electrical pulse circuit for operating the Skinner Magnelatch solenoid val e with a conventlonrl mechanical timer Is illustrated in Figure 9(a). However, with this simple circui the valve may operate unreliably during brief power interruptions or if the timer is manually switched on and off too fast. A better clrcult i comporating a time-delay relay to provide more liable valve operation is shown In Figure 9(b).

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9.1.3.4 The connecting lines between the sample inlet and th canister should be as short as possible to minimize their volume. The flow rate lnto the canister shoul remain relatively constant over the entire sampling period. If a critical orifice is used, some drop In the flow rate may occur near the end of the sample perlod as the canister pressure approaches the final calculated pressure.

9.1.3.5 As an option, a second electronic timer (see Section 7.1.1.6) may be used to start the auxiliary pump several hours prlor to the samplling perlod to flush and condition the inlet line.

9.1.3.6 Prior to field use, each sampling system must pass a humid zero air certification (see Section 12.2.2). All plumbing should be checked carefully for leaks. The canisters must also pass a humid zero air certification before use (see Section 12.1).

9.2 sampling Procedure

9.2.1 The sample canister should be cleaned and tested according to the procedure In Section 12.1.

9.2.2 A sample collection system is assembled as shown in Figure 2 (and Figure 3) and must meet certification requirements as outlined In Section 12.2.3. [Note: The sampling system should be contained In an appropriate enclosure.]

9.2.3 Prior to locating the samplling system, the user may want to perform "screening analyses" using a portable 6C system, as outlined in Appendix B, to determine potential volatile organics present and potential "hot spots." The information gathered from the portable 6t screening analysis would be used in developing I monitoring protocol, which Includes the sampling system location, based upon the "screening analysis" results.

9.2.4 After "screening analysis,' the sampling system IS located. Temperatures of ambient air rnd sampler box interior are recorded on canister sampling field data sheet (Figure 10). [Note: The following discussion is related to Figure 2.]

9.2.5 To verify correct sample flow. a "practice" (evacuated) canister is used in the sampling system. [Note: For a subatmospheric sampler, the flow meter and practice canister are needed. For the pump-driven system, the practice canister is not needed, as the flow can be measured at the outlet of the system.] A certified mass flow meter is attached to the inlet line of the manifold, just in front of the filter. The canister is opened. The sampler is turned on and the reading of the certified mass flow meter is compared to the sampler mass flow controller. The values should agree within ±10%. If not. the sampler mass flow meter needs to be recalibrated or there is r leak in the system. This should be Investigated and corrected. [Note: Mass flow meter readings may drift. Check the zero reading carefully and rdd or subtract the zero reading when reading or adjusting the sampler flow rate, to compensate for any zero drift.] After *two* minute' the desired canister flow rate is adjusted to the proper value (as indicated by the certified mass flow meter) by the sampler flow control unit controller (e.g., 35 cm^3/min for 24 hr, 7.0 cm^3/min for 12 hr). Record flnrl flow under 'CANISTER FLOW RATE,' Figure 10.

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9.2.6 The sampler is turned off and the elapsed time meter is reset to 000.0. Note: Any time the sampler is turned off, wait at least 30 seconds to turn the sampler back on.

9.2.7 The "practice" canister and certified mass flow meter are disconnected and a clean certified (see Section 12.1) canister is attached to the system.

9.2.8 The canister valve and vacuum/pressure gauge valve are open 1.

9.2.9 Pressure/vacuum in the canister is recorded on the canister sampling field data sheet (Figure 10) as indicated by the sampler vacuum/pressure gauge.

9.2.10 The vacuum/pressure gauge valve is closed and the maximum-minimum thermometer Is reset *to* current temperature. Time of day and elapsed time meter readings are recorded *On* the canister sampling field data sheet.

9.2.11 The electronic timer is set to begin and stop the sampling period at the appropriate times. Sampling commences and stops by the programmed electronic timer.

9.2.12 After the desired sampling period, the maximum, minimum, current interior temperature and current ambient temperature arc recorded on the sampling field data sheet. The current reading from the flow controller is recorded.

9.2.13 At the end of the sampllng period, the vacuum/pressure gauge valve on the sampler Is briefly opened and closed and the pressure/vacuum is recorded on the sampllng field data sheet. Pressure should be close to desired pressure. [Note: For a subatmospheric sampling system, If the canister is at atmospheric pressure when the field final pressure check is performed, the sampling perfod may be suspect. This information should be noted on the sampl-Ing field data sheet.] Time of day and elapsed time meter readings arc also recorded.

9.2.14 The canister valve is closed. The sampling line is disconnected from the canister and the canister is removed from the system. For a subatmospheric system, a certified mass flow meter is once rgaln connected to the inlet manifold in front of the in-line filter and a "practice" canister Is attached to the Magnelatch valve of the sampling system. The final flow rate is recorded on the canister sampllng field data sheet (set Figure 10). [Note: For a pressurized system, the final flow may be measured directly.] The sampler is turned off.

9.2.15 An identification tag is attached to the canister, Canister serial number, sample number, location, and date are recorded on the tag.

10. Analytical System (See Figures 4, 5 rnd 6)

10.1 System Description

10.1.1 GC-MS-SCAN System

10.1.1.1 The analytical system is comprised of a GC quipped with a mass-selective detector set In the SCAN mode (see Figure 4). All ions are sunned by the MS repeatedly during the

GC run, The system includes a computer and appropriate software for data acquisition, data reduction, and data reporting. A 400 cm^3 air-sample is collected from the canister into the analytical system. The sample air is first passed through 8 Nafion® dryer, through the 6-port chromatographic valve, then routed into a cryogenic trap. [Note: While the GC-multidetector analytical system does not employ a Nafion® dryer for drying the sample gas stream, it is used here because the GC-MS system utilizes 8 larger sample volume and is far r··· sensitive to excessive moisture than the GC-multidetector analytical system. Moisture can adversely effect detector precision. The Nafion® dryer also prevents freezing of moisture on the 0.32 mm I.D. column, which may cause column blockage and possible breakage.] The trap is heated (-160°C to 120°C in 60 sec) and the analyte is injected *onto* the OV-1 capillary column (0.32 mm x 50 m). [Note: Rapid heating of the trap provides efficient transfer of the sample components onto the gas chromatographic column.] Upon sample injection onto the column, the MS computer Is signaled by the GC computer to begin detection of compounds which elute from the column. The gas stream from the GC is scanned within a preselected range of atomic mass units (amu). For &tee-tion of compounds in fable 1, the range should be 18 to 250 amu, resulting in a 1.5 Hz repetitlon rate. Six (6) SUNS per eluting chromatographic peak are provided at this rate. 10-15 largest peaks are chosen by an automated data reduction program, the three scans neare the peak apex are averaged, and a background s traction is performed. A library search is the. performed and the top ten b a t matches for each peak are listed. A qualitative characterizatio

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of the sample is provided by this procedure, A typical chromatogram of VOCs determined by GC-MS SCAN is illustrated In Figure 11(a).

10.1.1.2 A Nafion® permeable membrane dryer is used to remove water vapor selectively from the sample stream. The permeable membrane consists of Nafion. tubing (a copolymer of tetrafluoroethyle and fluorosulfonyl monomer) that is coaxially mounted within larger tubing. The sample stream is passed through the Interior of the Nafion® tubing, allowing water (and other light, polar compounds) to permeate through the walls into a dry air purge stream flowing through the annular space between the Nafion® and outer tubing. [Note: To prevent excessive moisture build-up and any memory effects in the dryer, a clean-up procedure involving periodic heating of the dryer (100°C for 20 minutes) while purging with dry zero air (500 cm^3/min) should be implemented as part of the user's SOP manual. The clean-up procedure is repeated during each analysis (see Section 14, reference 7). Recent studies have indicated no substantial loss of targeted VOCs utilizing the above clean-up procedure (7). This cleanup procedure Is particularly useful when employing cryogenic preconcentration of VOCs with subsequent GC analysis using a 0.32 mm I.D. column because excess accumulated water can cause trap and column blockage and also adversely affect detector precision. In addition, the improvement in water removal fran the sampling stream will allow analyses of much larger volumes of sample air in the event that greater system sensitivity is rqulred for targeted compounds.]

10.1.1.3 The packed metal tubing used for reduced temperature trapping of VOCs is shown In Figure 1. The cooling unit is comprised of 8 0.32 cm outside diameter (O.D.) nickel tublng loop packed with 60-80 mesh Pyrex® beads (Nutech Model 320-01, or equivalent). The nickel tublng loop is wound onto 8 cylindrically formed tube heat (250 watt). A cartridge heater (25 watt) is sandwiched between pieces of aluminum plate at the trap inlet and outlet to provide additional heat to eliminate cold spots in the transfer tubing. During operrtlon, the trap Is inside a two-section stainless steel shell which is well insulated. Rrpld heating (-150 to +100°C in 55 s) is accomplished by direct thermal contact between the heater and the *trap* tubing. Cooling is achieved by vaporization of the cryogen. In the shell, efficient cooling (+120 to -150°C In 225 t) Is facilitated by confining the vaporized cryogen to the small open volume Surrounding the trap assembly. The trap assembly and chromatographic valve are mounted on a baseplate fitted into the injection and auxiliary tones of the 6C on rn insulated pad directly above the column oven when used with the Hewlett-Packard 5880 GC. [Note: Alternative trap assembly and connection to the GC may be used depending upon user's requirements.] The carrier gar line is connected to the injection end of the analytical column with a zero-dead-volume fitting that is usually held in the heated zone above the 6C oven. A 15 a x 15 a x 24 cm aluminum box It fitted over the sample hrndlln elements to complete the package. Vaporized cryogen is *vented* through the top of the box.

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10.1.1.4 As an option, the analyst may wish to split the gas stream exiting the column with A low dead-volume tee, passing one-third of the sample gas (1.0 mL/min) to the mass-selective detector and the remaining two-thirds (2.0 mL/min) through A flame ionization detector, at Illustrated as an option in Figure 4. The use of the specific detector (MS-SCAN) coupled with the non-specific detector (FID) enablts enhancement of data acquired from a single analysis. In particular, the FID provides the user:

- o Semi-real time picture of the progress of the analytical scheme;
- o Confirmation by the concurrent MS analysis of other labs that can provide only FID results; and
- o [illegible] other analytical laboratories with only GC-FID capability.

10.1.2 GC-MS-SIM System

10.1.2.1 The analytical system is canprlscd of A GC quipped with an OV-1 capillary column (0.32 mm x 50 m) and a mass-sclcctlvc detector set in the SIM mode (see Figure 4). The GC-MS is set up for automatic, repetitive analysis. The system Is programmed to acquire data for only the target compounds and to disregard all others. The sensitivity is 0.1 ppbv for a 250 cm^3 air sample with analytical precision of about 5% relative standard deviation. Con-ccntratlon of canpounds based upon a previously Installed calibration table It reported by an automated data reduction program. A Nafion® dryer is also employed by this analytical system prior to cryogenic preconcentration; therefore, many polar compounds are nut identified by thlr procedure.

10.1.2.2 SIM analysis is based on a combination of retention times and relative abundances of selected ions (see Table 2). These qualifiers are stored on the hard disk of the GC-MS computer and are applied for identification of each chromatographic peak. The retention time qualifier is determined to be ± 0.10 minute of the library retentlon time of the compound. The acceptance level for relative abundance is determined to be ± 15% of the expected abundance, except for rlnyl chloride and methylene chloride, which is determined to be ± 25%. Three ions are measured for most of the forty compounds. When compound identification is made by the computer, any peak that fails any of the qualifying test is flagged (e.g., with an *). All the data should be manually examined By the analyst to determine the reason for the flag and whether. the compound should be reported as found. While this adds some subjective judgment to the analysis, computer-generated identification problems can be clarified by an experienced operator. Manual inspection of the quantitative results should also be performed to verify concentrations outside the expected range. A typical chromatogram of VOCs determined by GC-MS-SIM mode is Illustrated In Figure 11(b).

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10.1.3 GC-Multidetector (GC-FID-ECD) System with Optional PID

10.1.3.1 The analytical system (see Figure 5) is comprised of a gas chromatograph equipped with a capillary column and electron capture and flame ionization detectors (see Figure 5) In typical operation, sample air from pressurized canisters is vented past the inlet to the analytical system from the canister at a flow rate of 75 cm^3/min. For analysis, only 25 cm^3/min of sample gas is used, while excess

is vented to the atmosphere. Sub-ambient pressure canisters arc connected directly to the Inlet. The sample gas stream is routed through a six port chromatographic valve and into the cryogenic trap for a total sample volume of 490 cm^3. [Note: This represents a 14 minute sampling period at a rate of 35 cm^3/min.] The trap (see Section 10.1.1.3) is cooled to -150°C by controlled release of a cryogen. VOCs and SVOCs are condensed on the trap surface while N_2, O_2, and other sample components are passed to the pump. After the organic canpounds are concentrated, the valve is switched and the trap is heated. The revolatilized compounds are transporttd by helium carrier gas at a rate of 4 cm^3/min to the head of the Megabore® OV-1 capillary column (0.53 mm x 30 m). Since the column initial temperature is at -50°C, the VOCs and SVOCs are cryofocussed on the head of the column. Then, the oven temperature is programmed to Increase and the VOCs/SVOCs in the carrier gas are chromatographically separated. The carrier gas containing the separated VOCs/SVOCs is then directed to two parallel detectors at a flow rate of 2 cm^3/min each. The detectors sense the presence of the speciated VOCs/SVOCs, and the response is recorded by either a strip chart recorder or a data processing unit.

10.1.3.2 Typical chromatograms of VOCs determined by the GC-FID-ECD analytical system are illustrated In Figures 11(c) and 11(d), respectively,

10.1.3.3 Helium is used as the carrier gas (4 cm^3/min) to purge residual air from the trap at the end of the sampling phase and to carry the revolatilized VOCs through the Megabore® GC column. Moisture and organic impurities are removed fcm the helium gas stream by a chemical purifier installed fn the GC (see

Section 7.2.1.11). After exiting the OV-1 Megabore® column, the carrier gas stream it split to the two detectors at fates of 2 cm^3/min each.

10.1.3.4 Gas scrubbers containing Drierite® or silica gel and SA molecular sieve are used to remove moisture and organic impurities from the zero air, hydrogen, and nitrogen gas streams. [Note: Purity of gar purifiers is checked prior to use by pasting humid zero-air through the gar purifier and analyzing according to Section 12.2.2.]

10.1.3.5 All lines should be kept as short as practical. All tubing used for the system should be chromatographic grade stainless steel connected with stainless steel fittings. After assembly, the system should be checked for leaks according to manufacturer's specifications.

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10.1.3.6 The FID burner air, hydrogen, nitrogen (make-up), and helium (carrier) flow rates should be set according to the manufacturer's instructions to obtaln an optimal FID response while maintaining a stable flame throughout the analysis. Typical flow rates are: burner air, 450 cm^3/min; hydrogen, 30 cm^3/min; nitrogen, 30 cm^3/min; helium, 2 cm^3/min.

10.1.3.7 The ECD nitrogen make-up gas and helium carrier flow rates should be set according to manufacturer's Instructions to obtrin m optimal ECD response. Typlcrl flow rates are: nitrogen, 76 cm^3/min and helium, 2 cm^3/min.

10.1.3.8 The GC-FID-ECD could be modified to include a PID (see Figure 6) for increased sensitivity (20). In the photoionization process, a molecule is ionized by ultraviolet light as follows $R + h\nu \rightarrow R^+ + e^-$, where R^+ is the ionized sp and a photon is represented by hv, with energy less than or equal to the ionization potential of

tk molecule, Generally all species with an ionization potential less than the ionization energy of the lamp are detected. Because the IonIration potential of all major components of air (02, N_2, .., CO_2, and H_2O) is greater than the ionization energy of lamps in general use, they are not detected. The sensor is comprised of an argon-filled, ultraviolet (W) light source where a portion of the organic vapors are ionized in the gas stream. A pair of electrodes arc contained in a chamber adjacent to the sensor. When a positive potential is applied to the electrodes, any ions formed by the absorption of W light are driven by the created electronic field to the cathode, and the current (proportional to the organic vapor concentration) is measured. The PID Is generally used for compounds having ionization potentials less than the ratings of the ultraviolet lamps. This detector is used for determination of most chlorinated and oxygenated hydrocarbons, aromatic compounds, and high molecular weight aliphatic compounds. Because the PID Is insensitive to methane, ethane, carbon monoxide. carbon dioxide, and water vapor, It Is an excellent detector. The electron volt rating is applied specifically to the wavelength of the most Intense emission line of the lamp's output spectrum. Some canpounds with ionization potentials above the lamp rating can still be detected due to the presence of small quantities of more Intense 'light. A typical system configuration associated with the GC-FID-ECD-PID Is Illustrated In Figure 6. This system It currently being used in EPA's FY-88 Urban Air Toxics Monitoring Program.

10.2 GC-MS-SCAN-SIM System Performance Criteria

10.2.1 GC-MS System Operation

10.2.1.1 Prior to analysis, the GC-MS system is assembl I and checked according to manufacturer's instructions.

10.2.1.2 Table 3.0 outlines general operating condition for the GC-MS-SCAN-SIM system with optional FID.

10.2.1.3 The GC-MS system is first challenged with humi zer fr (see Section 11.2.2).

10.2.1.4 Th. -MS and optional FID system is acceptabl if it contains less than 0.2 ppbv of targeted VOCs.

10.2.2 Dally GC-MS Tuning (See Figure 13)

10.2.2.1 At the beginning of each dry or prior to a calibration, the GC-MS system must be tuned to verify that acceptable performance criteria a achieved.

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10.2.2.2 For tuning the GC-MS, a cylinder containing 4-bromofluorobenzene is introduced via a sample loop valve injection system. [Mote: Some systems allow auto-tuning to facilitate this process.] The key ions and ion abundance criteria that must be met are Illustrated in Table 4. Analysis should not begin until all those criteria are met,

10.2 .2.3 The GC-MS tuning standard could also be used t assess GC column performance (chromatographic check) and as an internal standard. Obtain a background correction mass spectra of 4-bromofluorobenzene and check that all key ions criteria are met. If the criteria are not achieved, the analyst must retune the mass spectrometer nd repeat the test until all criteria are achie

1002.204 The performance criteria rust be achieved be. any samples, blanks or standards are analyzed. A

any key ion abundance observed for the daily 4-bromofluorobenzene mass tuning check differs by more than 10% absolute abundance from that obsem during the previous dally tuning, the instrument must be retuned or the sample and/or callbration gases reanalyzed until the above condition is met

10.2.3 GC-MS Calibration (See Figure 13)

[Note: Initial and routine callbratlon procedures are Illustrated in Figure 13.]

10.2.3.1 Initial Callbration - Initially, a multipoint dynamic callbration (three levels plus humid zero air) is performed on the GC-MS system, before sample analysis, with the assistance of a calibr. tlon system (see Figure 8). The calibration system uses NBS traceable standards or NBS/EPA CRMs in pressurized cylinders [containing a mixture of the targeted VOCs at nominal concentrations o 10 ppmv In nitrogen (Section 8.2)] as working standards to be diluted with humid zero air. Th contents of the working standard cylinder(s) are metered (2 cm^3/min) Into the heated mixing chamb where they are mixed wlth a 2-L/min humidified zero air gas stream to achieve a nominal 10 ppbv per compound callbratlon mixture (see Figure 8). This nominal 10 ppbv standard mixture is allowec to flow and equilibrate for a minimum of 30 minutes. After the equilibration period, the gas Standard mixture is sampled and analyzed by the real-time GC-MS system [see Figure 8(a) and Section 7.2.1]. The results of the analyses are averaged, flow audits are performed on the mass flow meters and the calculated concentration cor pared to generated values. After the GC-MS is calibrated at three concentration levels, a seci humid zero air sample is passed through the sys and analyzed. The second humid zero air test i used to verify that the GC-MS system is certifi clean (less than 0.2 ppbv of target compounds).

10.2.3.2 As rn alternative, 8 multipoint humid static calibration (three levels plus zero humid air) can be performed on the GC-MS system. During the humid static calibration analyses, three (3) SUMMA® passivated canisters are filled each at I different concentration between 1-20 ppbv from the calibration manifold using a pump and mass flow control arrangement [see Figure 8(c)]. The canisters are then delivered to the GC-MS to serve as calibration ttrndrrds. The canisters are analyzed by the MS in the SIM mode, each analyzed twice. The expected retention time and ion abundance (see Table 2 rnd Table 5) are used to verify proper operation of the GC-MS system. A calibration response factor is determined for each analyte, as illustrated in Table 5, and the computer calibration table is updated with this information, as illustrated in Table 60

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10.2.3.3 Routine Calibration - The GC-MS system is calibrated dally (and before sample analysis) wit.. 8 one-point calibration. The GC-MS system is calibrated either with the dynamic calibration procedure [see Figure 8(a)] or with a 6-L SUMMA® passivated canister filled with humid calibrat or standards from the calibration manifold (see Section 10.2.3.2). After the single point cal - bration, the GC-MS analytical system is challenge with a humidified zero gas stream to insure the analytical system returns to specification (le s than 02 ppbv of selective organics).

10.3 GC-FID-ECD System Performance Criteria (With Optional PID Syster. (See Figure 14)

10.3.1 Humid Zero Air Certification

10.3.1.1 Before system calibration and sample analys the GC-FID-ECD analytical system is assembled checked according to manufacturer's instructions

10.3.1.2 The GC-FID-ECD system is first challenged with humid zero air (see Section 12.2.2) and monitored.

10.3.1.3 Analytical systems contaminated with less than 0.2 ppbv of targeted VOCs are acceptable,

10.3.2 GC Retentlon Time Windows Determination (See Table 7)

10.3.2.1 Before analysis can be performed, the retentlon time windows must be established for tach analytc.

10.3.2.2 Make sure the GC system is within optimum operating conditions.

10.3.2.3 Make three injections of the standard containlng all compounds for retention tlmt window determination. [Note: The retention time window must be established for each analyte every 72 hours during continuous operation.]

10.3.2.4 Calculate the Standard deviation of the three absolute retentlon times for each single component standard. The retcntlon window is defined at the mean plus or minus three times the standard deviation of the individual retention times for each Standard. In those cases where the Standard deviation for A particular standard is zero, the laboratory must substitute the standard devlatlon of a closely-eluting, similar compound to develop 8 vrlfd rctcntlon time window.

10.3.2.5 The laboratory must calculate retention time windows for each standard (see Table 7) on each 6C column, whenever a new 6C column is installed or when major components of the 6C are changed. The data must be noted and retrined in a notebook by the laboratory as part of the user SOP and as 1quality assurance check of the analytical system.

10.3.3 GC Calibration

[Note: Initial and routine calibration procedures are illustrated in Figure 14.]

10.3.3.1 Initial Calibration - Initially, a multipoint dynamic calibration (three levels plus humid zero air) is performed on the GC-FID-ECD system, before sample analysis, with the assistance of a calibration system (see Figure 8). The calibration system uses NBS traceable standards or NBS/EPA CRMs In pressurized cylinders [containing a mixture of the targeted VOCs rt nominal concentrations of 10 ppav in nitrogen (Section 8.2)] as working standards to be diluted with humid zero air. The contents of the working standard cylinders are metered (2 cm^3/min) into tht heated mixing chamber where they are mixed witn a 2-L/min humidified zero air stream to achieve a nominal 10 ppbv per canpound calibration mixture (see Figure 8). This nominal 10 ppbv standard mixture is allowed to flow rnd equilibrate for an appropriate amount of time. After the equilibration period, the gar standard mixture is sampled and analyzed by the GC-MS system [see Figure 8(a)]. The results of the analyses are averaged, flow audits are performed on the mass flow controllers used to generate the standards and the appropriate response factors (concentration/area counts) are calculated for each canpound, as illustrated in Table 5. [Note: GC-FIDs are linear in the 1-20 ppbv range and may not require repeated multipoint calibrations; whereas, the GC-ECD will require frequent linearity evaluation.] Table 5 outlines typical calibration response factors

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and retention times for 40 VOCs. After the GC-FID-ECD is calibrated at the three concentration levels, a second humid zero air sample is passed through the system and analyzed. The second humid zero air test is used to verify that the GC-FID-ECD system is certified clean (less than 0.2 ppbv of target compounds).

10.3.3.2 Routine Calibration - A one polnt calibration is performed dally on the analytical system to verify the Initial multipoint callbrrtlon (see Section 10.3.3.1). The analyzers (GC-FID-ECD) are crllbrated (before sample analysis) using the static callbration procedures (see Section 10.2.3.2) Involving pressurized gas cylinders containing low concentrations of the targeted VOCs (10 ppbv) in nitrogen. After callbration, humid zero air is once again passed through the analytical system to verify residual VOCs are not present,

10.3.4 GC-FID-ECD-PID System Performance Crlteria

10.3.4.1 As an option, the user may wish to Include a photoionization detector (PID) to assist In peak Identification and increase sensitivity.

10.3.4.2 Thls analytical system is presently being used In U.S. Environmental Protection Agency's Urban Air Toxic Pollutant Program (UATP).

10.3.4.3 Preparation of the GC-FID-ECD-PID analytical system is identical to the GC-FID-ECD system (see Section 10.3).

10.3.4.4 Table 8 outlines typical retention times (minutes) for selected organics using tht GC-FID-ECD-PID analytical system.

10.4 Analytical Procedures

10.4.1 Canister Receipt

10.4.1.1 The overall condition of each sample canister is observed. Each canister should be received with an attached sample identification tag.

10.4.1.2 Each canister is recorded in the dedicated laboratory logbook. Also noted on the identification tag arc date received and initials of recipient,

10.4.1.3 The pressure of the canister is checked by attaching a pressure gauge to the canister Inlet, The canister valve is opened briefly rnd the pressure (kPa, psig) is recorded. [Note: If pressure is <83 kPa (<12 psig), the user may wish to pressurize the canisters, as an option, with zero grade nitrogen up to 137 kPa (20 psig) to ensure that enough sample is available for analysis. However, pressurizing the canister can Introduce additional error, increase the minimum detection limit (MDL), rnd is time consuming. The user should weigh these limitations as part of his program objectives before pressurizing.] Final cylinder pressure is recorded on canister sampling field data sheet (see Figure 10).

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10.4.1.4 If the canister pressure is Increased, a dilution factor (DF) is calculated and recorded on the sampling data sheet.

$$DF = \frac{Y_a}{X_a}$$

where:

X_a = canister pressure (kPa, psia) absolute before dilution.

Y_a = canister pressure (kPa, psia) absolute rfttr dilution.

After sample analysis, detected VOC concentrations are multiplied by the dilution factor to determine concentration In the sampled air.

10.4.2 GC-MS-SCAN Analysis (With Optional FID System)

10.4.2.1 The analytical system should be properly assembled, humid zero air certified (see Section 12.3), operated (see Table 3), and calibrated for accurate VOC determination.

10.4.2.2 The mass flow controllers are checked and adjusted to provide correct flow rates for the system.

10.4.2.3 The sample canister is connected to the Inlet of the GC-MS-SCAN (with optional FID) analytical system. For pressurized samples, a mass flow controller Is placed on the canister and the canister valve is opened and the canister flow Is vented past a tee Inlet to the analytical system at a flow of 75 cm^3/min so that 40 cm^3/min is pulled through the Nafion® dryer to the six-port chranatographic valve. [Note: Flow rate is not as important as acquiring sufficient sample volume.] Sub-ambient pressure samples are connected directly to the Inlet.

10.4.2.4 The GC Oven and cryogenic trap (inject position) are cooled to their set points of -50°C and -160°C, respectively.

10.4.2.5 As soon as the cryogenic trap reaches its lower set point of -160°C, the six-port chranatogrrphic valve is turned to its fill position to initiate sample collection.

10.4.2.6 A ten minute collection period of canister sample is utilized. [Note: 40 cm^3/min x 10 mtn = 400 cm^3 sampled canister contents.]

10.4.2.7 After the sample is preconcentrated In the cryogenic trap, the GC samplling valve is cycled to the Inject position and the cryogenic trap Is heated. The trapped analytes are thermally desorbed onto the head of the OV-1 capillary column (0.31 mm I.D. x 50 m length). The GC oven is programmed to start at -50°C and after 2 min to heat to 150°C at a rate of 8°C per minute.

10.4.2.8 Upon sample injection onto the column, the MS is signaled by the computer to scan the eluting carrier gas from 18 to 250 amu, resulting In 1.5 Hz repetition rate, This corresponds to about 6 scans per eluting chromatographic pea .

10.4.2.9 Primary identification Is based upon retention time and relative abundance of eluting ions as compared to the spectral library stored on the hard disk of the GC-MS datr computer.

10.4.2.10 The concentration (ppbv) is calculated using the previously established response factors (see Section 10.2.3.2), as illustrated in Table 5. [Note: If the canister is diluted before analysis, an appropriate multiplier is applied *to* correct for the volume dilution of the canister (Section 10.4.1.4).]

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10.4.2.11 The optional FID trace allows the analyst t record the progress of the analysis.

10.4.3 GC-MS-SIM Analysis (With Optional FID System)

10.4.3.1 When the MS is placed in the SIM mode of operation, the MS monitors only preselected ions, rather than scanning all masses continuously between two mass limits.

10.4.3.2 At a result, increased sensitivity and improved quantitative analysis can be achieved.

10.4.3.3 Similar to the GC-MS-SCAN configuration, the GC-MS-SIM analysis is b u d on e combination of retention times and relative abundances o selected ions (see Tabla 2 and tabla 5). Three qualifiers are stored on the hard disk of the GC-MS computer and are applied for identification of each chromatographic pert, *Once* the GC-MS-SIM has identified the peak, a cr tlon response factor is used to determine th analyte's concentration.

10.4.3.4 The Individual analyses are handled in three phases: data acquisition, data reduction, and data reporting. The data acquisition software is set In the SIM mode, where specific compound fragments are monitored by the MS at specific times In the analytical run. Data reduction Is coordinated by the postprocessing macro program that is automatically accessed after data acquisition is completed at the end of the 6C run. Resulting ion profiles are extracted, peaks are identified and Integrated, and an Internal Integration report is generated by the program, A reconstructed ion chromatogram for hardcopy reference is prepared by the program and various parameters of interest such as time, date, and Integration constants are prlnted. At the completlon of the macro program, the data reporting software Is accessed. The appropriate calibration table (see Table 9) is retrieved by the data reporting program from the computer's hard disk storage and the proper retention time and response factor parameters are applied to the macro program's integration file. With reference to certain pre-set acceptance criteria, peaks arc automatically identified and quantified and a final summary report is prepared, is illustrated In Table 10.

10.4.4 GC-FID-ECD Analysis (With Optional PID System)

10.4.4.1 The rnalytlcal system should be properly assembled, humid zero air certified (Set Section 12.2 and calibrated through a dynamic standard Calibration procedure (see Section 10.3.2). The FID detector is lit and allowed to stabilize.

10.4.4.2 Sixty-four minutes are required for each sample analysis • 15 min for system initialization, 14 min for sample collection, 30 min for analysis, and 5 min for post-time during which a report It printed. [Note: This may vary depending

10.4.4.3 The helium and sample mass flow controllers are checked and adjusted to provide correct flow rates for the system. Helium is used to purge residual air from the trap at the end of the sampling phase and to carry the revolatilized VOCs from the trap onto the GC column and into the FID-ECD. The hydrogen, burner air, and nitrogen flow rates should also be checked. The cryogenic trap is connected and verified to be operating properly while flowing cryogen through the system,

10.4.4.4 The sample canister Is connected to the Inlet of the GC-FID-ECD analytical system. The canister valve is opened and the canister flow Is vented past a tee Inlet to the analytical system at 75 cm^3/min using a 0-500 cm^3/min Tylan mass flow controller. During analysis, 40 cm^3/min of sample gas It pulled through the six-port chromatograph valve and routed through the trap rt the appropriate time while 'the extra sample Is vented. The VOCs are condensed In the trap while the excess flow Is exhausted through an exhaust vent, which assures that the sample air flowlng through the trap Is at atmospheric pressure.

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10.4.4.5 The six-port valve is switched to the Inject position and the canister valve Is closed,

10.4.4.6 The electronic integrator Is started,

10.4.4.7 After the sample Is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -50°C, the VOCs are cryofocussed on the column. Then, the oven temperature (programmed) increases and the VOCs elute from the column to the parallel FID-ECD assembly.

10.4.4.8 The parks eluting from the detectors are identified by retention time (see Table 7 and Table 8), while peak areas are recorded In area

counts. Figures 15 and 16 illustrate typical response of the FID and ECD, respectively, for the-forty (40) targeted VOCs. [Note: Refer to Table 7 for peak number and identification.]

10.4.4.9 The response factors (see Section 10.3.3.1) are multiplied by the area-counts for each perk to calculate ppbv estimates for the unknown sample. If the- canister is diluted before analysis, an appropriate dilution multiplier (DF) is applied to correct for the volume dilution of the canister (set Section 10.4.1.4).

10.4.4.10 Depending on the number of canisters to be analyzed, each canister is analyzed twice and the final concentrations for each analyte are the averages of the two analyses.

10.4.4.11 However, If the GC-FID-ECD analytical system discovers unexpected peaks which need further identification and separation of overlapping peaks are discovered, eliminating possible quantitation, the sample should then be subjected to a GC-MS-SCAN for positive identification and qwntltation.

11. Cleaning and Certification Program

11.1 canister Cleaning and Certification

11.1.1 All canisters must be clean and free of any contaminants before sample collection.

11.1.2 All canisters are leak tested by pressurizing them to approximately 206 kPa (30 psig) with zero air. [Note: The canister clernlng system In Figure 7 can be used for this task.] The initial pressure is measured, the canister valve is closed. and the final pressure is checked after 24 hours. If leak tight, the pressure should not vary more than ± 13.8 kPa (± 2 psig) over the 24 hour period.

11.1.3 A canister clernlng system may be assembled as illustrated in Figure 7. Cryogen is added to bath the vacuum pump md zero air supply traps. The canister(s)

are connected to the manifold. The vent shut-off valve and the canister valve(s) are opened to r· 'ease any remaining pressure In the canister(s). The ıcuum pump is started and the vent shut-off valve is ıen closed and the vacuum shut-off valve is opened. he ıanister(s) an evacuated to < 0.05 mm Hg (for at 1 st one hour). [Note: On a dally basis or more ofter f necessary, the cryogenic traps should be purged with zero air to remove any trapped water from previous canister cleaning cycles.

11.1.4 The vacuum and vacuum/pressure gauge shut-off valves are closed and the zero air Shut-off valve Is opened to pressurize the canister(s) with humid zero air to approximately 206 kPa *(30* psig). If a zero gas generator system Is used, the flow rate *ary* need to be limited to maintain the zero air qurllty.

11.1.5 The zero shut-off valve is closed and the canister(s) is allowed to vent down to atmospheric pressure through the vent shut-off valve. The vent strut-off valve is closed. Steps 11.1.3 through 11.1.5 are repeated two additional times for a total of three (3) evacuation/pressurization cycles for each set of canisters.

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11.116 At the end of the evacuation/pressurization cycle, the canister is pressurized to 206 kPa *(30* psig) with humid zero air. The canister is then analyzed by a GC-MS or GC-FID-ECD analytical system. Any canister that has not tested clean (compared to direct analysis of humidified zero air of less than 02 ppbv of targeted VOCs) should not be used, As *8* "blank" check of the canister(s) rnd cleanup procedure, the final humid zero rlr fill of 100% of the canisters is analyzed untll the cleanup system and canisters are proven reliable (less thrn 0.2 ppbv of targets VOCs). The check can then be reduced *to* a lower percentage of canisters.

11.1.7 The canister 1s reattached to the cleaning manifold and is then reevacuated to <0.05 mm Hg and remains in this condltlon until used, The canister valve is closed. canister is removed from the cleaning system and the canister connection is capped with a stainless steel fitting.

. The canister is now ready for collection of an air sample. An identification tag is attached to the neck of each canister for field notes and chain-of-custody purposes.

11.1.8 As an option to the humid zero air cleaning procedures. the canisters could be heated in an isothermal oven to 100°C during Section 11.1.3 to ensure that lower molecular weight compounds (C_2-C_8) are not retained on the walls of the canister. [Note: For sampling heavier, more complex VOC mixtures, the canisters should be heated to 250°C during Section 11.1.3.7.] Once kated, the canisters are evacuated to 0.05 mm Hg. At the end of the heated/evacuated cycle, the canisters are pressurized with humid zero air and analyzed by the CC-FID-ECD system. Any canister that has not tested clean (less than 0.2 ppbv of targeted cunpounds) should not be used. Once tested clean, the canisters are reevacuated to 0.05 mm Hg and remain in the evacuated state until used.

11.2 Sampling System Cleaning and Certification

11.2.1 Cleaning Sampling. System Components

11.2.1.1 Sample canponents are disassembled and cleaned before the sampler is assembled. Nonmetal llc parts are rinsed with HPLC grade deionized water and dried in a vacuum oven at 50°C. Typically, stainless steel parts and flttingt are cleaned by placing them In a beaker of methanol In an ultrasonic bath for 15 minutes. This procedure is repeated with hexane as the solvent.

11.2.1.2 The parts are then rinsed with HPLC grade deionized water and dried in a vacuum oven at 100°C for 12 *to* 24 hours.

11.2.1.3 Once the sampler is assembled, the entire system is purged with humid zero air for 24 hours.

11.2.2 Humid Zero Air Certification

[Note: In the following sections, "certification" is defined as evaluating the sampling system with humid

zero air and humid calibration gases that pass through all active components of the sampling system. The sys-tm is "certified" if no significant additions or deletions (less than ?? ppbv of targeted compounds) have occurred when challenged with the test gas stream.]

11.2.2.1 The cleanliness of the sampling system is determined by testing the sampler with humid zero rlr without an evacuated gas cylinder, Ui follows.

11.2.2.2 The calibration system and manifold are assembled, as Illustrated In Figure 8. The sampler (without an evacuated gas cylinder) is connected to the manifold rnd the zero air cylinder activated to generate a humid gas stream (2 L/min) to the calibration manifold [see Figure 8(b)].

11.2.2.3 The humid zero gas stream passes through the calibration manifold, through the sampling system (without an evacuated canister) to a GC-FID-ECD analytical system at 75 cm^3/min so that 40 cm^3/min is pulled through the six-port valve and route4 through the cryogenic trap (see Section 10.2.2.1) at the appropriate time while the extra sample is vented. [Note: The exit of the sampling system (without the canister) replaces the canister in Figure 4.] After the sample (400 mL) is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -50°C, the VOCs are cryofocussed on the column. Then, the oven temperature (programmed) increases rnd the VOCs begin to elute rnd are detected by a GC-MS (see Section 10.2) or the GC-FID-ECD (see Section 10.3). The analytical system should not detect greater than 0.2 ppbv of targeted VOCs in order for the sampling system to pass the humid zero air certification

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test, Chromatograms of a certified sampler and contaminated sampler are illustrated in Figures 17(a) and (b), respectively. If the sampler passes the humid zero air test, it is then tested with humid calibration gas standards containing selected VOCs at concentration levels expected in field sampling (e.g., 0.5 to 2 ppbv) as outlined in kctlon 11.2.3.

11.2.3 Sampler System Certification with Humid Calibration Gas Standards

11.2.3.1 Assemble the dynamic callbration system and manifold as illustrated in Figure 8.

11.2.3.2 Verify that the callbration system is clean (less than 0.2 ppbv of targeted compounds) by sampling a humidified gas stream, without gas callbratlon standards, with a previously certified clean canister (see Section 12.1).

11.2.3.3 The assembled dynamic. callbration system is certified clean if less than 0.2 ppbv of targeted compounds arc found.

11.2.3.4 For generating the humidified callbration standards, the callbratlon gas cylinder(s) (see Section 8.2) containing nominal concenttations of 10 ppmv in nitrogen of selected VOCs, are attached to the callbratlon system, as outlined in Section 10.2.3.1. The gas cylinders are opened and the gas mixtures art passed through 0 to 10 cm^3/min certified mass flow controllers to generate ppb levels of calibratlon standards.

11.2.3.5 After the appropriate quilibrium period, attach the sampling system (containing a certified evacuated canister) to the manifold, as illustrated In Figure 8(a).

11.2.3.6 Sample the dynamic calibration gas stream with the sampling system according to Section 9.2.1. [Note: To conserve generated calibration gas, bypass the canister sampling system manifold and attach the sampling system to the calibration gas stream at the inlet of the In-line filter of the sampling system so the flow will be less than 500 cm^3/min.]

11.2.3.7 Concurrent with the sampling system operation, realtime monitoring of the calibration gas stream is accomplished by the on-line GC-MS or GC-multidetector rnalytlcrl system [Figure 8(b)] to provide reference concentrations of generated VOCs.

11.2.3.8 At the end of the sampling period (normally same time period used for anticipated sampling), the sampling system canister is analyzed and compared to the reference GC-MS or GC-multidetector analytical system io determine if the concentration of the targeted VOCs was Increased or decreased by the sampling system.

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11.2.3.9 A recovery of between 90% and 110% is expected for all targeted VOCs.

12. Performance Criteria and Quality Assurance

12.1 Standard Operating Procedures (SOPs)

12.1.1 SOPs should be generated In each laboratory describing and documenting the following activities: (1) assembly, calibration, leak check, and operation of specific sampling systems rnd equipment used; (2) preparation, storage, shipment, and handling of samples; (3) assembly, leak-check, calibration, and operation Of the analytical system, addressing the specific equipment used; (4) canister storage and cleaning; and (5) all aspects of data recording and processing, including lists of computer hardware and software used.

12.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the laboratory personnel conducting the work.

12.2 Method Relative Accuracy and Linearity

12.2.1 Accuracy can be determined by injecting VOC standards (see Section 8.2) from an audit cylinder into a sampler. The contents are then analyzed for the components contained In the audit canister. Percent relative accuracy is calculated:

$$\% \text{ Relative Accuracy} = \frac{X - Y}{X} \times 100$$

Where: Y = Concentration of the targeted cunpound recovered from sampler.

X = Concentration of VOC targeted compound In tht NBS-SRM or EPA-CRM audit cylinders.

12.2.2 If the relative accuracy does not fall between 90 and and 110 percent, the field sampler should be removed from use, cleaned, and recertified accoming to initial certification procedures outlined in Section 11.2.2 and Section 11.2.3. Historically, concentrations of carbon tetrachloride, tetrachloroethylene, and hexachlorobutadiene have sometimes been detected at lower concentrations when using parallel ECD and FXD detectors. When these three canpounds arc present at concentrations close to calibration levels, both detectors usually agree on the reported concentrations. At concentrations below 4 ppbv, there is a problem with nonlinearity of the ECD, Plots of concentration versus peak area for calibration canpounds detected by the ECD have shown that the curves are nonllnear for carbon tetrachloride, tetrachloroethylene, and hexachlorobutadiene, as illustrated in Figures 18(a) through 18(c). Other targeted ECD and FID compounds scaled linearly for the range 0 to 8 ppbv, as shown for chloroform in Figure 18(d). For canpounds that arc not linear over the callbration

range, area ewntr generally roll off between 3 and 4 ppbv. To correct for the nonlinearity of these compounds, an additional calibration step Is performed. An evacuated stainless steel canister It pressurized with calibration gas at a nominal concentration of 8 ppbv. The sample It then diluted to approximately 3.5 ppbv with zero air and analyzed. The instrument response factor (ppbv/area) of the ECD for each of the three compounds is calculated for the 3.5 ppbv sample, Then, both the 3.5 ppbv and the 8 ppbv response factors are entered into the ECD crllbrrtlon table, The software for the Hewlett-Packard 5880 level 4 GC Is designed to accommodate multilevel crlibratlon entries, so the correct response factors are rutanatlcrlly calculated for concentrations In this range.

12.3 Method Modification

12.3.1 Sampling

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12.3.1.1 The sampling system for pressurired canister sampling could be modified to use a lighter, more compact pump. The pump currently bclng used weighs about 16 kilograms (35 lbs). Commercially available pumps that could be used as alternatives to the prescribed sampler pump are described below. Metal Bellows MB-41 pump: These pumps are cleaned at the factory; however some precaution should be taken with the circular (4.8 cm diameter) Teflon. rnd stainless steel part directly under the flange, It Is often dirty when received and should be cleaned before use. This part is cleaned by removing it from the pup, manually cleaning with deionized water, and placing in a vacuum oven at 100°C for at least 12 hours. Exposed parts of the pump head are also cleaned with swabs and allowed to air dry. These pumps have

proven to be very reliable; however, they are only useful up to an outlet pressure of about 137 kPa (20 psig). Neuberger Pump: Viton gaskets or seals must be specified with this pump. The 'factory direct' pump is received contaminate and leaky. The pump is cleaned by disassembling the pump head (which consists of three stainless steel parts and two gaskets), cleaning the gasket with deionized water and drying in a vacuum oven, and remachining (or manually lapping) the sealing surfaces of the stainless steel parts. The stainless steel parts are then cleaned with methanol, hexane, deionized water and heated in a vacuum oven. The cause for most of the problems with this pump has been scratches on the metal parts of the pump head. Once this rework procedure is performed, the pump is considered clean and can be used up to about 240 kPa (35 psig) output pressure. This pump is utilized in the sampling system illustrated in Figure 3.

12.3.1.2 Urban Air Toxics Sampler

The sampling system described in this method can be modified like the sampler in EPA's FY-88 Urban Air Toxics Pollutant Program. This particular sampler is described in Appendix C (see Figure

12.3.2 Analysis

12.3.2.1 Inlet tubing from the calibration manifold could be heated to 50°C (same temperature as the calibration manifold) to prevent condensation on the internal walls of the system.

12.3.2.2 The analytical strategy for Method TO-14 involves positive identification and quantitation by GC-MS-SCAN-SIM mode of operation with optional FID. This is a highly specific and sensitive detection technique. Because a specific detector system (GC-MS-SCAN-SIM) is more complicated and expensive than the use of non-specific det

(GC-FID-ECD-PID), the analyst may want to perfc.m a screening analysis and preliminary quantitation of VOC species in the sample, includir any po' r compounds, by utilizing the GC-multidetector (GC-FID-ECD-PID) analytical system prior to GC· S analysis. This system can be used for approximat quantitation. The GC-FID-ECD-PID provides a ": a shot' of the constituents in the sample, allow-lng the analyst to determine:

- Extent of misidentification due to overlapping peaks,
- Whether the constituents are within the calibration range of the anticipated GC-MS-SCAN-SIM analysis or does the sample require further dilution, and
- Are there unexpected perks which need f' identification through GC-MS-SCAN or ar. there peaks of interest needing attentic

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If unusual perks are observed from the GC-FID-E)-PID system, the analyst then performs a GC-MS-S.AN analysis, The GC-MS-SCAN will provide positive identification of suspect peaks from the GC-FID ECD-PID system. If no unusual peaks are identified and only a select number of VOCs are of co . cern, the analyst can then proceed to GC-MS-SIM. The GC-MS-SIM is used for final quantitation of selected VOCs. Polar compounds, however, cannot be identified by the GC-MS-SIM due to the use of a Nafion® dryer to remove rater from the sam.le ptlor to analysis. The dryer removes polar can-pounds al— with the water, The analyst often has to make this decision incorporating project objectives, detection limits, equipment availa-bility, cost rnd personnel capability in deve' ing an analytical strategy. Figure 20 outlin.. the use of the GC-FID-ECD-PID as a "screening" approach, with the GC-MS-SCAN-SIM for final identification and quantitation.

12.4 Method Safety

This procedure may Involve hazardous materials, operattons, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the user's responsibility to establish appropriate safety rnd htrlth practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

12.5 Quality Assurance (See Figure 21)

12.5.1 Sampling System

12.5.1.1 Section 9.2 suggests that a portable GC system be used as a "screening analysis' prior to locating fixed-site samplers (pressurized or subatmospheric)

12.5.1.2 Section 9.2 requires pre and post-sampling measurements with a certified mass flow controller for flow verification of sampling system.

12.5.1.3 Section 11.1 requires all canisters to be pressure tested to 207 kPa ± 14 kPa (30 psig ± 2 psig) over a period of 24 hours.

12.5.1.4 Section 11.1 requires that all canisters be certified clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.

12.5.1.5 Section 11.2.2 requires all field sampling systems to be certified initially clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.

12.5.1.6 Section 11.2.3 requires all field sampling systems to pass an initial humidified calibration gas certification [at VOC concentration levels expected in the field (e.g., 0.5 to 2 ppbv)] with a percent recovery of greater than 90.

12.5.2 GC-MS-SCAN-SIM System Performance Criteria

12.5.2.1 Section 10.2.1 requires the GC-MS analytical system to be certified clean (less thrn 0.2

ppbv of targeted VOCs) prior to sample analysis, through a humid zero air certification.

12.5.2.2 Section 10.2.2 requires the daily tuning of the GC-MS with 4-bromofluorobenzene (4-BFB) and that it meet the key ions and ion abundance criteria (10%) outlined In Table 5.

12.5.2.3 Section 10.2.3 requires both an initial multipoint humid static calibration (three levels plus humid zero air) and 8 daily calibration (one polnt) of the GC-MS analytical system.

12.5.3 GC-Multidetector System Performance Criteria

12.5.3.1 Section 10.3.1 rqufres the GC-FID-ECD analytical system, prior to analysis, to be certified clean (less than 0.? ppbv of targeted VOCs) through 8 humid zero air certification.

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12.5 .3.2 Section 10.3.2 requires that the GC-FID-ECD analytical system establish retention time windows for each analyte prior to sample analysis, when a new GC column is installed, or major components of the GC system altered since the previous determination.

12.5 .3.3 Section 8.2 requires that all calibration gases be traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to a NBS/EPA approved Certified Reference Material (CRM).

12.5.3.4 Section 10.3.2 requires that the retention time window be established throughout the course of a 72-hr analytical pcrlod.

13.5.3.5 Section 10.3.3 requires both an initial multipoint calibration (three levels plus humid zero air) rnd a daily calibration (one point) of the GC-FID-ECD analytical system with zero gas dilution of NBS traceable or NBS/EPA CRMs gases. [Note: Gas cylinders of VOCs at the ppm and ppb level are available for audits from the USEPA, Environmental Monitoring System-

Laboratory, Quality Assurance Division, MD-77B, Research Triangle Park, NC 27711, (919)541-4531. Appendix A outlines five groups of audit gas cylinders available from USEPA.]

13. Acknowledgements

The determination of volatile and some semi-volatile organic compounds In ambient air Is a complex tusk, primarily because of the wide variety of compounds of Interest and the lack of standardized sampling and analytical procedures. While there are numerous procedures for sampling and analyzing VOCs/SVOCs In ambient air, this method draws upon the best aspects of each one and combines them into a standardized methodology. To that end, the following individuals contributed to the research, documentation and peer review of this manuscript.

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Topic	Contact	Address	Telephone no.
Sampling System	Mr. Frank McElroy Mr. Vince Thompson	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-77 Research Triangle Park, N.C. 27711	919-541-2622 919-541-3791
	Dr. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Mr. Tom Merrifield	Anderson Samplers, Inc. 4215-C Wendell Drive Atlanta, GA 30336	1-800-241-6898
	Mr. Joseph P. Krasnec	Scientific Instrumentation Specialists, Inc. P.O. Box 8941 Moscow, Idaho, 83843	208-882-3860
Analytical System			
GC-FID	Mr. Vince Thompson	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-77 Research Triangle Park, N.C. 27711	919-541-3791
GC-FID-ECD	Dr. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Ms. Karen D. Oliver	Northrop Services, Inc. Environmental Sciences P.O. Box 12313 Research Triangle Park, N.C. 27709	919-549-0611
GC-FID-ECD-PID	Dave-Paul Dayton JoAnn Rice	Radian Corporation P.O. Box 13000 Process Center Research Triangle Park, N.C. 27709	919-481-0212

I

Topic	Contact	Address	Telephone No.
GC-MS-SCAN-SIM	Dr. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Apncy Environmental Monitoring Systems laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Nr. John V. Hawkins	Research Triangle Laboratories, Inc. P.O. Box 12507 Research Triangle Park, N.C. 27709	919-544-5115
Canister Cleaning Certification and VOC Canister Storage Stability	Mr. Vince Thompson	U.S. Environmental Protectton Agency tnvironmental Monitoring Systems laboratory MD-77 Research Triangle Park, H.C. 27711	919-541-3791
	Or. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory HO-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Dave-Paul Oayton JoAnn Rice	Radian Corporatton P.O. Box 13000 Progress Center Research Triangle Park, H.C. 21709	919-481-0212
	Or. R.K.M. Jayanty	Research Trirngle Institute P.O. Box 12194 Research Triangle Park, N.C. 27109	919-541-6000
Cryogenic Sampling Unit	Mr. Lou Ballard Mr. Pete Watson	NuTech Corporation 2806 Check Road Durham, N.C., 21104	919-682-0402
	Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems laboratory MD-44 Research Triangle Park, H.C. 27711	919-541-4680
U.S. EPA Audit Gas Standards	Mr. Bob Lamp	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-77B Research Triangle Park, N.C. 27711	919-541-4531

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TABLE 1. VOLATILE ORGANIC COMPOUND DATA SHEET

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT (°C)	MELTING POINT (°C)	CAS NUMBER
Freon 12 (Dichlorodifluoromethane)	Cl_2CF_2	120.91	-29.8	-158.0	
Methyl chloride (Chloromethane)	CH_3Cl	50.49	-24.2	-97.1	74-87-3
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	$ClCF_2CClF_2$	170.93	4.1	-94.0	
Vinyl chloride (Chloroethylene)	$CH_2=CHCl$	62.50	-13.4	-1538.0	75-01-4
Methyl bromide (Bromomethane)	CH_3Br	94.94	3.6	-93.6	74-83-9
Ethyl chloride (Chloroethane)	CH_3CH_2Cl	66.52	12.3	-136.4	75-00-3
Freon 11 (Trichlorofluoromethane)	CCl_3F	137.38	23.7	-111.0	
Vinylidene chloride (1,1-Dichloroethene)	$C_2H_2Cl_2$	96.95	31.7	-122.5	75-35-4
Dichloromethane (Methylene chloride)	CH_2Cl_2	84.94	39.8	-95.1	75-09-2
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	CF_2ClCCl_2F	187.38	41.7	-36.4	
1,1-Dichloroethane (Ethylidene chloride)	CH_3CHCl_2	98.96	57.3	-97.0	74-34-3
cis-1,2-Dichloroethylene	$CHCl=CHCl$	96.94	60.3	-80.5	
Chloroform (Trichloromethane)	$CHCl_3$	119.38	61.7	-63.5	67-66-3
1,2-Dichloroethane (Ethylene dichloride)	$ClCH_2CH_2Cl$	98.96	83.5	-35.3	107-06-2
Methyl chloroform (1,1,1-Trichloroethane)	CH_3CCl_3	133.41	74.1	-30.4	71-55-6
Benzene (Cyclohexatriene)	C_6H_6	78.12	80.1	5.5	71-43-2
Carbon tetrachloride (Tetrachloromethane)	CCl_4	153.82	76.5	-23.0	56-23-5
1,2-Dichloropropane (Propylene dichloride)	$CH_3CHClCH_2Cl$	112.99	96.1	-100.4	78-87-5
Trichloroethylene (Trichloroethene)	$ClCH=CCl_2$	131.29	87	-13.0	79-01-6
cis-1,3-Dichloropropene (cis-1,3-dichloropropylene)	$CH_3CCl=CHCl$	110.97	76		

TABLE 1. VOLATILE ORGANIC COMPOUND DATA SHEET (cont.)

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT (°C)	MELTING POINT (°C)	CAS NUMBER
trans-1,3-Dichloropropene (cis-1,3-Dichloropropylene)	$ClCH_2CH{=}CHCl$	110.97	112.0		
1,1,2-Trichloroethane (Vinyl trichloride)	$CH_2ClCHCl_2$	133.41	113.8	-36.5	79-00-5
Toluene (Methyl benzene)	$C_6H_5CH_3$	92.15	110.6	-95.0	108-88-3
1,2-Dibromoethane (Ethylene dibromide)	$BrCH_2CH_2Br$	187.88	131.3	9.8	106-93-4
Tetrachloroethylene (Perchloroethylene)	$Cl_2C{=}CCl_2$	165.83	121.1	-19.0	127-18-4
Chlorobenzene (Phenyl chloride)	C_6H_5Cl	112.56	132.0	-45.6	108-90-7
Ethylbenzene	$C_6H_5C_2H_5$	106.17	136.2	-95.0	100-41-4
m-Xylene (1,3-Dimethylbenzene)	$1,3\text{-}(CH_3)_2C_6H_4$	106.17	139.1	-47.9	
p-Xylene (1,4-Dimethylxylene)	$1,4\text{-}(CH_3)_2C_6H_4$	106.17	138.3	13.3	
Styrene (Vinyl benzene)	$C_6H_5CH{=}CH_2$	104.16	145.2	-30.6	100-42-5
1,1,2,2-Tetrachloroethane	$CHCl_2CHCl_2$	167.85	146.2	-36.0	79-34-5
o-Xylene (1,2-Dimethylbenzene)	$1,2\text{-}(CH_3)_2C_6H_4$	106.17	144.4	-25.2	
1,3,5-Trimethylbenzene (Mesitylene)	$1,3,5\text{-}(CH_3)_3C_6H_6$	120.20	164.7	-44.7	108-67-8
1,2,4-Trimethylbenzene (Pseudocumene)	$1,2,4\text{-}(CH_3)_3C_6H_6$	120.20	169.3	-43.8	95-63-6
m-Dichlorobenzene (1,3-Dichlorobenzene)	$1,3\text{-}Cl_2C_6H_4$	147.01	173.0	-24.7	541-73-1
Benzyl chloride (α-Chlorotoluene)	$C_6H_5CH_2Cl$	126.59	179.3	-39.0	100-44-7
o-Dichlorobenzene (1,2-Dichlorobenzene)	$1,2\text{-}Cl_2C_6H_4$	147.01	180.5	-17.0	95-50-1
p-Dichlorobenzene (1,4-Dichlorobenzene)	$1,4\text{-}Cl_2C_6H_4$	147.01	174.0	53.1	106-46-7
1,2,4-Trichlorobenzene	$1,2,4\text{-}Cl_3C_6H_3$	181.45	213.5	17.0	120-82-1
Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro-1,3-butadiene)					

TABLE 2. ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM

Compound	Ion/Abundance (amu/% base peak)	Expected Retention Time (min)
Freon 12 (Dichlorodifluoratthane)	85/100 87/ 31	5.01
Methyl chloride (Chloromethane)	50/100 52/ 34	5.69
Freon 114 (1,2-Dichloro-1,1,2,2-tettafluorocthanc)	85/100 135/ 56 87/ 33	6.55
Vinyl chloride (Chloroethtnc)	62/100 27/125 64/ 32	6.71
Methyl bromide (Bromomethane)	94/100 96/ 85	7.83
Ethyl chloride (Chloroethane)	64/100 29/140 27/140	8.4 3
Freon 11 (Trichlorofluoromethane)	101/100 103/ 67	9.97
Vinylidene chloride (1,1-Dichloroethylene)	61/100 961 S5 63/ 31	10.93
Dichloromethane (Methylene chloride)	49/100 84/ 65 86/ 45	11.21
Freon 113 (1,1,2-Trichloro-1,2,2-trifluorocthane)	151/100 101/140 103/ 90	11.60
1,1-Dichlotoethane (Ethylidene dichloride)	631100 27/ 64 651 33	12.50
cis-1,2-Dichloroethylene	61/100 961 60 98/ 44	13.40
Chloroform (Trichloromethane)	83/100 aS: 65 47/ 35	13.75
1,2-Dichloroethane (Ethylene dichloride)	62/100 27/ 70 64/ 31	14.39
Methyl chloroform (1.1,1-Trichloroethane)	97/100 99/ 64 61/ 61	14.62
Benzene (Cyclohexatriene)	781100 771 25 50/ 35	15.04
Carbon tttrachloride (Tetrachloromethane)	117/100 119/ 97	15.18

(continued

TABLE 2. ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM (cont.)

Compound	Ion/Abundance (amu/% base peak)	Estimated Retent Tiny (min)
1,2-Dichloropropne (Propylene dichloride)	631100 41/ 90 62/ 70	15.83
Trichloroethylene (TrIchloroethene)	130/100 132/ 92 95/ 87	16.10
cis-1,3-Dichloropropene	75/100 39/ 70 77/ 30	16.96
trans-1,3-Dichloropropene (1.3 dichloro-1-propene)	75/100 39/ 70 77/ 30	17.49
1,1,2-Trichloroethane (Vinyl trichloride)	97/100 83/ 90 61/ 82	17.61
Toluene (Methyl benzene)	91/100 921 S7	I7.66
1,2-Dibromoethane (Ethylene dibromide)	107/100 109/ 96 27/115	18.48
Tetrachloroethylene (Perchloroethylene)	166/100 164/ 74 1311 60	19.01
Chlorobenzene (Benzene chloride)	1121100 77/ 62 114/ 32	19.73
Ethylbenzene	911100 106/ 23	20.20
m,p-Xylene(1,3/1,4-dimethylbenzene)	91/100 106/ 40	20.4 1
Styrene (Vinyl benzene)	104/100 78/ 60 103/ 49	20.81
1,1,2,2-Tetrachloroethane (Tctrrchloroethane)	83/100 85/ 64	20.92
o-Xylene (1,2-Dimethylbenzene)	91/100 106/ 40	20.92
4-Ethyltoluene	105/100 120/ 29	22.53
1.3,5-Trimethylbenzene (Mes1tylene)	105/100 120/ 42	22.65
1,2,4-Trimethylbenzene (Pseudocumene)	105/100 120/ 42	23.18
m-Dichlorobenzene (1,3-Dichlorobenzene)	146/100 148/ 65 111/ 40	23.31

(continued)

TABLE 2. ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM (cont.)

Compound	Ion/Abundance (amu/% base peak)	Expected Retention Time (min)
Benzyl chloride (a-Chlorotoluene)	911100 126/ 26	23.32
p-Dichlorobenzene (1,4-Dichlorobenzene)	146/100 1481 65 111/ 40	23.41
o-Dichlorobenzene (1,2-Dichlorobenzenc)	146/100 148/ 65 111/ 40	23.88
1,2,4-Trichlorobenzene	180/100 182/ 98 184/ 30	26.71
Hexachlorobutadiene (1,1,2,3,4,4 Hexachloro-1,3-butadiene)	225/100 227/ 66 223/ 60	27.68

TABLE 3. GENERAL GC AND MS OPERATING CONDITIONS

Chromatography	
Column	Hewlett-Packard OV-1 crosslinked methyl silicone (50 m x 0.31-mm I.D. 17 um film thickness), or equivalent
Carrier Gas	Helium (2.0 cm^3/min at 250°C)
Injection Volume	Constant (1-3 uL)
Injection Mode	Splitless
Temperature Program	
Initial Column Temperature	-50°C
Initial Hold Time	2 min
Program	8°C/min to 150°C
Final Hold Time	15 min
Mass Spectrometer	
Mass Range	18 to 250 amu
Scan Time	1 sec/scan
EI Condition	70 eV
Mass Scan	Follow manufacturer's Instruction for selective mass selective detector (MS) and selected ion monitoring (SIM) mode
Detector Mode	Multiple ion detection
FID System (Optional)	
Hydrogen Flow	30 cm^3/minute
Carrier Flow	30 cm^3/minute
Burner Air	400 cm^3/minute

TABLE 4. 4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 602 of mast 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of matt 174
176	>95% but< 101% of mass 174
177	5 to 9% of mass 176

TABLE 5. RESPONSE FACTORS (ppbv/area count) AND EXPECTED RETENTION TIME FOR GC-MS-SIM ANALYTICAL CONFIGURATION

Compounds	Response Factor (ppbv/area count)	Expected Retention Time (minutes)
Freon 12	0.6705	5.01
Methyl chloride	4.093	5.64
Freon 114	0.4928	6.55
Vinyl chloride	2.343	6.71
Methyl bromide	2,647	7.83
Ethyl chloride	2,954	8.43
Freon 11	0.5145	9.87
Vinylidene chloride	1.037	10.93
Dichloromethane	2.255	11.21
Trichlorotrifluorocthrnc	0.9031	11.60
1,1-Dichloroethane	1.273	12.50
cis-1,2-Dichloroethylene	1.363	13.40
Chloroform	0.7911	13.75
1,2-Dichloroethane	1,017	14.39
Methyl chloroform	0.7078	14.62
Benzene	1,236	15.04
Carbon tetrachloride	0.5880	15.18
1,2-Dichloropropane	2.400	15.83
Trichloroethylene	1.383	16.10
cis-1,3-Dichloropropene	1.877	16.96
trans-1,3-Dichloropropene	1.338	17.49
1,1,2-Trichloroethane	1.891	17.61
Toluene	0.9406	17.86
1,2-Dibromoethane (EDB)	0,8662	18.48
Tetrachloroethylene	0.7357	19.01
Chlorobenzene	0.8558	19.73
Ethyl benzene	0.6243	20.20
m,p-Xylene	0.7367	20.41
Styrene	1.888	20.80
1,1,2,2-Tetrachloroethane	1.035	20.92
o-Xylene	0.7498	20.92
4-Ethyltoluene	0,6181	22.53
1,3,5-Trimethylbenzene	0.7088	22.65
1,2,4-Trimethylbenzene	0.7536	23.18
m-Dichlorobenzene	0.9643	23.31
Benzyl chloride	1,420	23.32
p-Dichlorobenzene	0,8912	23.41
o-Dichloroknrenc	1.004	23.88
1,2,4-Trichlorobenzene	2.150	26.71
Hexachlorobutadiene	0.4117	27.68

TABLE 6. GC-MS-SIM CALIBRATION TABLE

*** External Standard ***

Operator: JDF 8 Jan 87 10:02 am
Sample Info : SYR 1
Misc Info:
Integration File Name : DATA:SYR2A02A.I
Sequence Index: 1 Bottle Number : 2

Last Update: 8 Jan 87 8:13 am
Reference Peak Window: 5.00 Absolute Minutes
Non-Reference Peak Window: 0.40 Absolute Minutes
Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.667

Peak Num	Type	Int Type	Ret Time	Signal Description			Compound Name	Area	Amount
1		1	FF	5.020	Mass	85.00	amu FREON 12	12893	4011 ppbv
2		1	PP	5.654	Mass	50.00	amu METHYLCHLORI	4445	2586 ppbv
3		1	BF	6.525	Mass	85.00	amu FREON 114	7067	1215 ppbv
4		1	PB	6.650	Mass	62.00	amu VINYLCHLORID	2892	1929 ppbv
5		1	BF	7.818	Mass	94.00	amu METHYLBROMID	2401	1729 ppbv
6		1	BB	8.421	Mass	64.00	amu ETHYLCHLORID	2134	2769 ppbv
7		1	BV	9.940	Mass	101.00	amu FREON 11	25069	6460 ppbv
8		1	BP	10.869	Mass	61.00	amu VINDENECHLOR	5034	1700 ppbv
9		1	BF	11.187	Mass	49.00	amu DICHLOROMETH	4803	2348 ppbv
10		1	PP	11.225	Mass	41.00	amu ALLYLCHLORID	761	8247 ppbv
11		1	BF	11.578	Mass	151.00	amu 3CHL3FLUETHA	5477	1672 ppbv
12		1	BF	12.492	Mass	63.00	amu 1,1DICHLOETH	5022	1738 ppbv
13		1	VP	13.394	Mass	61.00	amu c-1,2DICHLET	4761	1570 ppbv
14		1	PH	13.713	Mass	83.00	amu CHLOROFORM	5327	1678 ppbv
15		1	BF	14.578	Mass	62.00	amu 1,2DICHLETHA	5009	2263 ppbv
16		1	FB	14.594	Mass	97.00	amu METHCHLOROFO	6656	2334 ppbv
17		1	VF	15.009	Mass	78.00	amu BENZENE	8352	2167 ppbv
18		1	VF	15.154	Mass	117.00	amu CARBONTETRAC	5888	1915 ppbv
19		1	BB	15.821	Mass	63.00	amu 1,2DICHLPROP	3263	1799 ppbv
20		1	BB	16.067	Mass	130.00	amu TRICHLETHENE	4386	2109 ppbv
21		1	FB	16.941	Mass	75.00	amu c-1,3DICHLPR	2228	957.2 ppbv
22		1	BP	17.475	Mass	75.00	amu t-1,3DICHLPR	1626	689.2 ppbv
23		1	BB	17.594	Mass	97.00	amu 1,1,2CHLETHA	2721	1772 ppbv
24		1	BV	17.844	Mass	91.00	amu TOLUENE	14417	2735 ppbv
25		1	PB	18.463	Mass	107.00	amu EDB	4070	1363 ppbv
26		1	PH	18.989	Mass	166.00	amu TETRACHLETHE	6674	2065 ppbv
27		1	FB	19.705	Mass	112.00	amu CHLOROBENZEN	5648	1524 ppbv
28		1	BF	20.168	Mass	91.00	amu ETHYLBENZENE	11084	1842 ppbv
29		1	FB	20.372	Mass	91.00	amu m,p-XYLENE	17989	3790 ppbv
30		1	BV	20.778	Mass	104.00	amu STYRENE	3145	1695 ppbv
31		1	BH	20.887	Mass	83.00	amu TETRACHLETHA	4531	1376 ppbv
32		1	BF	20.892	Mass	91.00	amu o-XYLENE	9798	2010 ppbv
33		1	VV	22.488	Mass	105.00	amu 4-ETHYLTOLUE	7694	1481 ppbv
34		1	VB	22.609	Mass	105.00	amu 1,3,5METHBEN	6781	1705 ppbv
35		1	BB	22.144	Mass	105.00	amu 1,2,4METHBEN	7892	2095 ppbv
36		1	BV	23.275	Mass	146.00	amu m-DICHLBENZE	3046	1119 ppbv
37		1	VV	23.279	Mass	91.00	amu BENZYLCHLORI	3880	1006 ppbv
38		1	VB	23.378	Mass	146.00	amu p-DICHLBENZE	6090	2164 ppbv
39		1	BP	23.850	Mass	146.00	amu o-DICHLBENZE	2896	1249 ppbv
40		1	BB	26.675	Mass	180.00	amu 1,2,4CHLBENZ	562	767.1 ppbv
41		1	BB	27.657	Mass	225.00	amu HEXACHLBUTAD	6309	1789 ppbv

TABLE 7. TYPICAL RETENTION TIME (MIN) AND CALIBRATION RESPONSE FACTORS (ppbv/area count) FOR TARGETED VOCs ASSOCIATED WITH FIC AND.ECD ANMKICK SYSTEM

Peak Number[1]	Canpound	Retentton Time (RT), minutes	FID Response Factor (RF) (ppbv/area count)	ECD Response Factor ppbv/ire8 ount x 10^{-5})
1	Freon 12	3.65	3.465	13.89
2	Methyl chloride	4.30	0.693	
3	Freon 114	5.13	0.578	22.32
4	Vinyl chloride	3.28	0.406	
5	Methyl branide	6.44		26.34
6	Ethyl chlorldc	7.06	0.413	
7	Freon 11	8.60	6.367	1.367
8	Vinylidene chloride	9.51	0.347	
9	Dlchloromethane	9e84	0.903	
10	Trichlorotrifluoroethane	10.22	0.374	3.955
11	1,1-Dichloroethane	11.10	0.359	
12	cis-1,2-Dichloroethylene	11.99	0.368	
13	Chloroform	12.30	1.059	11.14
14	1,2-Dichloroethane	12.92	0.409	
15	Methyl chloroform	13.12	0.325	3.258
16	Benzene	13.51	0.117	
17	Carbon tetrachloride	13.54	1.451	1.077
18	1,2-Dichloropropane	14.26	0.214	
19	Trichlorocthylene	14.50	0.327	8.910
20	cis-1,3-Dichloropropene	15.31		
21	trans-1,3-Dichloropropene	15.83		
22	1,1,2-Trichloroethane	15a93	0.336	
23	Toluene	16.17	0.092	
24	1,2-Dibromoethane (EDB)	16.78	0.366	5.137
25	Tetrachloroethylene	17.31	0.324	1.449
26	Chlorokrzene	18.03	0.120	
27	Ethylbenzene	10.51	0.092	
28	m,p-Xylene	18.72	0.095	
29	Styrene	19.12	0.143	
30	1,1,2,2-Tetrachloroethane	19020		9.856
31	o-Xylene	19.23		
32	4-Ethyltoluene	20.82	0.100	
33	1,3,5-Trimethylbenzene	0.94	0.109	
34	1,2,4-Trimethylbenzene	1.46	0.111	
35	m-Dichlorobenzene	21.50		
36	Benzyl chloride	21.56		
37	p-Dichlorobenzene	21.67	0.188	
38	o-Dichlorokrzene	22.12	0.188	
39	1,2,4-Trichlorobenzene	24.80	0.667	
40	Hexachlorobutadiene	2s.82	0.305	1.055

[1] Refer to Figures 15 and 16 for peak location

TABLE 8. TYPICAL RETENTION TIME (minuter) FOR SELECTED ORGANICS USING GC-FID-ECD-PID* ANALYTICAL SYSTEM

Compound	Retention Time (minutes)		
	FID	ECD	PID
Acetylene	2.984	--	--
1,3-Butaditnt	3.599	--	3.594
Vinyl chloride	3.790	--	3.781
Chloromethane	5.137	--	--
Chloroethanc	5.738	--	--
Bromoethane	8.154	--	--
Methylene Chloride	9.232	--	9.210
trans-1,2-Dichloroethylene	10.077	--	10.065
1,1-Dichloroethane	11.190	--	--
Chloroprtne	11.502	--	11.491
Perfluorobenzene	13.077	13.078	13.069
Bromochloromethanc	13.397	13.396	13.403
Chloroform	13.768	13.767	13.771
1,1,1-Trichloroethane	14.151	14.153	14.158
Carbon fctrachloride	14.642	14.667	14.686
Benzene/1,2-Dichlorocthane	15.128	--	15.114
Perfluorotoluene	15.420	15.425	154 12
Trichloroethylene	17.022	17.024	17.014
1,2-Dichloropropene	17.491	17.805	17.522
Bromodichloranethane	18.369	--	--
trans-1,3-Dichloropropylene	19.694	19.693	19.688
Toluene	20,658	--	20.653
cis-1,3-Dichloropropylcne	21.461	21.357	21.357
1,1,2-Trichloroethane	21.823	--	--
Tetrachlofoethylene	22.340	22.346	22.335
Dibromochloromethane	22.955	22.959	22,952
Chlorobenzene	24.866	--	24.861
m/p-Xylene	25.763	--	25.757
Styrentlo-Xylene	27.036	--	27.030
Bromofluorobenzene	28.665	28.663	28.660
1,1,2,2-Tetrachloroethane	29.225	29.227	29.228
m-Dichlorobenzene	32.347	32.345	32.342
p-Dichlorobenzene	32.673	32.669	32.666
o-Dichlorobenzene	33.885	33.883	33.880

* Varian® 3700 GC equipped with J & W Megabore® DB 624 Capillary Column (30 m X 0.53 I.D. mm) using helium carrier gas.

TABLE 9. GC-MS-SIM CALIBRATION TABLE

Last Update: 18 Dec 86 7:54 am
Reference Peak Window: 5.00 Absolute Minutes
Non-Reference Peak Window: 0.40 Absolute Minutes
Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.000

Rot Time	Pk#	Signal	Descr	Amt ppbv	Lvl	[Area]	Pk-Type	Partial Name
5.008	1	Mass	85.00 amu	13620	1	72974	1	FREON 12
5.690	2	Mass	50.00 amu	12720	1	36447	1	METHYLCHLORID
6.222	3	Mass	85.00 amu	8380	1	81231	1	FREON 114
6.709	4	Mass	62.00 amu	0010	1	20118	1	VINYLCHLORIDE
7.831	5	Mass	94.00 amu	12210	1	28265	1	METHYLBROMIDE
8.431	6	Mass	64.00 amu	12374	1	16149	1	ETHYLCHLORIDE
9.970	7	Mass	101.00 amu	12380	1	80088	1	FREON 11
10.927	8	Mass	61.00 amu	7890	1	38954	1	VINDENECHLORI
11.209	9	Mass	49.00 amu	12760	1	43507	1	DICHLOROMETHA
11.331	10	Mass	41.00 amu	12650	1	1945	1	ALLYLCHLORIDE
11.595	11	Mass	151.00 amu	7420	1	40530	1	3CHL3FLUETHAN
12.502	12	Mass	63.00 amu	12710	1	41595	1	1,1DICHLOETHA
13.403	13	Mass	61.00 amu	12630	1	50900	1	c-1,2DICHLETH
13.747	14	Mass	83.00 amu	7470	1	40585	1	CHLOROFORM
14.387	15	Mass	62.00 amu	9040	1	35556	1	1,2DICHLETHAN
14.622	16	Mass	97.00 amu	8100	1	38503	1	METHCHLOROFOR
15.038	17	Mass	78.00 amu	10760	1	69119	1	BENZENE
15.183	18	Mass	117.00 amu	8340	1	42737	1	CARBONTETRA
15.829	19	Mass	63.00 amu	12780	1	38875	1	1,2DICHLPRO
16.096	20	Mass	130.00 amu	8750	1	30331	1	TRICHLETHENE
16.956	21	Mass	75.00 amu	4540	1.17078		1	c-1,3DICHLPRO
17.492	22	Mass	75.00 amu	3380	1	12294	1	t-1,3DICHLPRO
17.610	23	Mass	97.00 amu	12690	1	32480	1	1,1,2CHLETHAN
17.562	24	Mass	91.00 amu	10010	1	86036	1	TOLUENE
18.465	25	Mass	107.00 amu	6710	1	33350	1	EDB
19.012	26	Mass	166.00 amu	7830	1	43454	1	TETRACHLETHEN
19.729	27	Mass	112.00 amu	7160	1	44224	1	CHLOROBENZENE
20.19:	28	Mass	91.00 amu	12740	1	127767	1	ETHYLBENZENE
20.407	29	Mass	91.00 amu	25400	1	100973	1	m,p-XYLENE
20.806	30	Mass	104.00 amu	12390	1	38332	1	STYRENE
20.916	31	Mass	83.00 amu	11690	1	64162	1	TETRACHLETHAN
20.921	32	Mass	91.00 amu	11085	1	90096	1	o-XYLENE
22.228	33	Mass	105.00 amu		1	106747	1	4-ETHYLTOLUEN
22.648	34	Mass	105.00 amu	12620	1	83666	1	1,3,5METHBENZ
23.179	35	Mass	105.00 amu	12710	1	19833	1	1,2,4METHBENZ
23.307	36	Mass	146.00 amu	12650	1	57409	1	m-DICHLBENZEN
23.317	37	Mass	91.00 amu	7900	1	10774	1	BENZYLCHLORID
23.413	38	Mass	146.00 amu	12390	1	58127	1	p-DICHLBENZEN
23.885	39	Mass	146.00 amu	13510	1	12233	1	o-DICHLBENZEN
26.714	40	Mass	180.00 amu	12520	1	18967	1	1,2,4CHLBENZE
27.680	41	Mass	225.00 amu	7470	1	43920	1	HEXACHLBUTADI

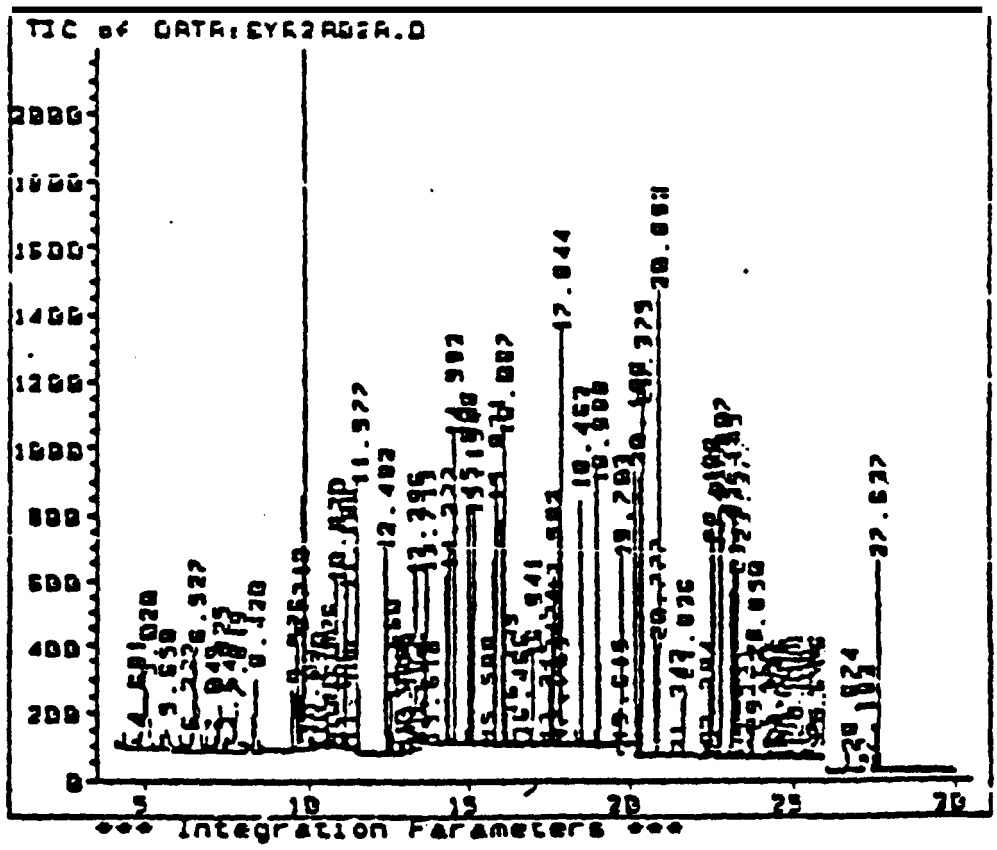
TABLE 10. EXAMPLE OF HARD-COPY OF GC-MS-SIM ANALYSIS

Data file: DATA:SYR2A02A.D
File type: GC / MS DATA FILE

Name Info: SYR 1
Misc Info:
Operator : JDF

Date : 8 Jan 87 10102 am
Instrment: MS_5970
Inlet : GC

Sequence index : 1
Als bottle num : 2
Replicate num : 1



FALSE t Shoulder Detection Enabled
0.020 : Expected Peak Width (Min)
11 8 Initial Peak Detection Threshold

4.000 THRESHOLD 5.000
4.000 PEAK_WIDTH 0.200
9.800 PEAK_WIDTH 0.060

TABLE 10. EXAMPLE OF HARD-COPY OF GC-MS-SIM ANALYSIS (cont.)

Operator: JDP 8 Jan 87 10:02 am
Sample Info : SYR 1
Misc Info:
Integration File Name : DATA:SYR2A02A.I
Sequence Index: 1 Bottle Number : 2

Last Update: 8 Jan 87 8:13 am
Reference Peak Window: 0.00 Absolute Minutes
Non-Reference Peak Window: 0.40 Absolute Minutes
Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.667

Fmal. Num.	Type	Int Type	Ret Time	Signal Description			Compound Name	Area	Amount
1		1	PP	5.020	Mass	85.00	amu FREON 12	12893	4011 pptv
2		1	PP	5.654	Mass	50.00	amu METHYLCHLORI	4445	2286 pptv
3		1	BP	6.322	Mass	85.00	amu FREON 114	7067	1215 pptv
4		1	PB	6.650	Mass	62.00	amu VINYLCHLORID	2892	1929 pptv
5		1	BP	7.815	Mass	94.00	amu METHYLBROMID	2401	1729 pptv
6		1	BP	8.421	Mass	64.00	amu ETHYLCHLORID	2134	2769 pptv
7		1	[illegible]	[illegible].940	Mass	101.00	amu FREON 11	22069	6460 pptv
8		1	BP	10.689	Mass	61.00	amu VINDENECHLOR	5034	1700 pptv
9		1	BP	11.167	Mass	49.00	amu DICHLOROMETH	4802	2342 pptv
10		1	PP	11.222	Mass	41.00	amu ALLYLCHLORID	761	8247 pptv
11		1	BP	11.578	Mass	151.00	amu 3CHL3FLUETHA	5477	1672 pptv
12		1	BP	12.492	Mass	63.00	amu 1,1DICHLOETH	5022	1738 pptv
13		1	VP	13.394	Mass	61.00	amu c-1,2DICHLET	4761	1970 pptv
14		1	PM	13.713	Mass	83.00	amu CHLOROFORM	5327	1678 pptv
15		1	BP	14.378	Mass	62.00	amu 1,2DICHLETHA	5009	2263 pptv
16		1	PB	14.594	Mass	97.00	amu METHCHLOROFO	6656	2224 pptv
17		1	VP	15.009	Mass	78.00	amu BENZENE	8322	2167 pptv
18		1	VP	15.154	Mass	117.00	amu CARBONTETRAC	5888	1915 pptv
19		1	BP	15.821	Mass	63.00	amu 1,2DICHLPROP	3263	1799 pptv
20		1	BP	16.067	Mass	130.00	amu TRICHLETHENE	4386	2109 pptv
21		1	PB	16.941	Mass	75.00	amu c-1,3DICHLPR	2226	967.2 pptv
22		1	BP	17.475	Mass	75.00	amu t-1,3DICHLPR	1626	689.2 pptv
23		1	BP	17.594	Mass	97.00	amu 1,1,2CHLETHA	2721	1772 pptv
24		1	BV	17.844	Mass	91.00	amu TOLUENE	14417	2722 pptv
25		1	PB	18.463	Mass	107.00	amu EDB	4070	1362 pptv
26		1	PM	18.989	Mass	166.00	amu TETRACHLETHE	6674	2062 pptv
27		1	PB	19.702	Mass	112.00	amu CHLOROBENZEN	5648	1524 pptv
28		1	BP	20.168	Mass	91.00	amu ETHYLBENZENE	11084	1842 pptv
29		1	PB	20.372	Mass	91.00	amu m,p-XYLENE	17989	3790 pptv
30		1	BV	20.778	Mass	104.00	amu STYRENE	3142	1692 pptv
31		1	BM	20.887	Mass	83.00	amu TETRACHLETHA	4531	1376 pptv
32		1	BP	20.892	Mass	91.00	amu o-XYLENE	9798	2010 pptv
33		1	VV	22.488	Mass	105.00	amu 4-ETHYLTOLUE	7694	1481 pptv
34		1	VB	22.609	Mass	105.00	amu 1,3,5METHBEN	6781	1705 pptv
35		1	PB	22.144	Mass	105.00	amu 1,2,4METHBEN	7892	2095 pptv
36		1	BV	22.272	Mass	146.00	amu m-DICHLBENZE	3046	1119 pptv
37		1	VV	22.279	Mass	91.00	amu BENZYLCHLORI	3880	1006 pptv
38		1	VB	22.378	Mass	146.00	amu p-DICHLBENZE	6090	2164 pptv
39		1	BP	23.850	Mass	146.00	amu o-DICHLBENZE	2896	1249 pptv
40		1	PB	26.673	Mass	180.00	amu 1,2,4CHLBENZ	562	767.1 pptv
41		1	BP	27.637	Mass	225.00	amu HEXACHLBUTAD	6309	1789 pptv

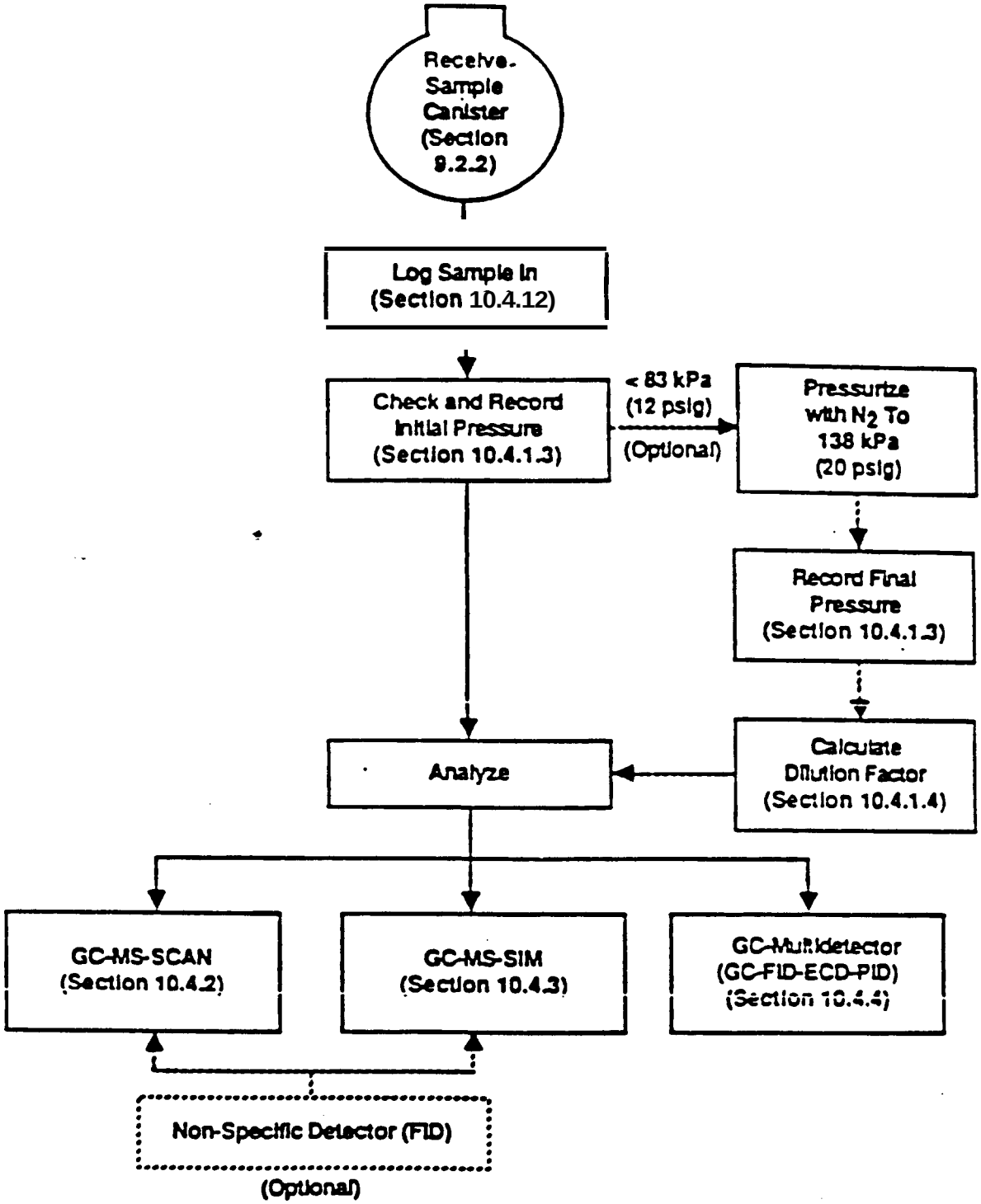


FIGURE 1. ANALYTICAL SYSTEMS AVAILABLE FOR CANISTER VOC IDENTIFICATION AND QUANTITATION

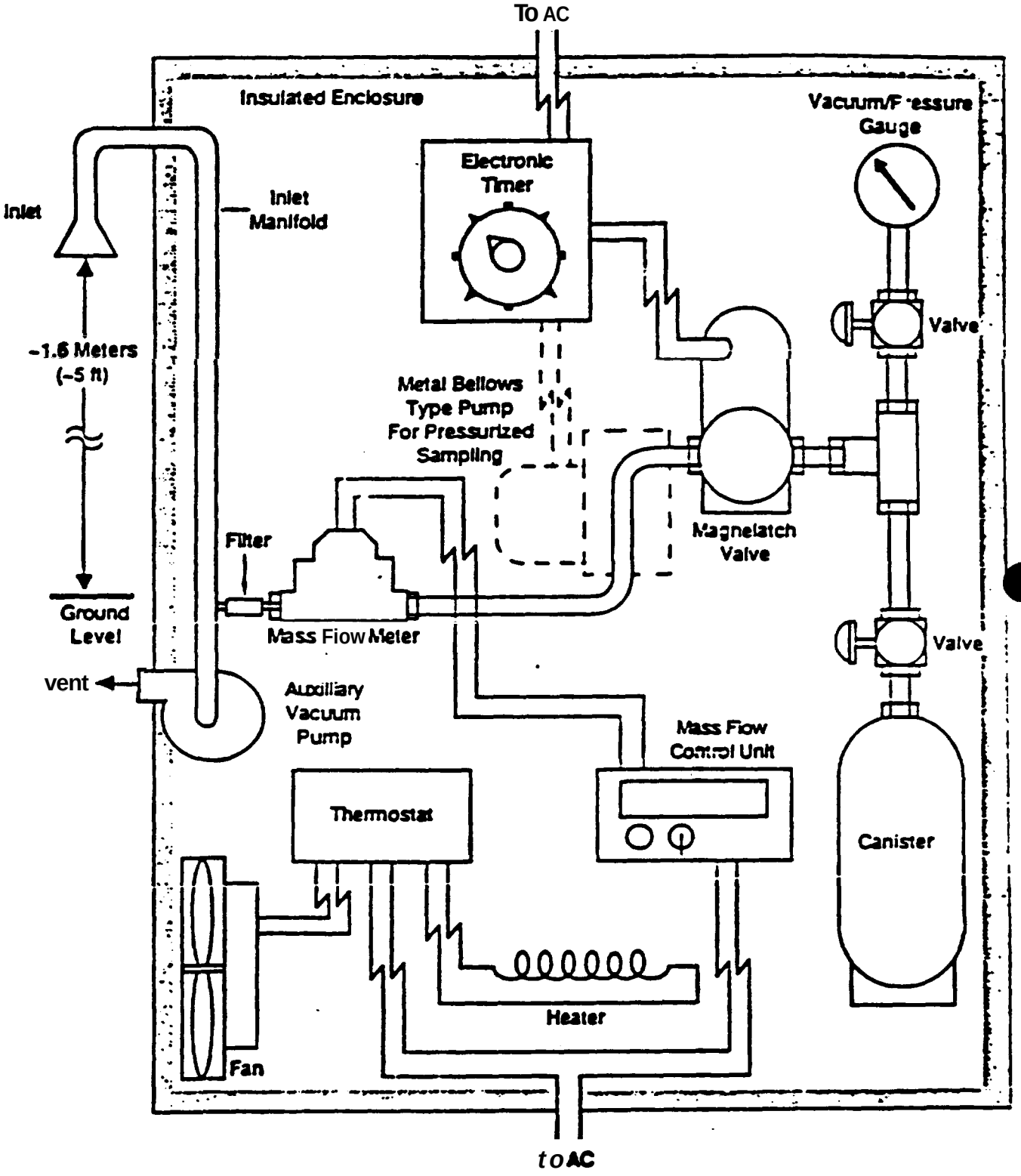


FIGURE 2 SAMPLER CONFIGURATION FOR SUBATMOSPHERIC PRESSURE OR PRESSURIZED CANISTER SAMPLING

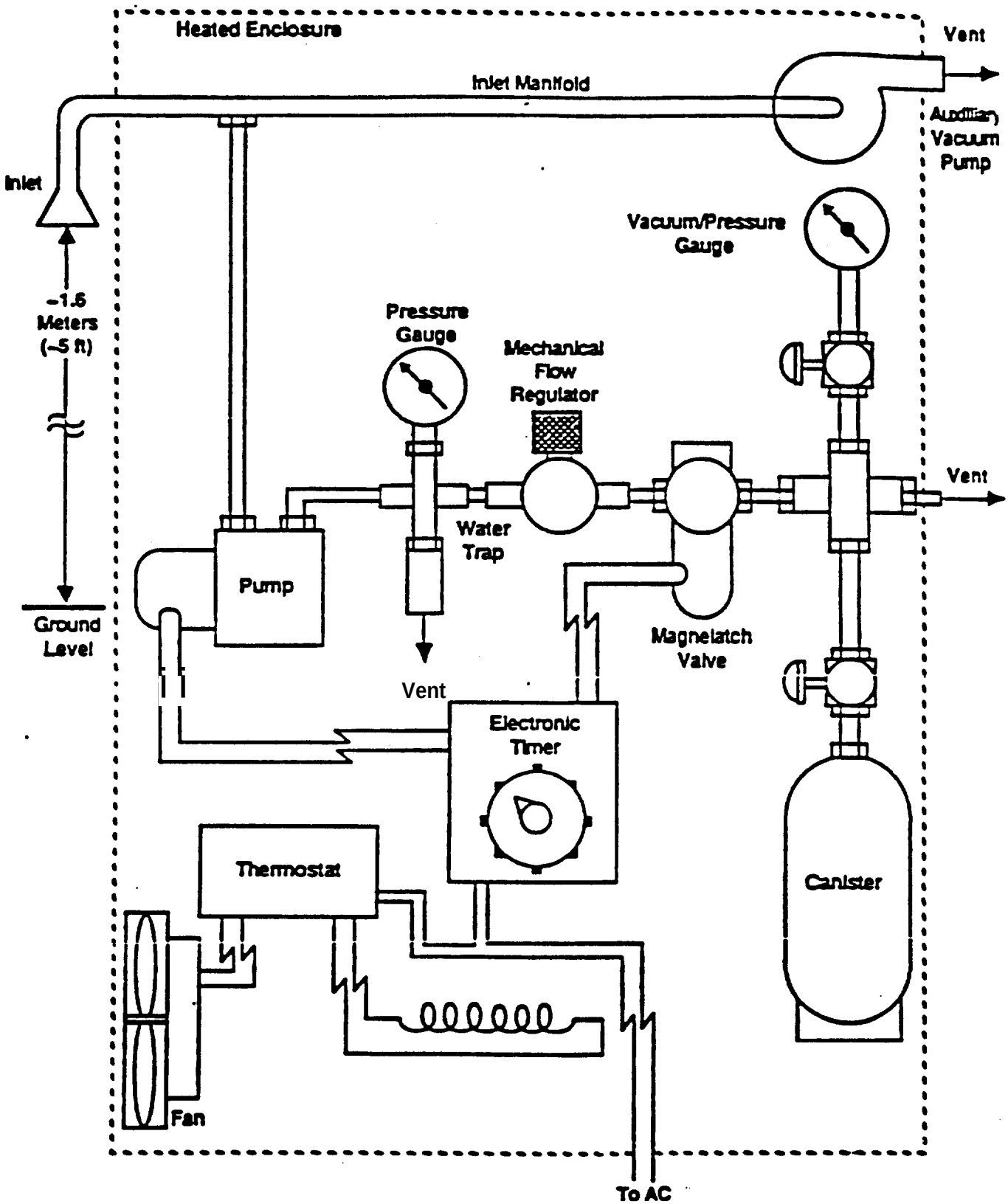


FIGURE 3. ALTERNATIVE SAMPLER CONFIGURATION FOR PRESSURED CANISTER SAMPLING

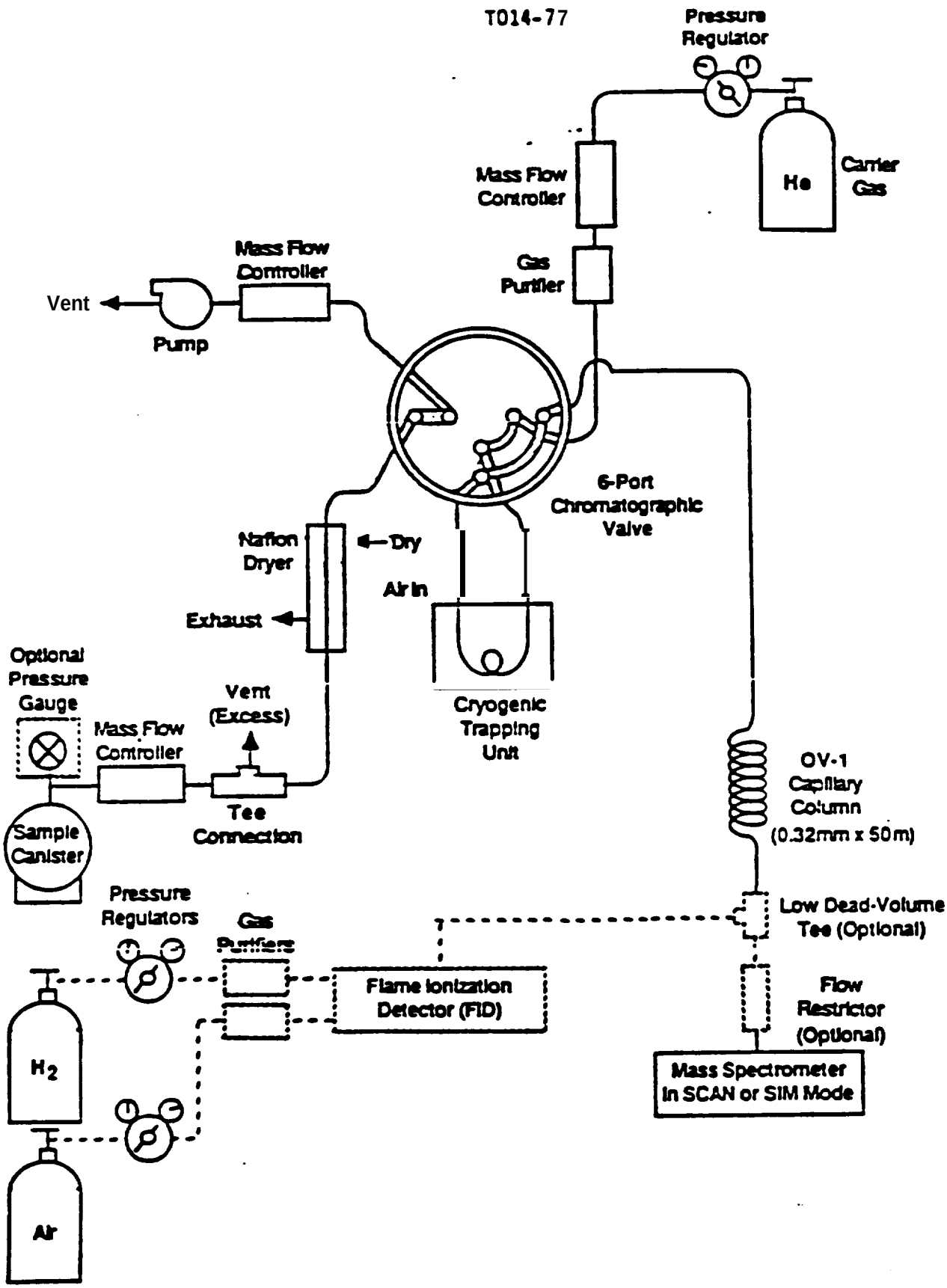
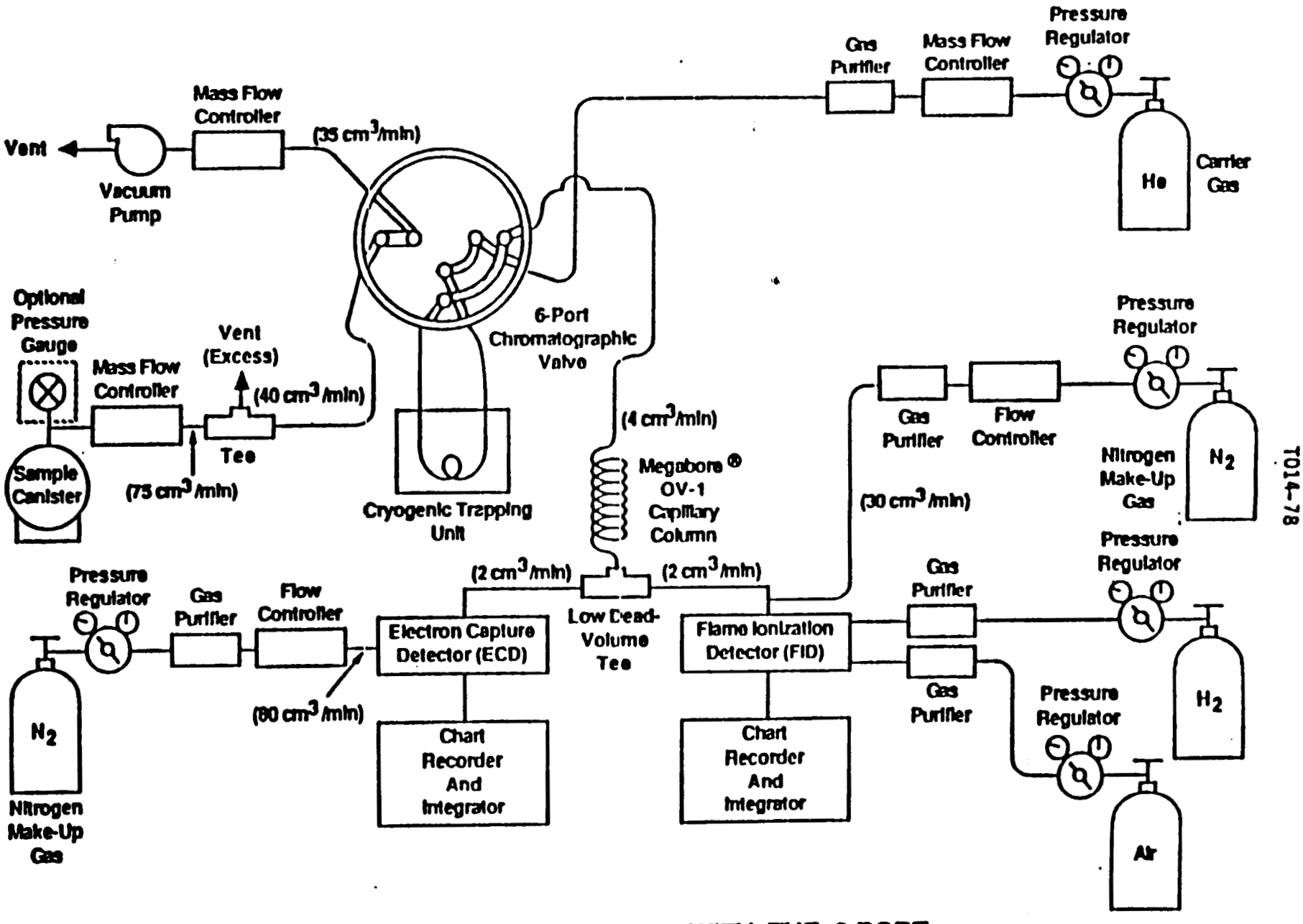
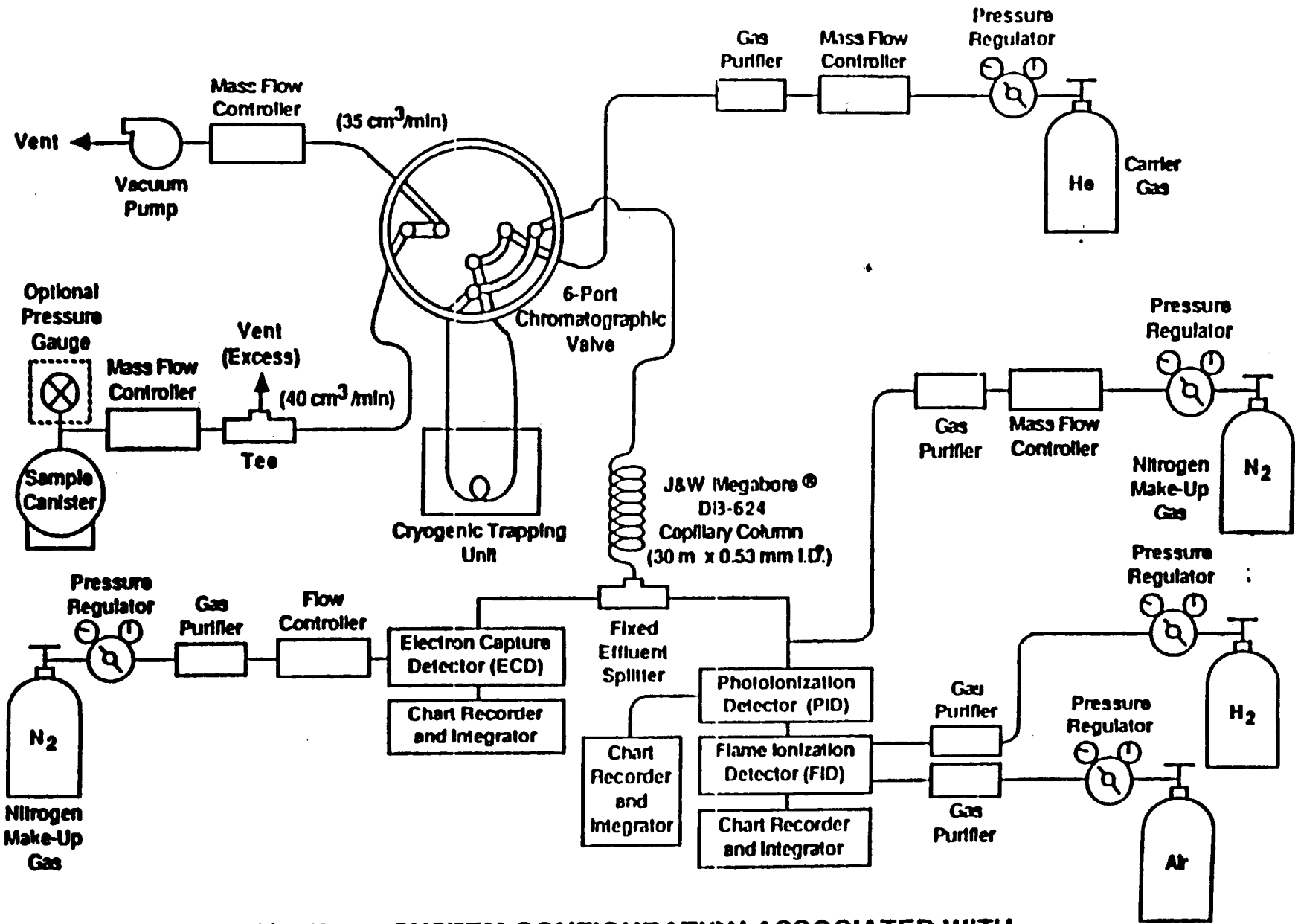


FIGURE 4. CANISTER ANALYSIS UTILIZING GC-MS-SCAN-SIM ANALYTICAL SYSTEM WITH OPTIONAL FLAME IONIZATION DETECTOR WITH THE 6-PORT CHROMATOGRAPHIC VALVE IN THE SAMPLE



'IRE 5 GC-FID-ECD ANALYTICAL SYST' WITH THE 6-PORT CHROMATOGRAPHIC VALVE IN ...E SAMPLE DESORPTION MODE



T014-79

FIGURE 6. SYSTEM CONFIGURATION ASSOCIATED WITH THE GC-FID-ECD-PID AN[illegible]YTICAL SYSTEM WITH THE 6-PORT CHRO[illegible]TOGRAPHIC VALVE IN THE SAMPLE DESOR[illegible]N [illegible]DE

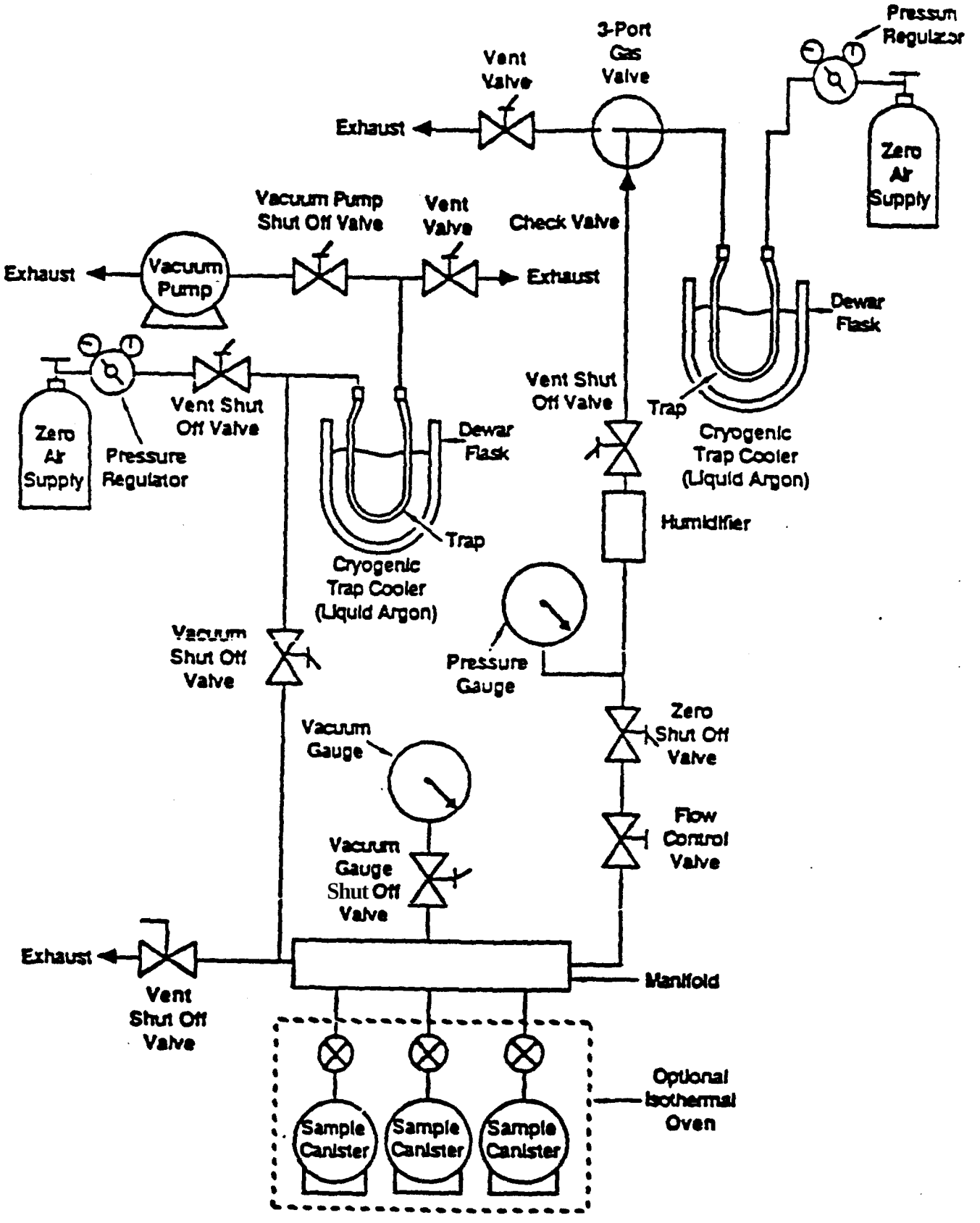
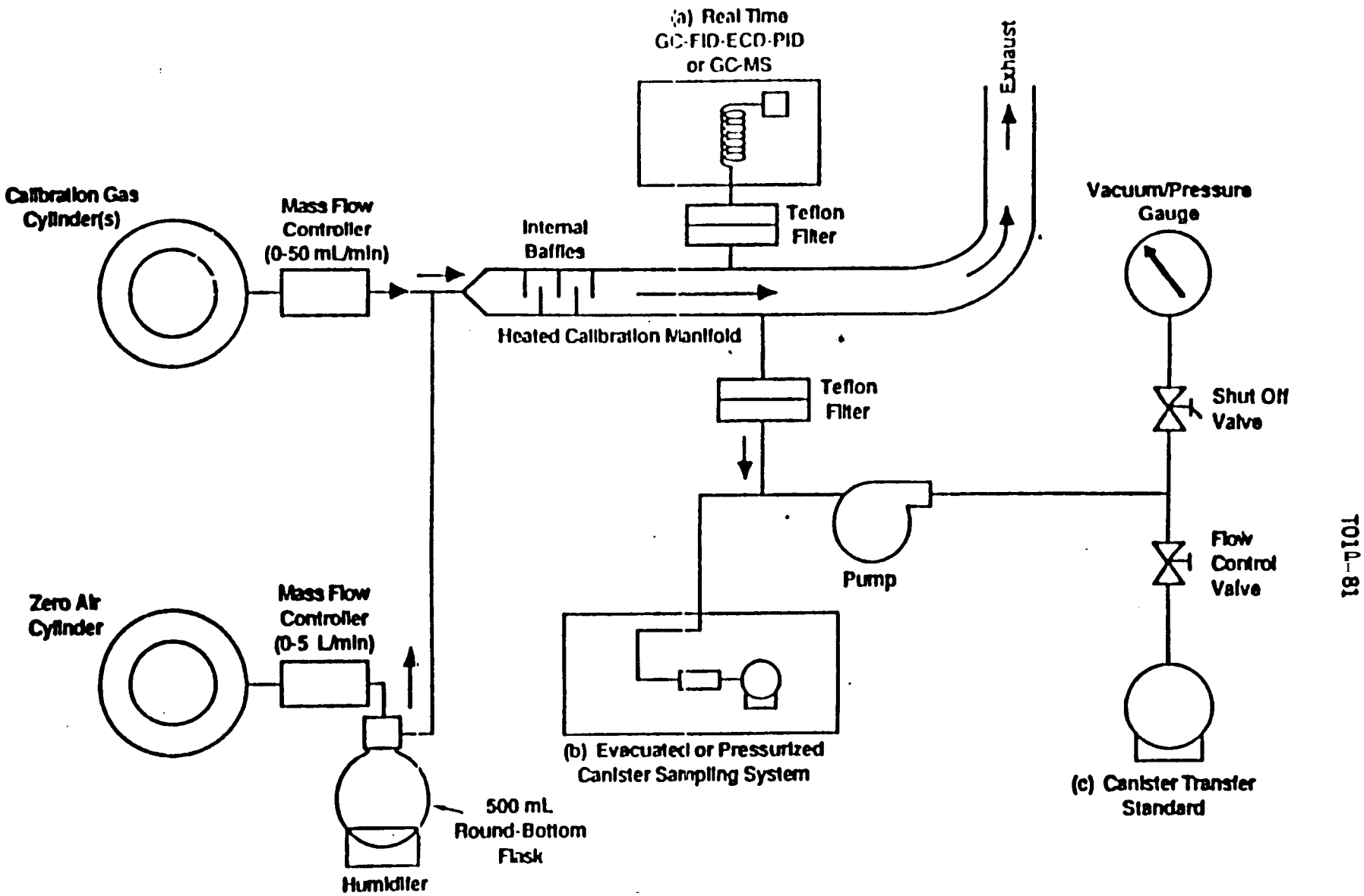
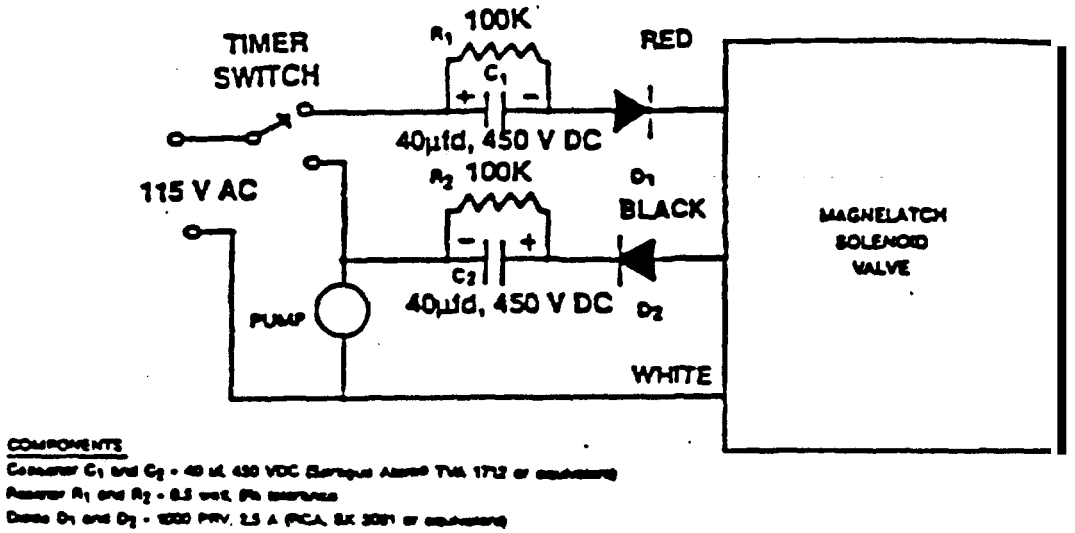


FIGURE 7. CANISTER CLEANING SYSTEM

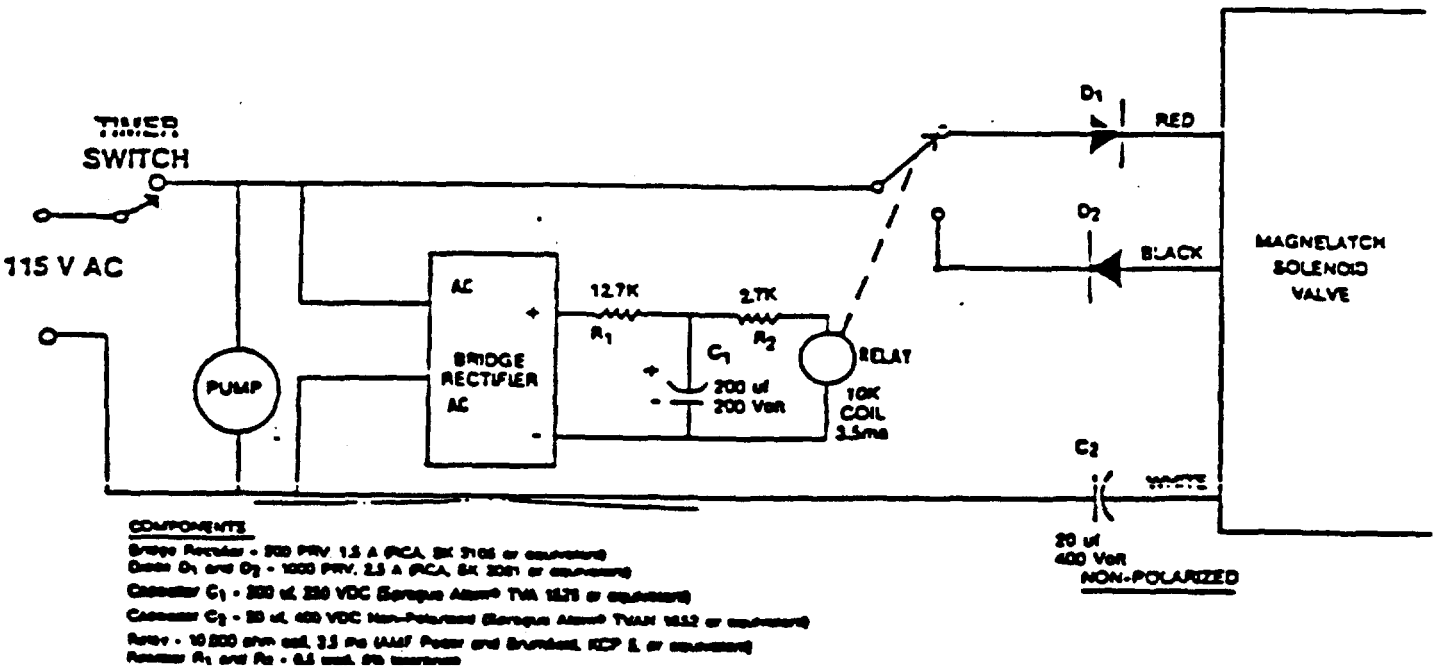


T01D-81

FIGURE 8. SCHEMATIC OF CALIBRATION SYSTEM AND MANIFOLD FOR (a) ANALYTICAL SYSTEM CALIBRATION, (b) TESTING CANISTER SAMPLING SYSTEM AND (c) PREPARING CANISTER TRANSFER STANDARDS.



(a). Simple Circuit For Operating Magnelatch Valve



(b). Improved Circuit Designed To Handle Power Interruptions

FIGURE 9. ELECTRICAL PULSE CIRCUITS FOR DRIVING SKINNER MAGNELATCH SOLENOID VALVE WITH A MECHANICAL TIMER

CANISTER SAMPLING FIELD DATA SHEET

A GENERAL INFORMATION

SITE LOCATION: _____
SITE ADDRESS: _____
_____
_____
SAMPLING DATE _____

SHIPPING DATE: _____
CANISTER SERIAL NO.: _____
SAMPLER ID: _____
OPERATOR: _____
CANISTER LEAK CHECK DA _____

B. SAMPUNG INFORMATION

TEMPERATURE

	INTERIOR	AMBIENT	MAXIMUM	MINIMUM
START				
STOP				

CANISTER PRESSURE	

SAMPLMG TIMES

	LOCAL TIME	ELAPSED TIME METER READING
START		
STOP		

FLOW RATES

MANIFOLD FLOW RATE	CANISTER FLOW RATE	FLOW CONTROLLER READOUT

SAMPLING SYSTEM CERTIFICATION DATE: _____
QUARTERLY RECERTIFICATION DATE: _____

C. LABORATORY INFORMATION

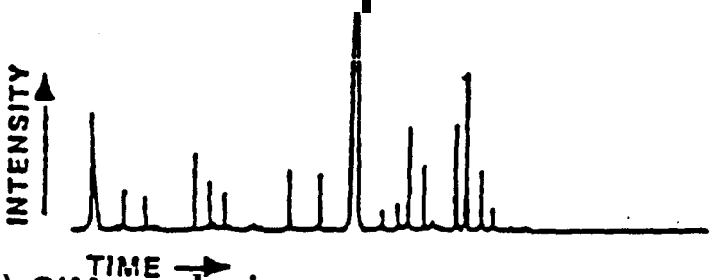
DATE RECEIVED: _____
RECEMD BY: _____
INITIAL PRESSURE _____
FINAL PRESSURE: _____
DILUTION FACTOR: _____
ANALYSIS
GC-FID-ECD DATE: _____
GC-MSD-SCAN DATE: _____
GC-MSD-SIM DATE: _____
RESULTS': _____
_____
GC-FID-ECD: _____
GC-MSD-SCAN: _____
GC-MSD-SIM: _____

_____
SIGNATURE/TITLE

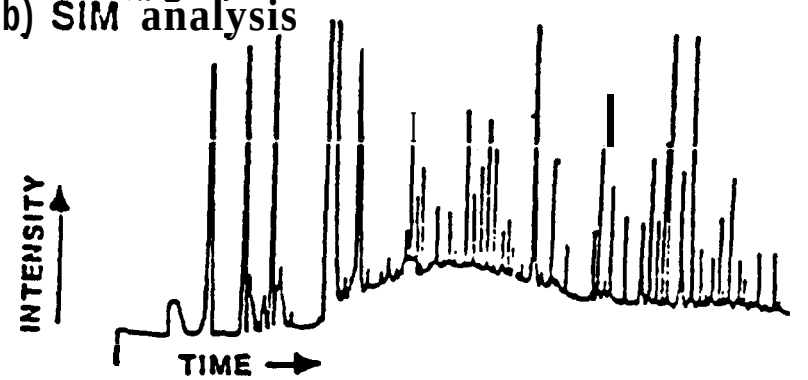
* ATTACH DATA SHEETS

FIGURE 10. CANISTER SAMPLING FIELD DATA SHEET

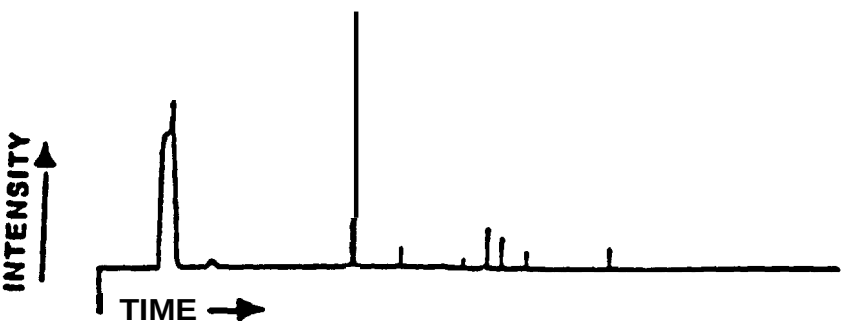
(a) SCAN analysis



(b) SIM analysis



(c) FID analysis



(d) ECD analysis

FIGURE 11. TYPICAL CHROMATOGRAMS OF A VOC SAMPI ANALYZED BY GC-MS-SCAN-SIM MODE AND GC-MULTIDETECTOR MODE

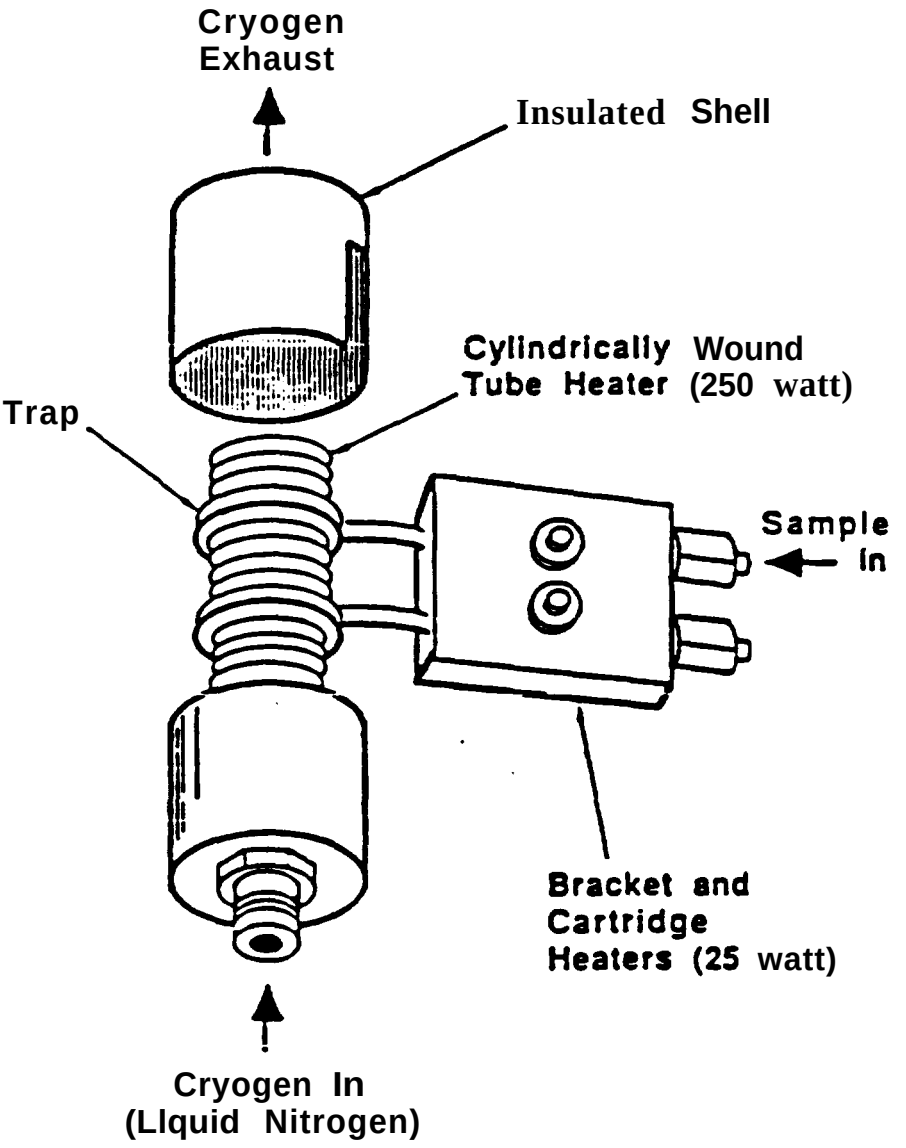


FIGURE 12. CRYOGENIC TRAPPING UNIT

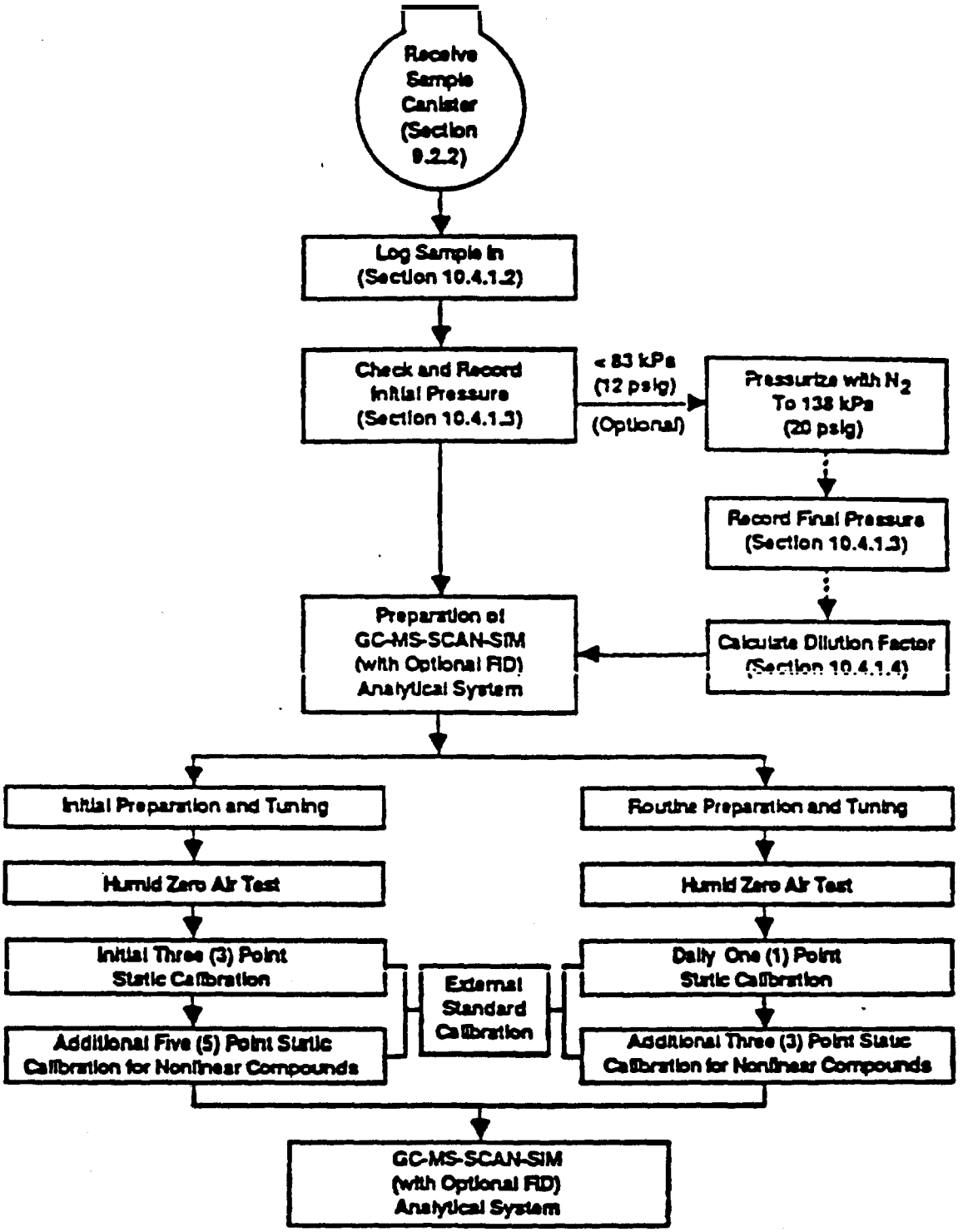


FIGURE 13. FLOWCHART OF GC-MS-SCAN-SIM ANALYTICAL SYSTEM PREPARATION (WITH OPTIONAL FID SYSTEM)

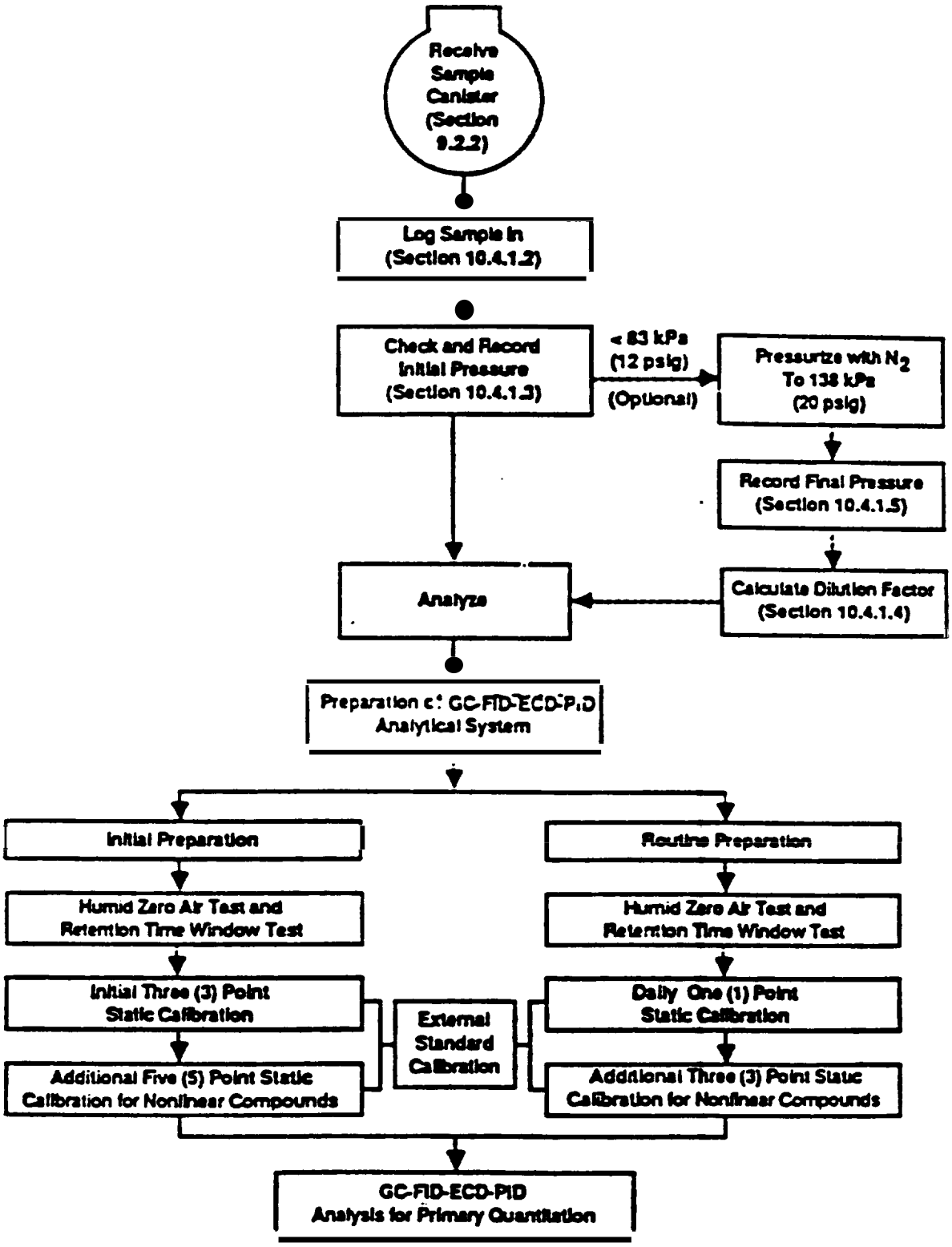
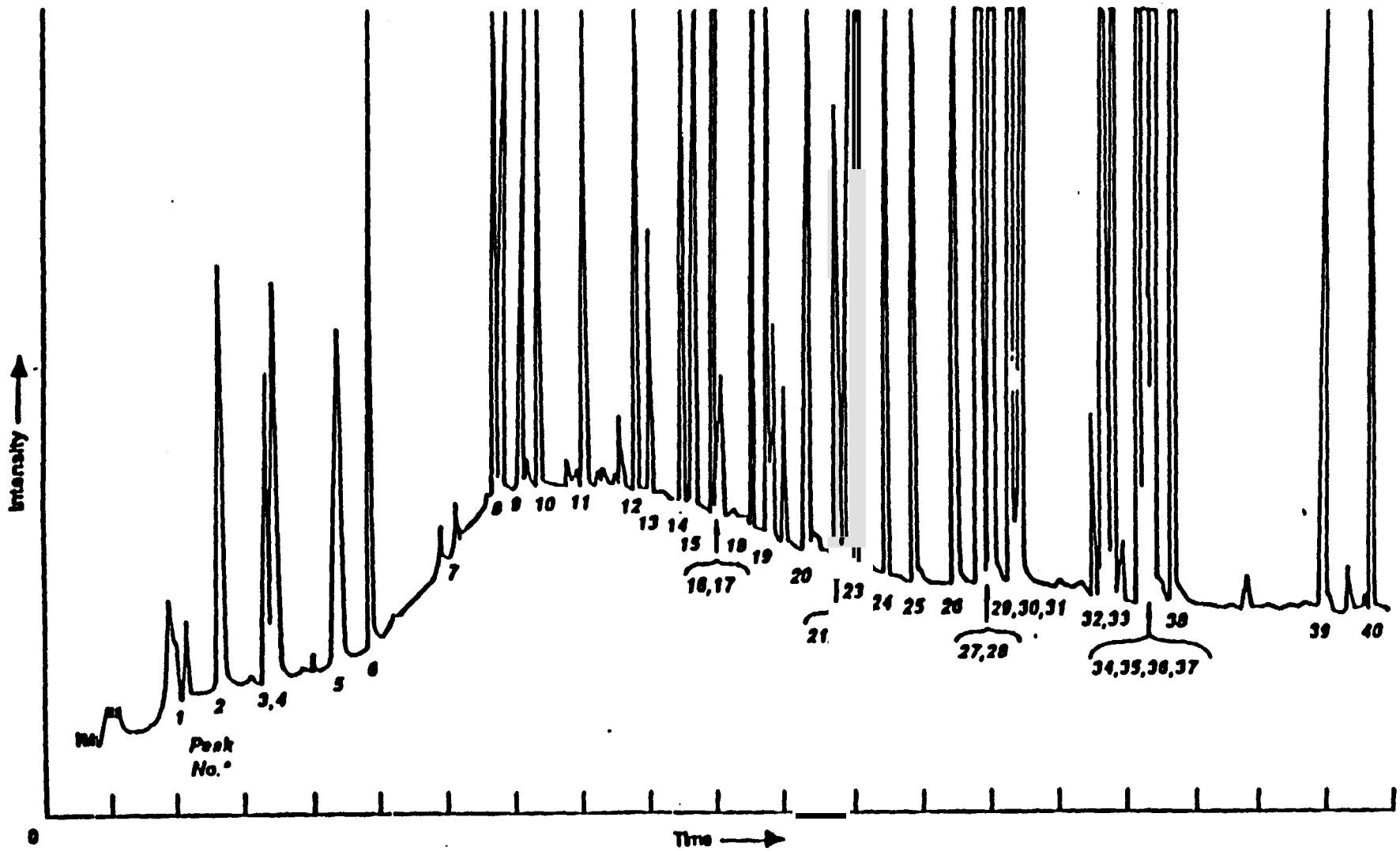
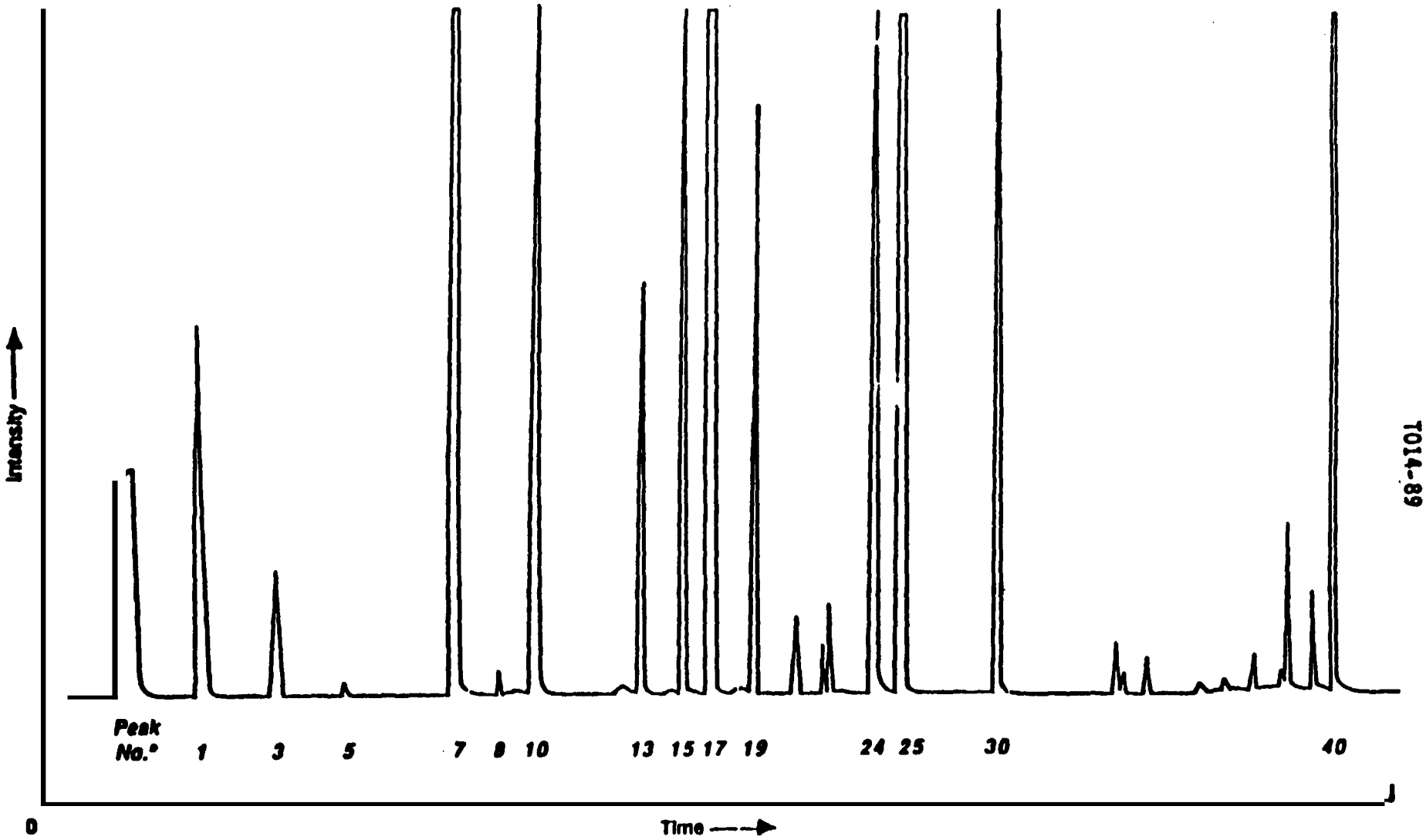


FIGURE 14. FLOWCHART OF GC-FID-ECD-PID ANALYTICAL SYSTEM PREPARATION



* See Table 7.0 For Peak Identification

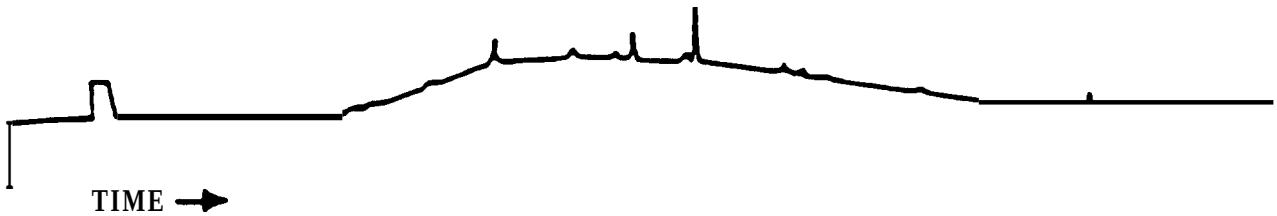
FIGURE 15. TYPICAL FID RESPONSE TO SELECTIVE VOCs



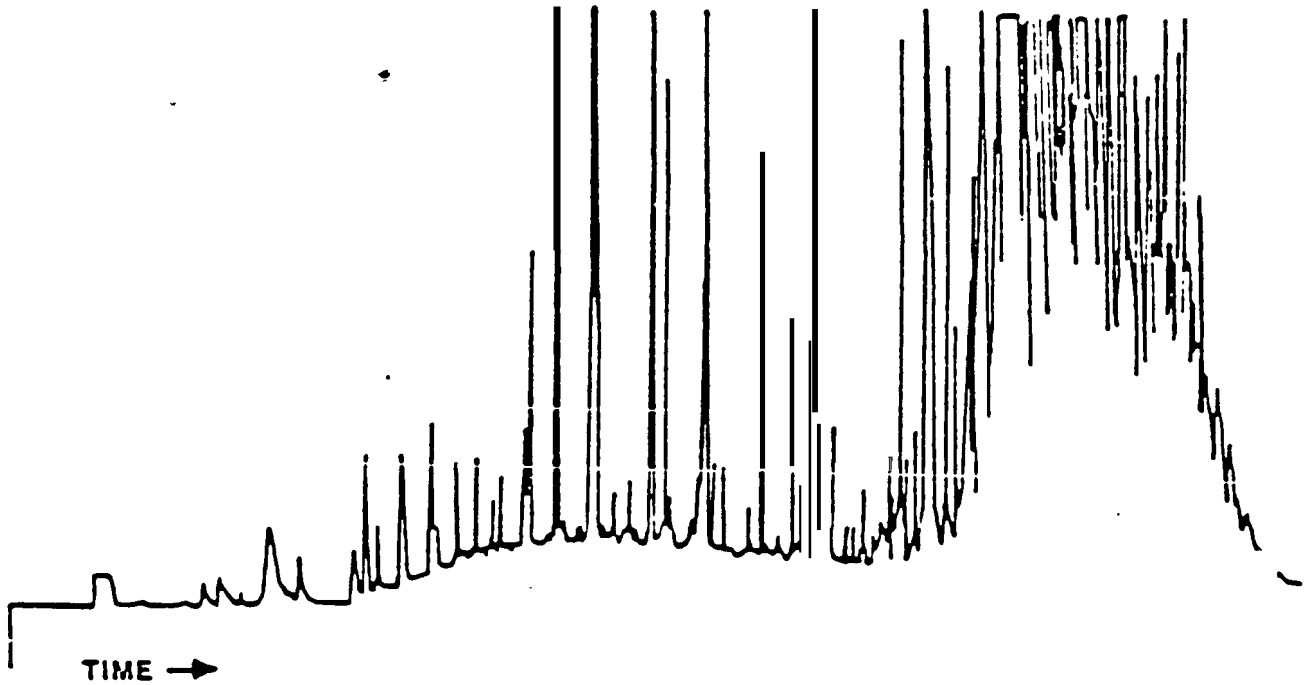
* See Table 7.0 For Peak Identification

FIGURE 16. TYPICAL ECD RESPONSE TO SELECTIVE VOCs

T014-90



(a). Certified Sampler



(b). Contaminated Sampler

FIGURE 17. EXAMPLE OF HUMID ZERO AIR TEST RESULTS FO' CLEAN SAMPLER (a) AND A CONTAMINATED SAMP

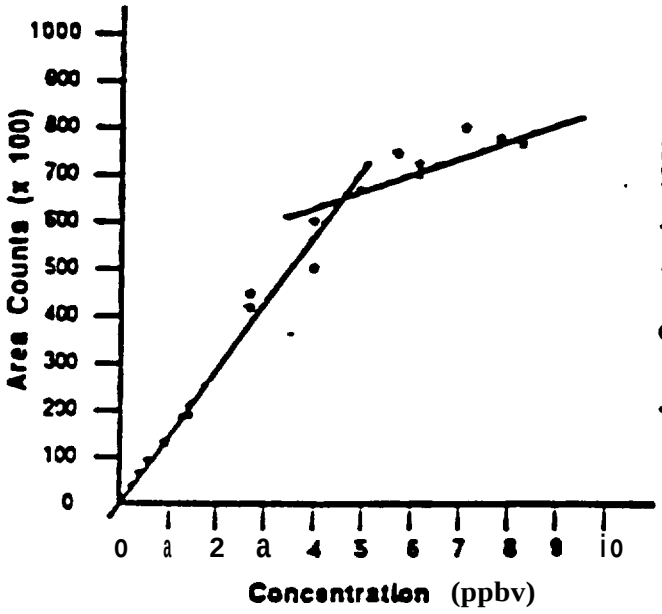


FIGURE 18(a). NONLINEAR RESPONSE OF TETRACHLOROETHYLENE ON THE ECD

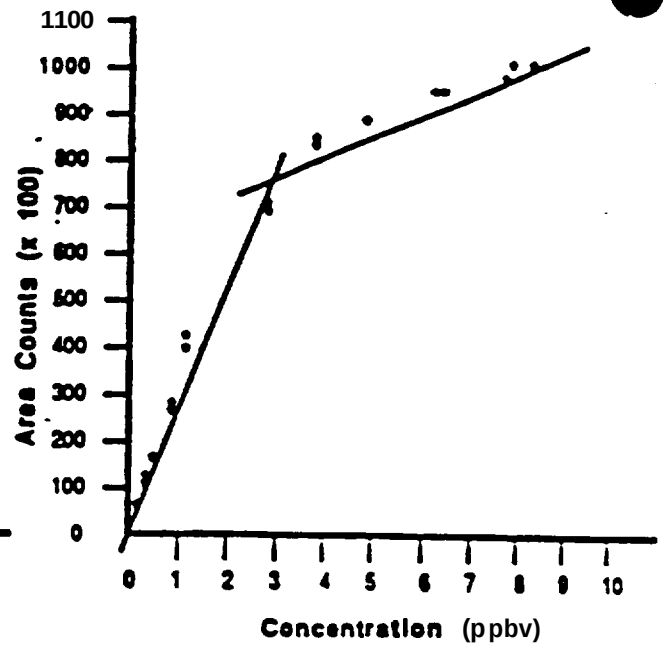


FIGURE 18(b). NONLINEAR RESPONSE OF CARBON TETRACHLORIDE ON THE ECD

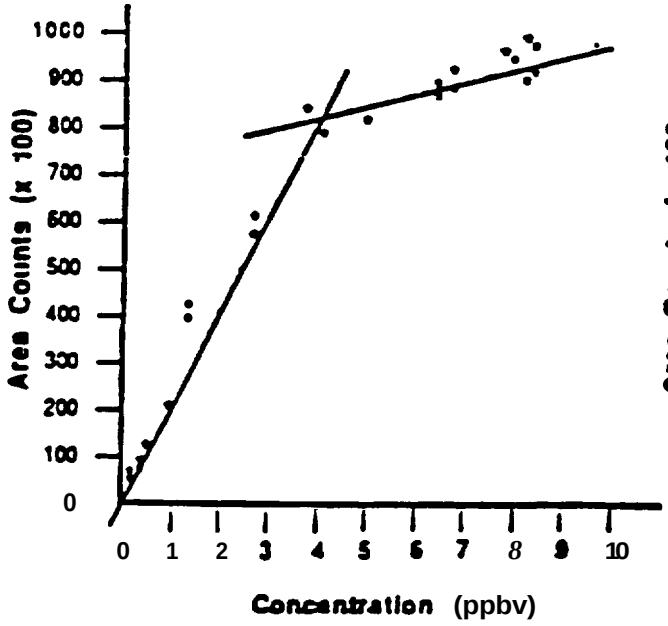


FIGURE 18(c). NONLINEAR RESPONSE OF HEXACHLOROBUTADIENE ON THE ECD

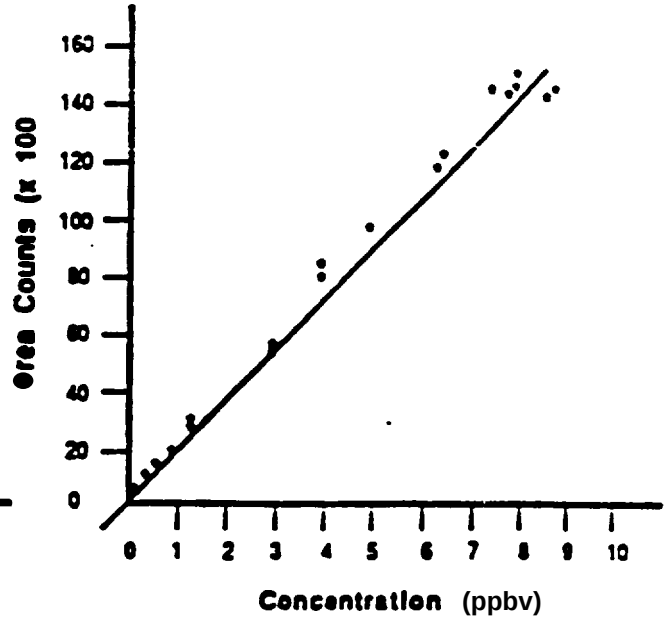


FIGURE 18(d). LINEAR RESPONSE Of CHLOROFORM ON THE ECD

FIGURE 18. RESPONSE OF ECD TO VARIOUS VOCs

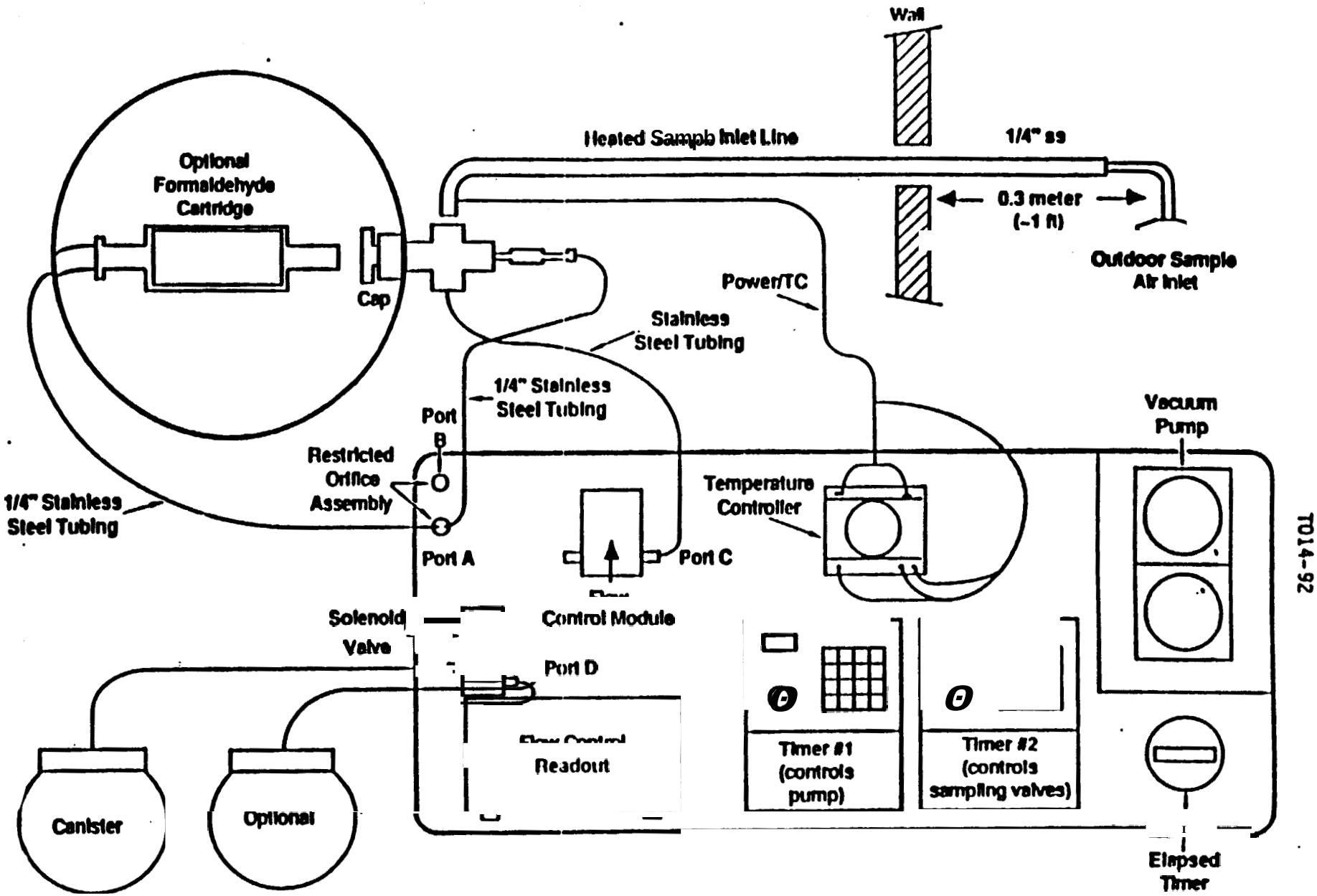


FIGURE 19. U.S. ENVIRONMENTAL PROTECTION AGENCY UATP, SCHEMATIC OF SAMPLE INLET CONNECTIONS SAMPLER

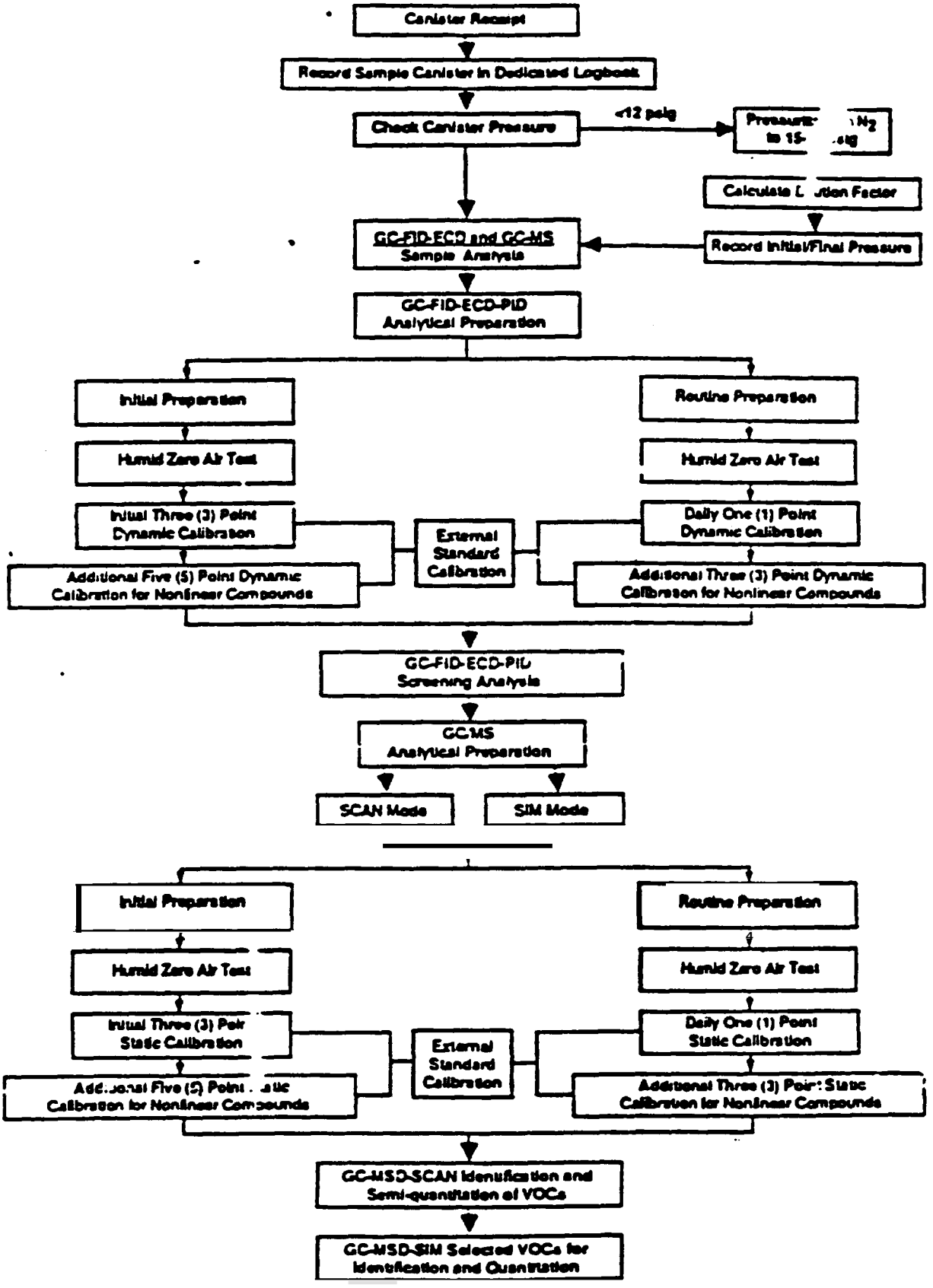


FIGURE 20. FLOWCHART OF ANALYTICAL SYSTEMS PREPARATION.

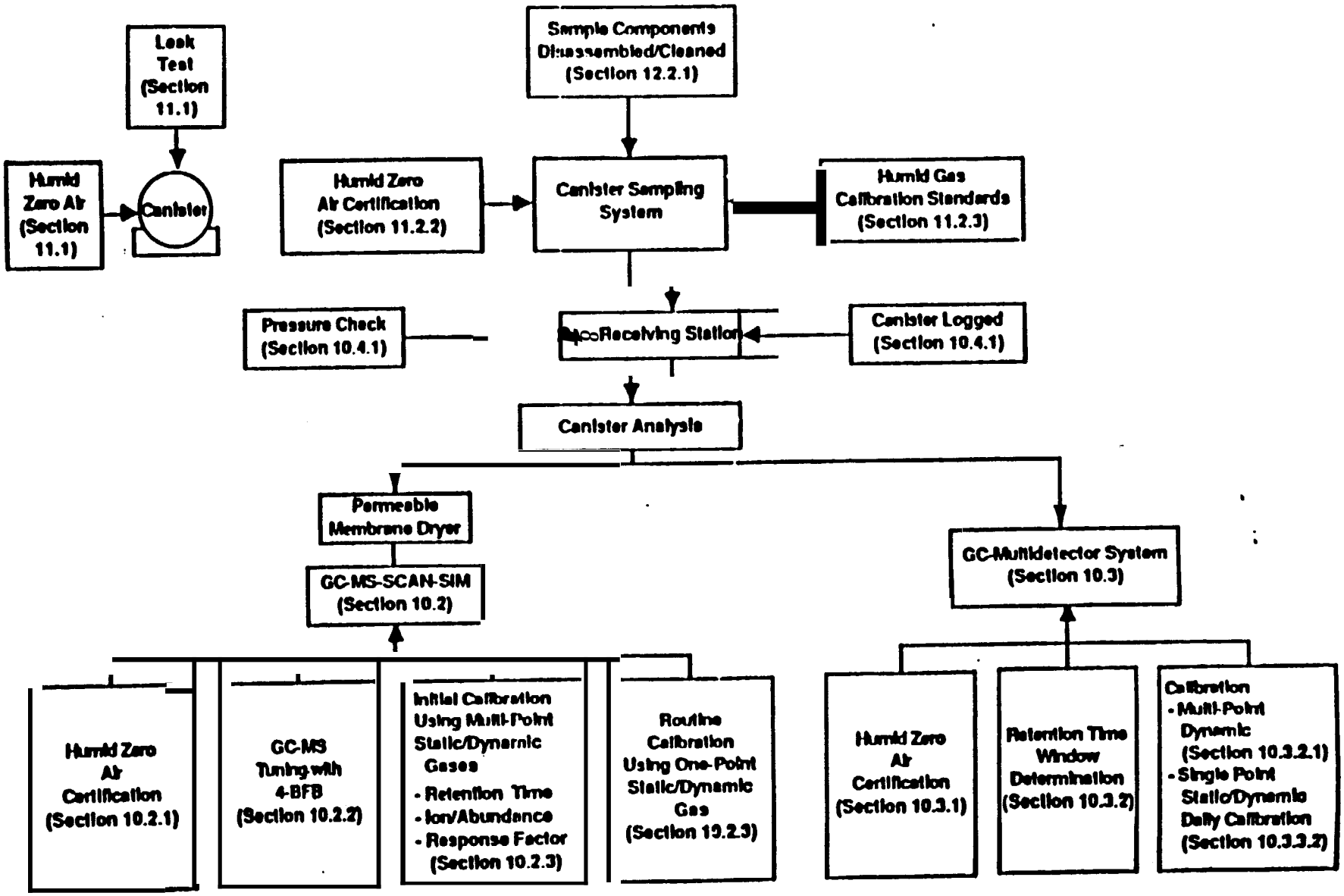


FIGURE 21. SYSTEM QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) ACTIVITIES ASSOCIATED WITH VARIOUS ANALYTICAL SYSTEMS

TABLE A-1. AVAILABLE USEPA PERFORMANCE AUDIf CYLINDERS

Group I Canpounds	Group II Canpounds	Croup III Compounds
Carbon tetrrchlorlde	Tr1ch1oroethy1ene	Pyrldlne (Pyrldlne in Group III cylinders but certified analysis not available)
Chloroform	1,2-dichloroethane	Vinylidene chlorlde
Perchloroethylcnt	1,2-dibromoethane	1,1,2-trichloro-1,2,2-trifluoroethrnt (Freon-I13)
Vinyl chloride	Acetonitrile	1,2-dichloro-1,1,2,2-tetraf1uoroethrne (Freon-114)
Benzene	Trlchlorofluoromethane (Freon-11)	ketone
	Dichlorodifluoromethane (Freon-12)	1-4 Dioxane
	Bromomethane	Toluene
	Methyl ethyl ketone	Chlorobenzene
	1,1,1-trichloroethane	

Grouo 1 Ranges	Group II Ranges	Group III Ranges
7 to 90 ppb	7 to 90 93b	7 to 90 ppb
90 to 430 ppb	90 to 430 ppb	90 to 430 ppb
430 to 10,000 ppb		

Group IV	Group V	
Acrylonitrile	Carbon tetrachloride	Methylene chloride
1,3-butadiene	Chloroform	Trichlorof1uoromethane (Freon-11)
Ethylene oxide	Perch1oroethylene	8romomethane
Methylene chlor1de	Vinyl chlorlde	Toluene
Propylene oxide	Benzene	Chlorobenzene
o-xylene	Trichloroethylene	1,3-Butadiene
	1,2-dichloroethane	a-xylene
	1,2-dibromoethane	Ethyl benzene
	1,1,1-trichlorochtane	1,2-dichloropropane

Group IV Rrnacs	Grouo V Ranges
7 to 90 ppb	1 to 40 ppb
430 to 10,000 ppb	

APPENDIX A

AVAILABILITY OF AUDIT CYLINDERS FROM UNITED STATES ENVIRONMENTAL PROTECTION AGENCY USEPA PROGRAMS/ REGIONAL OFFICES, STATE AND LOCAL AGENCIES AND THEIR CONTRACTORS

1. Availability of Audit Cylinders

   1.1 The USEPA has available, at no charge, cylinder gas standards of hazardous organic compounds rt the ppb level that may be used to udit the performance of indoor air source measurement systems.

   1.2 Each audit cylinder contains 5 to 18 hazardous organic can-pounds in a balance of N_2 gas. Audit cylinders are available in several concentration ranges. The concentration of each organic compound in the audit cyllndcr is within the range illustrated in Table A-1.

2. Audit Cylinder Certification

   2.1 All audit cylinders are periodically analyzed to assure tnrt cylinder concentrations have remained stable.

   2.2 All stability analyses include quality control analyses of ppb hazardous organic gar standards prepared by the National Bureau of Standards for USEPA.

3. Audit Cylinder Acquisition

   3.1 USEPA program/regional offices, state/local agencies, and their contractors may obtain audit cylinders (and an audit gas delivery system, if applicable) for performance audits during:

   - RCRA Hazardous Waste Trial Burns For PHOC's; and
   - Ambient/Indoor Air Measurement of Toxic Organics.

   3.2 The audit cylinders may be acquired by contacting:

Robert L. Lampe
U.S. Environmental Protection Agency
Environmental Monitoring Systems Laboratory
Quality Assurance Division
MD-77B
Research Triangle Park, WC 27711
919-541431

APPENDIX B

OPERATING PROCEDURES FOR A PORTABLE GAS CHROMATOGRAPH EQUIPPED WITH A PHOTOIONIZATION DETECTOR

1. Scope

This procedure is Intended to screen ambient air environments tor volatile organic compounds. Screening it accomplished by collection of VOC samples within an area and analysts onsite using a portable gas chromatograph/integrator (Photovrc Models 10S10, 10S50) or equivalent. This procedure is not intended to yield quantitative or definite qualitative information regarding the substances detected. Rather, it provides a chromatographic "profile" of the occurrence and intensity of unknown volatile conpounds which assists in placement of fixed-site samplers.

2 Applicable Documents

21 ASTW Standards

E260 - Recommended Practice for General Gas Chromatography Procedures

E355 - Practice for Gas Chromatography Terms and Relationships

22 Other Documents

Portable Instruments User's Manual for Monitoring VOC Sources, EPA-34011-86-015, U.S. Environmental Protectton Agency, Washington, DC, June, 1986.

3. Summary of Method

3.1 An air sample is extracted directly fran ambient air and analyzed on-site by a portable GC.

3.2 Analysis is accomplished by drawing an accurate volume of ambient rlr through a sampling port and into a concentrator, then the Sample air is transported by carrier gas onto a packed column and into 8 PID, resulting In response peak(s). Retention times are compared with those in a standard chromatogram to predict the probable identity of the sample components.

4. Significance

4.1 VOCs ate emitted into the atmosphere fran a variety of sources Includlng petroleum refineries, synthetic organic chemical plants,

natural gas processing plants, and automobile exhaust. Many of these VOC emissions are acutely toxic; therefore, their determination in indoor air is necessary to assess human health impacts.

4.2 Conventional methods for VOC determination use solid sorbent and canister sampling techniques.

4.3 Collection of Indoor rlr samples In canisters provides (1) convenient integration of ambient samples over a specific time period, (e.g., 24 hours); (2) remote sampling and central analysis; (3) ease of storing and shipping samples, if necessary; (4) unattended sample collection; (5) analysis of samples from multiple sites with one analytical system; and (6) collection of sufficient sample volume to allow assessment of measurement precision and/or analysis of samples by several analytical systems.

4.4 The use of portable 6t equipped with multidetectors has assisted air toxics programs by using the portable GC as a "screening tool" to determine "hot spots," potential interferences, and semi-quantitation of VOCs/SVOCs, prior to locating more traditional fixed-site samplers.

5. Definitions

Definitions used In this document and In any user-prepared Standard Operating Procedures (SOPs) should be consistent with ASTM Methods D1356 and E355. Abbreviations and symbols pertinent to this method are defined at point of use.

60 Interferences

6.1 The most significant Interferences result from extreme differences in limits of detection (LOD) among the target VOCs (Table B-1). Limitations in resolution associated with ambient temperature, chromatography and the relatively large number of chemicals result In coelution of many of the target components. Coelution of canpounds with significantly different PID sensitivities will mask canpounds with more modest sensitivities. This will be most dramatic in interferences from benzene rnd toluene.

6.2 A typical chromatogram and peak assignments of a standard mixture of target VOCs (under the prescribed analytical conditions of this method) ate illustrated in Figure B-1. Simpler which contain *I* highly complex mixture of components and/or interfering levels of benzene and toluene are analyzed on a second, longer chromatographic column. The same liquid phase In the primary column is contained In the alternate column but rt a hfghcr percent lording.

6.3 Recent designs In commercially available GCs (Table B-2) have pre-concentrator capabilities for sampling lower concentrations of VOCs, pre-column detection with back-flush capability for shorter analytical time, constant column temperature for method precision and accuracy and multidetector (PID, ECD, and FID) capability for versatility. Many of these newer features address the weaknesses and interferences mentioned above.

7. Apparatus

7.1 Gas chromatograph. "A GC (Photovrr Inc., 739 B Parks Ave, Huntington, NY, 11743, Model 10S10 or 10S50), or equivalent used for surveying ambient air environments (which could employ *8* multidetector) for sensing numerous VOCs compounds eluting from a packed column at roan temperatures. This particular portable CC procedure is written employing the photoionization detector as its major Sensing device, as part of the Photovrc Model 10S10 portable GC survey tool. Chromatograms are developed on a column of 3% SP-2100 on 100/120 Supelcoport (0.66 m x 32 mm I.D.) with a flow of 30 cm^3/min air.

7.2 GC accessories. In addition to the basic gas chromatograph, several other pieces of equipment are required to execute the survey sampling. Those include gas-tight syringes for standard Injection, alternate carrier gas supplies, high pressure connections for filling the internal carrier gas reservoir, and if the Model 10S10 is used, a recording integrator (Hewlett Packard, Avondale, PA, Model 3390A), or equivalent.

8. Reagents and Materials

8.1 Carrier gas. "Zero" air [<0.1 ppm total hydrocarbon (THC)] is used as the carrier gas. This gas is conveniently contained in 0.84 m3 (30 ft3) aluminum cylinders. Carrier gas of poorer quality

my result in spurious peaks in sample chromatograms. A Brooks, Type 1355-0051AAA rotameter (or equivalent) with an R-215-AAA tube and glass float is used to set column flow.

8.2 System performance mlxture, A mixture of three target compounds (e.g., benzene, trichloroethylene, and styrene) in nitrogen is used for monitoring instrument performance. The approximate concentration for each of the compounds in tblt mixture is 10 parts per blllion (ppb). This mixture is manufactured in small, disposable gas cylinders [at 275 kPa (40 psi)] from Scott Specirlty Gases, or equivalent.

8.3 Reagent grade nitrogen gas. A small disposable cylinder of hlgh purity nitrogen gas is used for blank injections.

8.4 Sampling syringes. Gas-tight syringes, without attached shut-off valves (Hamilton Model 1002LT), or equivalent are used to introduo accurate sample volumes into the hlgh pressure injectors an the portable gas chromatograph. Gas syringes with shut-off valves are not recommended because of memory problems associated with the valves. For samples suspected of containing hlgh concentrations of volatile compounds, disposable glass syringes (e.g. Glaspak, or equivalent) with stainless steel/Teflon® hub needles are used.

8.5 High pressure filler. An adapter (Photorrc SA101, or equivalent) for filling the internal carrier gas reservoir on the portable 6C is used to deliver 'zero' air.

9. Procedure

9.1 Instrument Setup

9.1.1 The portable gas chromatograph must be prepared prior to use in the ambient survey sampling. The pre-sampling activities consist of filling the lnttmrl carrier gas cylinder, charging the internal power supply, adjusting individual column carrier gas flows, and stabilizing the photoionization detector.

9.1.2 The internal reservoir is filled with "zero" air. The internal 12V, 6AH lead/acid battery can be recharged to provide up to eight hours of operation. A battery

which It discharged will automatically cause the power to the instrument to be shut down and will rqulrc an overnight charge. Doring AC operation, the batteries will automatically be trickle-charged or In a standby mode.

9.1.3 The portable 6C should be operated (using the Internal battery power supply) at least forty minutes prior to collection of the first sample to insure that the photoionzation detector hat stabilized. Upon arriving at the area to be sampled, the unit should be connected to AC power, if available.

9.2 Sample Collection

9.2.1 After the portable gas chromatograph is located and connected to 110V AC, the carrier gas flows must be adjusted. Flows to the 1.22 meter, 5% Sf-30 And 0.66 meter, 3% SP2100 columns are adjusted with needle valves. Flows of 60 cm^3/min (5% SE-30) and 33 cm^3/min (3% SP2100) are adjusted by means of a calibrated rotameter. Switching between the two columns Is accomplished by turning the valve located beneath the electronic module. During long periods of inactivity, the flows to both columns should be reduced to conserve pressure In the Internal carrier gas supply. The baseline on the recorder/integrator is set to 20% full scale.

9.2.2 Prior to analysis of actual samples, an injection of the performance evaluation mixture must be made to verify chromatographic and detector performance. This Is accomplished by withdrawing 1.0 mL samples of this mixture from the calibration cylinder and injecting it onto the 3% SP2100 column. The next sample analyzed should be a blank, consisting of rtagtnt grade nitrogen.

9.2.3 Ambient air samples are injected onto the 3% SP2100 column. The chromatogram is developed for 15 minutes. Samples which produce particularly complex chromatograms,

especially for early eluting components, are reinjected on the SS SE-30 column. (Note: In no instance should a syringe which has been used for the injection of the calibrant/system performance mixture be used ?or the acquisition and collection of samples, or vice versa.)

9.2.4 Samples have generally been collected from tk ambient air at sites which are near suspected sources of VOCs and SVOCs and compared with those which are not. Typically, selection of sample locations is based on the presence of chemical odon. Samples collected in areas without detectable odon have not shown significant PID responses. Therefore, sampling efforts should be lnltlally concentrated on "suspect" environments (i.e., those which have appreciable odon). The objective of the sampling is to locate sources of the target compounds. Ultlartely, samples should be collected throughout the entire location but with particular attention given to areas of hlgh or frequent occupatlon.

9.3 Sample Analysis

9.3.1 Qualitative analysis. Positive identification of sample components is not the objective of this 'screening' procedure. Visual comparison of retention times to those in I standard chromatogram (Figure B-1) arc used only to predict the probable sample component types.

9.3.2 Estimation of levels. As with qualltrtlvc analysis, estimates of component concentrations are extremely tentrtlvc and are based on instrument responses to the calibrant species (e.g., benzene, trichloroethylene, styrene), the proposed component identification, and the difference in response between sample component and calibrant. For purposes of locating pollutant emission sources, roughly estimated concentrations and suspected compound types are considered sufficient.

10. Performance Criteria an8 Quality Assurance

Required quality assurance measures and guidance concerning performance criteria that should be achieved within each laboratory are summarized and provided in the following section.

10.1 Standard Operating Procedures

10.1.1 SOPs should be generated by the users to describe and document the following activities in their laboratoy: (1) assembly, calibration, leak check, and operation of tht specific portable GC Sampling system and equipment ustd; (2) preparation, storage, shipment, and handling of the portable 6C sampler: (3) purchase, certification, and transport of standard reference materials; and (4) all aspects of data recording and processing, including lists of computer hardware and software used.

10.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the personnel conducting the survey wort.

10.2 Quality Assurance Program .

10.2.1 Reagent and materials control . The carrier gas employed with the portable 6C is "zero air" containing less than 0.1 ppm VOCs. System performance mixtures are certified standard mixtures purchased from Scott Specialty Gases, or qulvalent ,

10.2.2 Sampling protocol and chain of custody. Sampling protocol sheets mutt be completed for each sample. Specifics of the sample with regard to sampling location, sample volume, analysis conditions, rnd supporting calfbrrtton and visual inspection information are detailed by these documents. An example form is exhibited in Table B-3.

10.2.3 Blanks, Duplicates, rnd System Performance Samples

10.2.3.1 Blanks and Duplicates. tan percent of all Injections made to the portable GC are blanks,

where the blank is reagent grade nitrogen gas. This is the second injection in each sampling location. An additional 10% of all injections made are duplicate injections. This will enhance the probability that the chromatogram of a sample reflects only the composition of that sample and not any previous injection. Blank injections showing a significant amount of contaminants will be cause for remedial rctlon.

10.2.3.2 System Performance Mixture. An Injectlon of the system performance alxture will be made at the beginning of a rltlt *ta* a particular sampling location (i.e., the first injection). The range of acceptable chromatographic system performance criteria and detector response is shown in Table B-4. These criteria are selected with regard to the intended rpplicrtlon of thlt protocol and the limit/ availability of standard mixtures In this area. Corrective rctlon should be taken with the column or PID before sample injections are made If the performance is deemed out-of-range. Under thls regimer of blanks and system performance samples, approximately eight samples can be collected and analyzed in a three hour rltlt to each sampling location.

10.3 Method Precision and Accuracy

The purpose of the analytical approach outlined in this method Is to provide presumptive information regarding the presence *of* selected VOCs and SVOCs emissions. In thlt context, precision and accuracy are to be determined. However, qurlity assurance criteria are described in Section 10.2 which insure the samples collected represent the indoor environment.

10.4 Range and Limits of Detection

The range and llaltr of detection of thlt method are highly compound dependent due to large differences in response of the portable GCs photoionization detector to the various

target compounds. Aromatic compounds and olefinic halogenated compounds will be detected at lower levels than the halomethanes or aliphatic hydrocarbons. The concentration range of application of this method is approximately two orders of magnitude.

TABU B-1

ESTIMATED LIMITS OF DETECTION (LOD) FOR SELECTED VOCs
BASED ON 1 uL SAMPLE VOLUME

Compound	LOD (ng)	LOD (ppb)
Chloroform[a]	2	450
1,1,1-Trichloroethane[a]	2	450
Carbon tetrachloride[a]	2	450
Benzene	.006	2
1,2-Dichloroethane[b]	.05	14
Trichloroethylene[b]	.05	14
Tetrachloroethylene[b]	.05	14
1,2-Dibromoethane	.02	2
p-Xylene[c]	.02	4
m-Xylene[c]	.02	4
o-Xylene[d]	.01	3
Styrene[d]	.01	3

[a]Chloroform, 1,1,1-Trichloroethane, and Carbon tetrachloride coelute on 0.66 m 3% SP2100.
[b]1,2-Dichloroethane, Tricholroethylene, and Tetrachloroethylene coelute on 0.66 m 3% SP2100.
[c]p-Xylene and m-Xylene coelute on 0.66 m 3% SP2100.
[d]Styrene and o-Xylene coelute on 0.66 m 3% SP2100.

TABLE B-2

CMMRCIALLY AVAILABLE PORTABLE VOC DETECTION INSTRUMENTS

Monitor	Detection principle	Range, ppm	Sensitivity	Response time, s	Accessories	Calibration Techniques	Weaknesses	Service Rate	Lack of Response	Cost,$	Samp Rate L/m
550,551 555,580 (AIB, Inc.	PID, FID	0-200, 0-2000, 0-10,000	0.1 ppm at 0-200 ppm	<5		• Bag Sampling	• Umbilical cord too short • Digital readout hard to read • Flame out frequently	8 hrs		4,300	1.5
OVA 108, 128 Century Systems, Inc. (Foxboro)	FID	0-10, 0-100, 0-1000, 0-10,000, 0-100,000	0.2 ppm (Model 128) 0.5 ppm (Model 108)	2 2	• Internal Desorbers available • Optional GC available	• Head Space • Direct Injection • Bag Samp.	• Battery failure • Sample line blocks • Compounds containing O_2/N give low response • Reg. resp. to CO/CO_2	8 hrs		6,300	
PI-101 (Nuc Systems, Inc)	FID	1 1-20 1-200 1-2000	0.1 ppm Low molecular weights aromatics	<5	• Three lamps available • 9.5 (aromatics) • 10.2 (2-4 compounds) • 11.7 (halocarbons)	• External Gas Cyl. • Bag Samp.	• Three lamps - may miss something	10 hrs	• Cl hydrocarbons • CH_4	4,355	0.5
TLV Sniffer (Bacharach)	Catalytic combustion	0-500 0-5000 0-50,000	2.0 ppm	5		• Bag Samp. • Head Space				900	
Ecolyzer 400 (Energetics Science)	Catalytic combustion	0-100% LFL	1% LFL	15		• Bag Samp.	• Changes in gas temp/humidity effects response				
Miran 1A (Foxboro)	IR	ppm to %	1 ppm	1,4,10 and 40						9,500	
Miran 1B (Foxboro)	IR	ppm to %								12,500	
Scanter (Santec)	GC/EC, Argon Ionization PID		0.01 ppb Cl organics	2	Preconcentrator Thermal Desorption GC Columns Auto Cal. from Integral Gas Cylinder	• Internal gas cyl. • Preconcentrator • GC Column				12,850	
Photovac Standard Automatic Computer Auto Comp. Communication	PID (UV Light)	0	0.1 ppb Benzene with signal-to-noise ratio 4:1. Good for aromatics	2	• Dual Column • Manual/Auto Injection • Column Cond. • Pre-flush • Auto Dial Modem • Programmable		• Column operates at ambient temp. • STD in lab, then to field at diff. temp • Can't inject liquid samp. • Light fractions interfere		• H_2O • O_2	6,995 8,995 10,500 10,955 12,955	
Photovac Tip	PID	0-2000 ppm	0.05 ppm Benzene	4							

TO14-B12

TABLE B-3

PORTABLE CAS CHROMATOGRAPH
SAMPLING DATA SHEET

DATE: _____ LOCATION: _____ TIE: _____

CHROMATOGRAPHIC CONDITIONS : _____

COLUMN 1: COLUMN TYPE:

I.D. (mm):_____ LENGTH (an):_____ FLOW (mL/min):_____

COLUMN 2: COLUMN TYPE:

I.D. (mm):_____ LENGTH (mm):_____ FLOW (mL/min):_____

INJ. NO.	INJ. VOL.	COLUMN NO.	SETTING	LOCATION

SITE PLAN (Indicate sampling locations):

DATE _____ SIGNATURE _____

TABLE B-4

SYSTEM PERFORMANCE CRITERIA FOR PORTABLE GC[a]

Criteria	Test Compound	Acceptable Range	Suggested Corrective Action
PID Response	Trichloroethylene	$\geq 10^8$ uV-sec/ng	Re-tune or replace lamp
Elution Time	Styrene	2.65 ± 0.15 min	Inspect for leaks, adjust carrier flow
Resolution[b]	Benzene/Trichloroethylene	≥ 1.4	Replace column

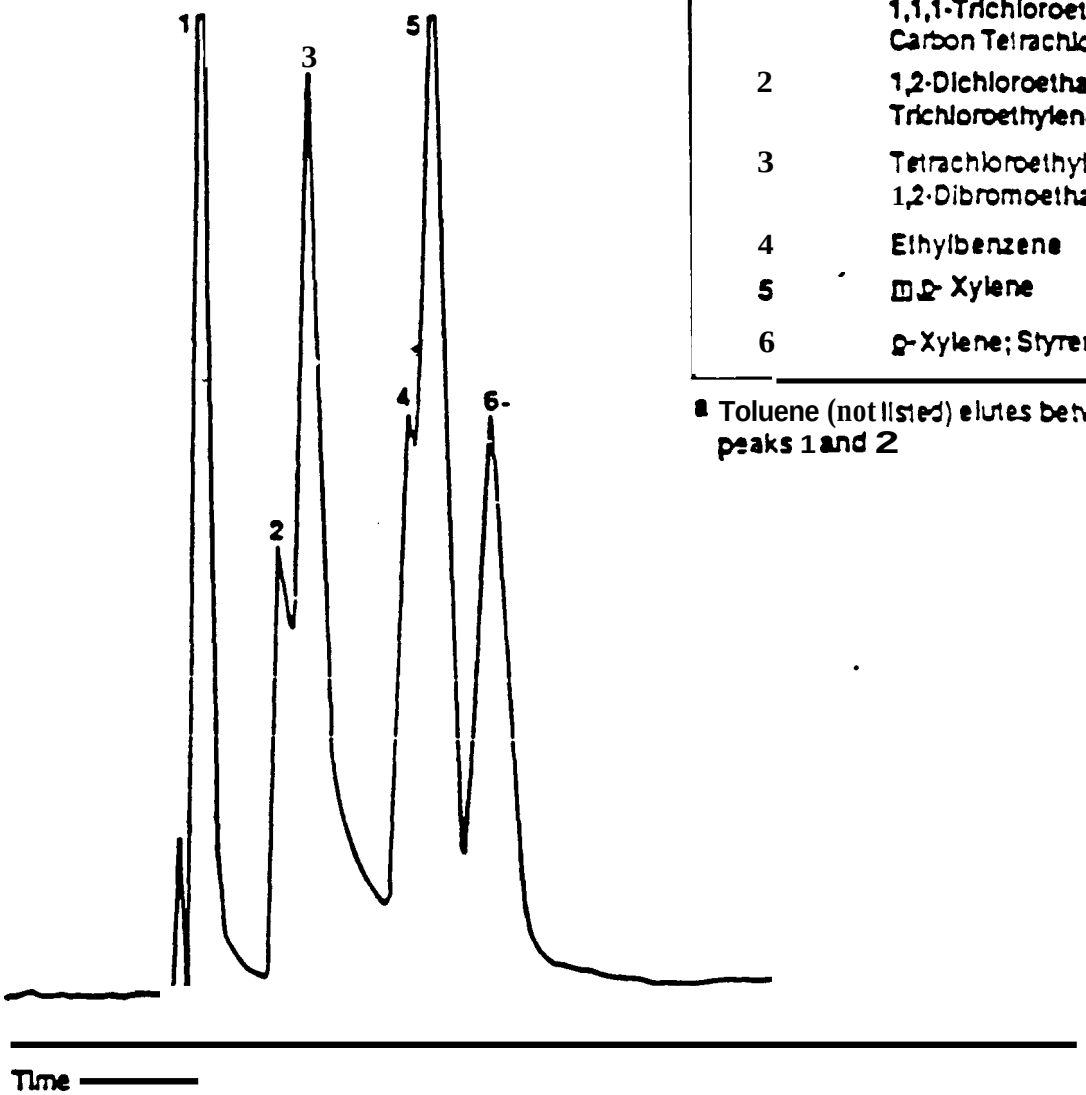
[a]Based on analysis of a vapor mixture of benzene, styrene, and trichloroethylene.
[b]Define by: $R = 2d/(W_1+W_2)$; where d = distance between the peaks and W = peak width at base.

TABLE B-5

ESTIMATED LIMITS OF DETECTION (LOO) FOR SELECTED VOCs

Compound	LOD (ng)	LOD (ppb)
Chloroform[a]	2	450
1,1,1-Trichloroethane[a]	2	450
Carbon tctrachloride[a]	. 2	450
Benzene	.006	2
1,2-Dichloroethane[t]	.0S	14
Trichloroethylen[b]	.0S	14
Tetrachloroethylene[b]	.0S	14
1,2-Dibromoethane	.02	2
p-Xylene[c]	.02	4
m-Xylene[c]	.02	4
o-Xylene[d]	.01	3
Styrene[d]	.01	3

[a]Chloroform, 1,1,1-Trichloroethane, and Carbon tetrachloride coelute on 0.66 m 3% SP2100.
[b]1,2-Dichloroethane, Trichloroethylene, and Tetrachloroethylene coelutc on 0.66 m 3% SP2100.
[c]p-Xylene and m-Xylene coelute on 0.66 m 3% SP2100.
[d]Styrene and o-Xylene coelute on 0.56 m 3% SP2100.



Peak Assignments For Standard Mixture

Peak NO.	Compound(s)[a]
1	Benzene; Chloroform; 1,1,1-Trichloroethane; Carbon Tetrachloride
2	1,2-Dichloroethane; Trichloroethylene
3	Tetrachloroethylene; 1,2-Dibromoethane
4	Ethylbenzene
5	m,p- Xylene
6	o-Xylene; Styrene

[a] Toluene (not listed) elutes between peaks 1 and 2

FIGURE B-1. TYPICAL CHROMATOGRAM OF VOCs DETERMINED BY A PORTABLE GC

APPENDIX C

INSTALLATION AND OPERATION PROCEDURES FOR U.S. ENVIRONMENTAL PROTECTION AGENCY'S URBAN AIR TOXIC POLLUTANT PROGRAM SAMPLER

1. Scope

1.1 The subatmospheric sampling system described In this method has ken modified and redesigned specifically for use In USEPA's Urban Air Toxic Pollutant Program (UATP), a jolnt project of USEPA's Office of Air Quality Planning and Standards, the Environmental Monitoring Systems Laboratory, and the participating state air pollution control agencies. The purpose of UATP Is to provide analytical support to the states In their assessment of potential health risks from certain toxic organic compounds that may be present in urban atmospheres. The sampler is described In the paper, "Automatic Sampler for Collection of 24-Hour Integrated Whole-Air Samples for Organic Analysis," to be presented at the 1988 Annual Meeting of APCA, Dallas, Texas, June, 1988 (Paper No. 88-150.3).

1.2 The sampler is based on the collection of whole air samples in 6-liter, SUMMA® passivated stainless steel canisters. The sampler features electronic timer for ease, accuracy rnd flexibility of sample period programming, an independently setable presample warm-up and ambient air purge period, protection from loss of sample due to power interruptions, and a self-contained configuration housed in an all-metal portable case, as illustrated In Figure C-1.

1.3 The design of the sampler is pumpless, using an evacuated canister to draw the ambient sample air Into Itself at a fixed flow rate (3-5 cm^3/min) controlled by an electronic mass flow controller. Because of the relatively low sample flow rates necessary for the integration periods, auxiliary flushing of the sample inlet line is provided by a small, general-purpose vacuum pump (not In contact with the sample air stream). Further, experience has shown that lnltt lines and surfaces sometimes build up or accumulate substantial concentrations of organic materials under Stagnant (zero flow rate) conditions. Therefore such lines and surface need to be purged and equilibrated to the sample air for some time prior to the beginning of the actual sample collection period. For this reason, the sampler Includes dual timers, one of which is set to start the pump several hours prior to the specified start of the sample period to purge the Inlet lines rnd

surfaces. As illustrated in Figure C-1, sample air drawn into the canister passes through only four components: the heated inlet line, a 2-micron particulate filter, the electron flow controller, and the latching solenoid valve.

2. Summary of Method

2.1 In operation, timer 1 It set to start the pump about 6 hours before the scheduled sample period. The pump draws sample air in through the sample inlet and particulate filter to purge and equilibrate these components, at a flow rate limited by the capillary to approximately 100 cm^3/min. Timer 1 also energizes the heated inlet line to allow it to come up to its controlled temperature of 65 to 70 degrees C, and turns on the flow controller to allow it to stabilize. The pump draws additional sample air through the flow controller by way of the normally open port of tk 3-way solenoid valve. This flow purges the flow controller and allows It to achieve a stable controlled flow at the specified sample flow rate prior to the sample period.

22 At the scheduled start of the sample period, timer 2 is set to activate both solenold valves. Ykn activated, the 3-way solenoid valve closes its normally open port to stop the flow controller purge flow and opens its normally closed port to start flow through the aldehyde sample cartridges. Simultaneously, the latching solenoid valve opens to start sample flow Into the canister.

2.3 At the end of the sample period, timer 2 closes the latching solenoid valve to stop the sample flow and seal the sample in the canister and also de-energizes the pump, flow controller, 3-way solenoid. and heated Inlet llne. During operation, the pump and sampler are located external to the sampler. The 2.4 meter (8 foot) heated inlet llne is installed through the outside wall, with most of ltt length outside and terminated externally with an inverted glass funnel to exclude precipitation. The Indoor end is terminated In a stainless steel cross fitting to provide connections for the canister sample and the two optional formaldehyde cartridge samples.

3. Sampler Installation

3.1 The sampler must be operated indoors with the temperature between 20-32°C (68 to 90°F). The sampler case should be located conveniently

on a table, shelf, or other flat surface. Access to a source of 115 vac line power (500 watts min) Is also required. The pump is removed from the sampler case and located remotely from the sampler (connected with a 1/4 Inch O.D. extension tubing and a suitable electrical extension cord).

3.2 Electrical Connections (Figure C-1)

3.2.1 The sampler cover is removed. The sampler Is not plugged Into the 115 vac power until all other electrical connections are completed.

3.2.2 Tht pump Is plugged Into its power connector (if not already connecttd) and the battery connectors are mapped onto the battery packs on the covers of both timers.

3.2.3 The sampler power plug is inserted into a 115 volts ac line grounded receptacle. The sampler must be grounded for operator safety. The electrical wires are routed and tied so they remain out of the way.

3.3 Pneumatic Connections

3.3.1 The length of 1/16 inch O.D. stainless steel tubing is connected fran port A of the sampler (an the right side of the flow controller module) to the air inlet line.

3.3.2 The pump Is connected to the sampler with 1/4 inch O.D. plastic tubing. This tubing may be up to 7 meters (20 feet) long. A short length of tubing is Installed to reduce pump noise. All tubing is conveniently routed and, If necessary, tied in place.

4. Sampler Preparation

4.1 Canister

4.1.1 The sample canister is Installed no more than 2 days before the scheduled sampling dry.

4.1.2 With timer #1 ON, the flow controller is allowed to warm up for at least 15 minutes, longer If possible.

4.1.3 An evacuated canister is connected to one of the short lengths of 1/8 Inth O.D. stainless steel tubing from port B (soltnoid valve) of the sampler. The canister valve is left closed. The Swagelok fitting on the canister must not be cross-threaded. Tht connection is tightened snugly with a wrench.

4.1.4 The end of the other length of stainless steel tubing from port B (solenoid valve) is connected with a Swagelok plug.

4.1.5 If duplicate canisters are to be sampled, the plug is removed from the second 1/8 inch O.D. stainless steel tubing from port B (solenoid valve) rnd the second canister is connected, The canister valve is left closed.

4.1.6 The ON button of timer #2 is pressed. The flow through the flow controller should be stopped by this action.

4.1.7 The flow controller switch is turned to 'READ' and the zero flow reading is obtained. If this reading is nut stable, wait until the reading is stabilized,

4.1.8 The flow controller switch is turned to 'SET' and the flow setting is adjusted to the algebraic SUM of the most recent entry on Table C-1 and the zero reading obtained in step 4.1.7 (If the zero reading is negative, SUBTRACT the zero reading from the Table C-1 value). Be sure to use the correct Tabla C-1 flow value for one or two canisters, as appropriate. [Note: If the analytical laboratory determines that the canister sample pressure is too low or too high, a new flow setting or settings will be issued for the sampler. The new flow setting should be recorded In Table C-1 and used until superseded by new settings.]

4.1.9 Timer I2 is turned OFF to again start the flow through the flow controller, With the pump (tlwr #1) ON rnd the sampling valve (timer #2) OFF, the flow controller is turned to 'READ' and the flow is verified to be the same as the flow setting made in step 4.1.8. If not, the flow setting Is rechecked In step 4.1.8 and the flow setting is readjusted If necessary,

4.1.10 The OFF button of timer #1 is pressed to stop the pump.

4.1.11 The canister valve(s) are fully opened,

4.2 Timers

4.2.1 Timer #2 is set to turn ON rt the scheduled ON time for the sample period, rnd OFF at the scheduled OFF time. (See the subsequent section on rettlng the timers.)

Normal ON time: 12:00 AM on the scheduled sampling day.

Normal OFF time: 11:59 PM on the scheduled sampling day.

(The OFF time is 11:59 PM instead of 12:00 AM so that the dry number for the OFF time is the same as the day number for the ON time.) Be sure to set the correct day number.

4.2.2 Timer #1 is set to turn ON six (6) hours before the beginning of the scheduled sample period and OFF at the scheduled OFF time for the sample period (same OFF time as for timer #2). (See the subsequent section on setting the timers.) Normal ON time: 06:00 PM on the day prior to the scheduled sampling day. Normal OFF time: 11:59 PM on the scheduled sampling day. [Note: The timers are wired so that the pump will be on whenever either timer is on. Thus the pump will run if timer #2 is ON even if timer #1 is OFF.]

4.2.3 The elapsed time meter is set to 0.

4.3 Sampler Check

4.3.1 The following must be verified before leaving the sampling site:

(1) Canister(s) is (are) connected properly and the unused connection is capped if only one canister is used.
(2) Canister valve(s) is (are) opened.
(3) Both timers are programmed correctly for the scheduled sample period.
(4) Both timers are set to "AUTO".
(5) Both timers are initially OFF.
(6) Both timers are set to the correct current time of day and dry number.
(7) Elapsed time meter is set to 0.

4.4 Sampler Recovery (Post Sampling)

4.4.1 Left valve on the canister is closed.

4.4.2 The canister is disconnected from the sampler, the sample data sheet is completed, and the canister is prepared for shipment to the analytical laboratory.

4.4.3 If two canisters were sampled, step 2.4.2 is repeated for the other canister.

5. Timer Setting

Since the timers are 7-day timers, the days of the week are numbered from 1 to 7. The assignment of day numbers to days of the week is

indicated on the timer keypad: 1 • Sunday, 2 • Monday, 3 • Tuesday, 4 • Wednesday, 5 • Thursday, 6 • Friday, and 7 • Saturday, This programming is quite simple, but some timers may malfunction or operate erratically if not programmed exactly right, To assure correct operation, the timers should be reset and completely reprogrammed 'from scratch' for each sample. The correct current time of day is re-entered to reprogram the timer. Any program in the timer's memory is erased by resetting the timer (pressing the reset button). The timer is set by the following:

(1) pressing the reset button,
(2) entering the correct dry number and time of day,
(3) entering the ON and OFF times for the sample period, and
(4) verifying that the ON and OFF time settings are correct.

5.1 Timer Reset

The timer reset button is pressed, which is recessed In a small hole located just above the LED (light emitting diode) indicator light. A small object that will fit through the hole, such as a pencil, match, or pen is used to press the timer. After reset, the timer display should show |1| |10:00|. [Mote: The ti —— may operate erratically when the batteries ara discharged, which happens when the sampler is unplugged or without power for several hours. When the sampler is again powered up, several hours may be required to recharge the batteries. To avoid discharging the batteries, the battery pack should be disconnected from the timer when the sampler is unplugged.]

5.2 Date and Time Entry

The selector switch Is turned to SET and the number button corresponding to the day number Is pressed. (For example, a '2' is pressed for Monday.) The current time of day is entered. (For example, if the time is 9:00 AM, 900 It pressed.) AM or PM is pressed as applicable. (Display should show |2| |'9:00| for 9:00 AM Monday.) (Mote: ' indicates AM and indicates PM.)' The CLOCK button is pressed, (Display should show |-| |--:--|) If an error is made, |E| |EE:EE| is shorn on the display. The CLEAR button is pressed and the above steps are repeated, The selector switch is turned to AUTO or MAN to verify correct time-setting.

5.3 ON and OFF Entry

The selector switch is turned to SET. The ON and OFF program is entered In the following order: dry, number, time, AM or PM, ON or OFF, (Example: To turn ON at 12:00 AM on day 5 (Thursday); 5, 1200, AM, ON Is entered). (Example: To turn OFF at 11:59 PM on day 5 (Thursday), 5, 11:59, PM, OFF Is entered.) If the display Indicates an error (|E| |EE:EE|), the timer is reset. The selector switch is turned to AUTO.

5.4 ON and OFF Verification

5.4.1 The selector switch is turned to REYIEY. The number of the scheduled sample day Is pressed. ON Is pressed. The , dlsplay should show the time of the beginning of the sample period (for example, |5| |'12:00|). [' indicates AM.] ON is pressed again. The dlsplay should show |5| |--:--|, indicating no other ON times are programed.

5.4.2 OFF is pressed. The display should show tht time of the end of the sample period, (for example, |5| |, 11:59|). PM is indicated by the ',' nark before the time. OFF is pressed again. The display should show |5| |--:--|, indicating .no other OFF times are programmed. The selector is switched to AUTO. If anything is Incorrect, the timer Is reset and reprogrammed.

TABLE C-1

WET FLOW CONTROLLER SETTING

DATE	1 CANISTER	2 CANISTERS

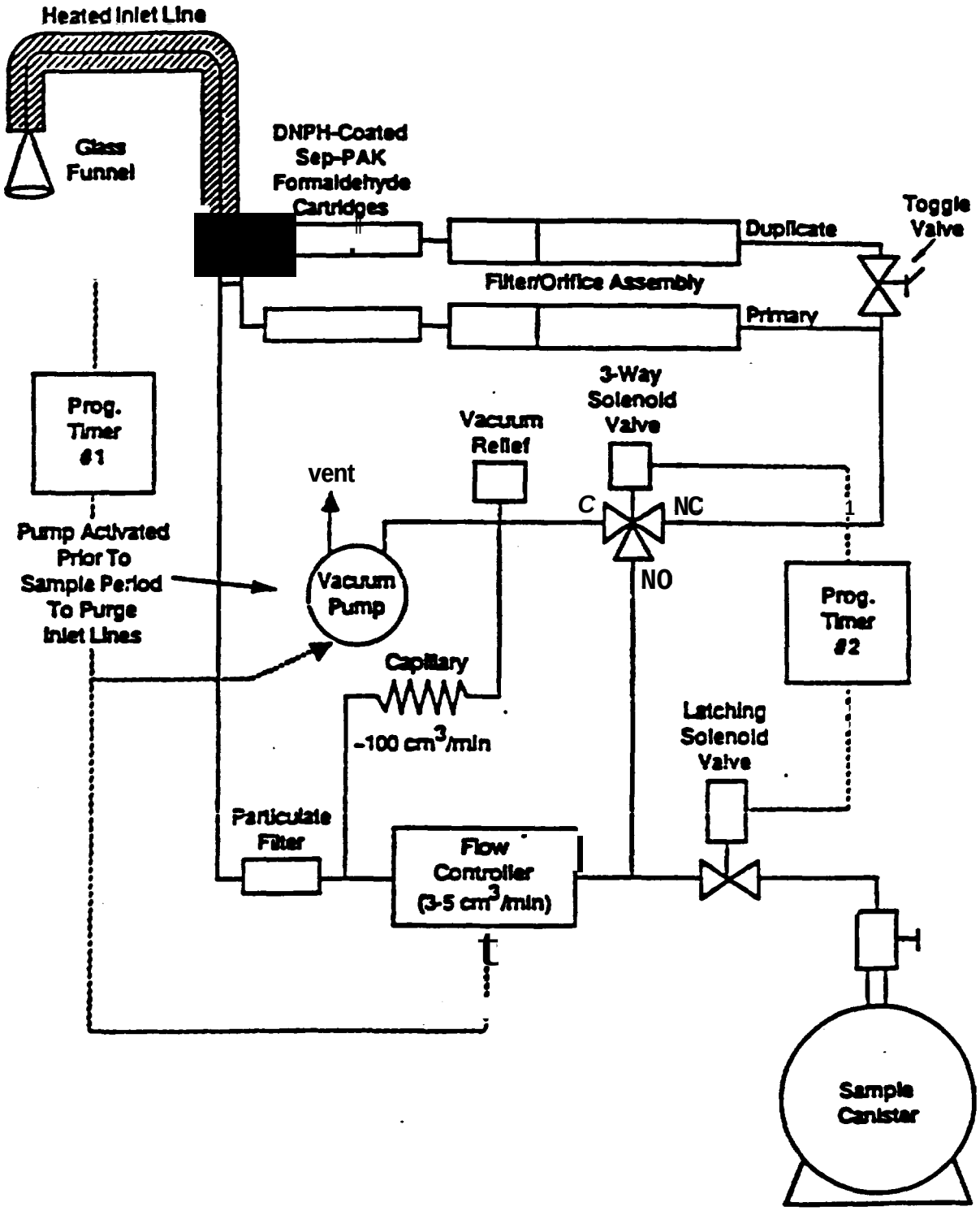


FIGURE GI. U.S. ENVIRONMENTAL PROTECTION AGENCY UATP SAMPLER SCHEMATIC OF SAMPLE INLET CONNECTIONS

APPENDIX N

PROCEDURES FOR SLUG TESTING

PROCEDURES FOR SLUG TESTING

Objective

The objective of these procedures is to define the requirements for conducting and analyzing slug tests.

Background

Slug testing is a rapid and easy way to estimate the hydraulic conductivity and transmissivity of an aquifer. An advantage of slug testing over pump testing in most remedial investigations is that little or no contaminated water requiring containment and disposal is produced.

Slug testing is accomplished by adding (or removing) a known volume to (or from) the well or piezometer to create a rapid rise (or fall) in water level. Water levels are then measured as the well returns to the static water level. The most common practice is to use a weighted cylinder of known volume or a pneumatic system. A bailer may be used in place of a slug or weight under low-recharge aquifer conditions. The rate of water-level recovery is then measured using a pressure transducer and data recorder or a water-level meter and stopwatch (the former method is preferable in most environments). Data, as depth-time pairs, are then graphed and used in equations to determine hydraulic conductivity and aquifer transmissivity.

Procedures for Falling-Head Slug Test

Before Slug Testing:

1. Place plastic sheeting around the wellhead. Arrange needed equipment and decontamination materials on the sheet.
2. Put on personnel protective clothing, as specified in the site-specific health and safety plan.
3. Open the locking and vented caps and inspect the wellhead. Note in particular the condition of the surveyed reference mark, if any.
4. Measure and record the static water level and the depth to the bottom of the well. Record these data in the appropriate logbook.

During Slug Testing:

5. If a transducer and analog or digital recorder are to be used to measure water-level changes, and, if necessary, calibrate the transducer at two different depths in the well. Calibration depths should be widely separated. Leave the transducer at the lower calibration point.
6. Switch on the data recorder, or set the water level meter probe near the level at which water is expected to rise.
7. Rapidly lower the float or slug into the water.
8. Record fall in water level versus time. If a transducer and recording device are used, record depth at predetermined intervals for the first 2 minutes or longer, based on aquifer conditions. If using a water-level indicator and chronometer, record depth-time data at the fastest rate possible for the first 5 minutes of well recovery. Subsequent recording intervals may be adjusted to suit the rate of well recovery.
9. Continue recording depth-time data until the well has recovered to nearly the static water level. When using data recorders, it is advisable to check and record the reading every few minutes to ensure that data are being properly recorded.

After Slug Testing:

10. Record the time of test completion in the logbook. If a data recorder with random access memory (RAM) or erasable programmable read only memory (EPROM) was used, record the file name used.
11. Decontaminate all equipment according to Appendix O. Clean up the site, and close and lock the well before leaving. Take contaminated plastic sheeting and disposable protective clothing to a designated disposal container.

NOTE: Both rising- and falling-head slug tests may be *carried* out in the same operation by first measuring the rate of water-level fall immediately after slug insertion, then measuring the rate of water-level rise after slug withdrawal. Be sure that the well has recovered to the static water level before conducting the rising-head test.

Procedures for Rising-Head Slug Test

Before Slug Testing:

1. Lower a clean float or solid slug of known volume into the well until it is floating or fully submerged. Allow the well to re-equilibrate to static water level.
2. Turn on the data recorder, if used, or verify that static water level has been re-established with a water-level meter.

During Slug Testing:

3. Withdraw the float or slug quickly, but avoid surging. Record the time of withdrawal to the second. *Start* the stop watch, if used, at the instant the slug is withdrawn.
4. Record the rise in water level versus time. If a transducer and recording device are used, record depth at predetermined intervals for the first 2 minutes or longer, based on aquifer conditions. If using a water-level indicator and chronometer, record depth-time data at the fastest rate possible for the first 5 minutes of well recovery. Subsequent recording intervals may be adjusted to suit the rate of well recovery.
5. Continue recording depth-time data until the well has recovered to nearly the static water level. When using data recorders, it is advisable to check and record readings every few minutes to ensure that data are being properly recorded.
6. Record the time of .test completion in the field logbook. If a data recorder with RAM or EPROM memory was used, record the file name used.

After Slug Testing:

7. Decontaminate all equipment. Clean up the site, and close and lock the well before leaving. Take contaminated plastic sheeting and disposable protective clothing should be taken to a designated disposal container. Dispose of water removed from the well in accordance with the site work plan.

Restriction/Limitations

In wells in which the static water level and water levels induced during testing are above the top of the screened or open hole interval. Both rising-head and falling-head tests should be conducted to provide a check of results.

Quality Control Requirements for Slug Tests

- A pressure transducer and data logger, or strip chart recorder, must be used to perform these tests.
- Slug tests shall be performed after groundwater sampling to minimize contamination.
- All equipment must be decontaminated before each test.
- If possible, both falling and rising head tests shall be performed.
- In monitoring wells where the static water level is below the top of the screened or open interval, falling-head tests shall not be performed. In these instances, rising-head tests shall be performed by the sudden removal of water.

APPENDIX *O*

DECONTAMINATION PROCEDURES

GENERAL DECONTAMINATION PROCEDURES

Nonsampling Equipment (e.g., drill rigs)

- Decontaminate with high pressure steam
- If necessary, scrub with a laboratory-grade detergent and clean water wash solution.
- Rinse with clean water as necessary.

Sampling Equipment (e.g., augers, drill pipe, split barrel samplers, sampling utensils, Teflon-coated stainless steel leaders, bailers, etc.)

- Prior to decontaminating the equipment, replace protective gloves.
- Wash and scrub with a laboratory-grade detergent and clean water wash solution or decontaminate with high pressure steam.
- Rinse with clean water.
- Rinse with ASTM Type III water.
- Rinse twice with isopropyl alcohol.
- Rinse with ASTM Type III water.
- Air dry.
- Wrap in aluminum foil for storage if the sampling equipment is not used immediately.
- Augers and drill rods will be wrapped in plastic after decontamination, if possible.

Personal Decontamination

- Personal decontamination procedures are outlined in the site-specific Health & Safety plan.

Decontamination Waste Disposal

- All wastes generated during decontamination shall be placed in 55-gallon drums. The drums will be labeled and stored in a designated area for future disposal.

APPENDIX P

CALIBRATION/STANDARDIZATION PROCEDURES

CALIBRATION/STANDARDIZATION PROCEDURES

Temperature Probe

All thermometers will be initially calibrated against an NIST certified thermometer or one traceable to NBS certification. Each glass mercury filled thermometer will be inspected before each field trip to ensure integrity. Temperature probes will be tested for calibration quarterly according to manufacturer's instructions. Thermometer calibration entails checking the unit versus an NIST certified thermometer. If the values obtained during this procedure do not fall within the specified ranges, the probe will be shipped back to the manufacturer for servicing.

Field temperature meters are checked daily/before use with field grade thermometers.

Specific Conductivity

The designed cell constant of the conductivity cell is given in the manufacturer's instruction book. The actual cell constant will be determined each day before use of the cell. Conductivity calibrator solutions are used to check the cell constant according to the procedure outlined in the instruction book. If the values obtained during the check are within specification, the measured error will be used to improve the accuracy of sample readings using the equation in the instructions. If accuracy is out of specification, the cell must be repaired by the manufacturer or replaced.

Each meter will be checked before each field trip to ensure that the probe is intact, batteries are sufficiently charged, and that the cell is clean. Calibration procedures entail checking the conductivity cell with one potassium chloride standard in the expected range of the sample(s) to be collected during the sampling day. If a conductivity meter which does not automatically compensate for temperature is used, the meter readings will be manually recalculated to account for temperature. All calibration procedures and calculations will be entered in the site log and on other pertinent field records.

pH Meter

The pH meter shall be checked before each field trip for any mechanical or electrical failures, weak batteries, and cracked or fouled electrodes. The pH electrode will be tested for noise and offset and a 2-point calibration performed each day in the field before use and quarterly in-house according to the method in the manufacturer's instruction book. The slope of the meter shall be checked initially using pH 4,7, and 10 buffer solutions. Once it has been confirmed that the electrode offset is functioning within specification, the calibration will be performed. Buffer solutions at pH 7.00 and 4.00 or 7.00 and 10.00 will be used, depending on the expected pH value of the samples to be analyzed. The calibration check process should be repeated between sampling locations using at least two buffer solutions. All calibration procedures and calculations will be entered in the Site Log and on other pertinent field records.

Photoionization Detector

The photoionization detector (PID) or equivalent instrument will be calibrated in-house quarterly using the calibration kit provided by the manufacturer and following the instructions in the user's manual. In addition, each PID/OVA used in the field will be checked daily/before use with the manufacturer supplied calibration *span gas.* The meter will be zeroed prior to transport to the area of concern in order to avoid bias due to ambient site conditions. To ensure peak performance of the instrument, routine maintenance consisting of cleaning the ultraviolet lamp window, replacing the dust filter and cleaning the exterior of the instrument is necessary. The lamp window is cleaned by disassembling the detector cell and lamp and wiping the window with a methanol-moistened tissue. This procedure should only be performed when a high span setting is insufficient to give an adequate readout. The dust filter of the instrument should be replaced when the sensitivity of the instrument increases by more than 10 percent when the filter is moved. The instrument should not be run for more than one minute without the filter in position. The exterior of the instrument will be cleaned quarterly by wiping with a damp cloth, using a mild detergent when necessary.

All field readings taken in ambient air will be corrected for ambient background. This correction is accomplished by manually subtracting the measured ambient PID reading from the metered response for an individual sample (i.e. boring headspace, split spoon sample). Under no circumstances will the meter be zeroed based on ambient conditions in the area of concern.

Background correction is not necessary for readings taken in closed vessels (i.e. glass jar soil headspace readings when using an aluminum foil lid seal).

The battery of the instrument will be recharged when the low battery indicator appear. The instrument should be allowed to discharge until the indicator appears and then it should be fully recharged. This will maximize the operating time of the instrument. Consistent charging before full discharge will diminish the operating time of the instrument. Charging instructions in the user's manual should be followed for the appropriate charger.

APPENDIX *Q*

RÉSUMÉS

SCOTT RYAN
GEOLOGIST

EDUCATION M.S., Geology, 1986
B.S., Geology, 1982

RELEVANT EXPERIENCE:

- **Project Geologist**
  Responsible for the planning and field implementation of RFI and UST studies at Naval facilities. Responsibilities include drilling supervision, soil sampling and geological characterization, monitoring well installation and groundwater sampling.

- Project consultant for a field investigation for groundwater exploration in the Kalahari Desert, Gaborone, Botswana, Africa. His responsibilities included surveying, drilling supervision, and coordination with Bostwanan authorities.

- Groundwater protection regulatory analysis with the Connecticut Department of Environmental Protection. Responsibilities included technical review and enforcement at hazardous waste sites and sanitary landfills.

- Experience in RCRA groundwater protection standards and implementation of those standards in an industrial context.

ALLISON LEIGH DENNEN

GEOLOGIST

EDUCATION: M.S., **Geology,** Memphis State University/1990

B.S., Geology, Memphis State University/1986

CERTIFICATIONS: Registered Geologist TN0788

RELEVANT EXPERIENCE:

- Project Geologist
  Authored a Sampling and Analysis Plan for a Remedial Investigation at an industrial Wastewater Treatment facility for the U.S. Navy.

- Project Geologist
  Performed contaminated assessment for leaking UST site in Kingsville, Texas.

- Assistant Project Manager for tank closures, site investigations, and remedial activities for 16 underground storage tank sites. Coordinated two field geologists, drill crews, and sampling technicians. Assimilated raw field data and analytical data into final technical reports.

- Project Coordinator and Hydrogeologist for the Tellijohn Sanitary Landfill, LeSueur, Minnesota. Authored a work plan,

performed hydrogeologic investigation, authored a technical report with groundwater monitoring protocol and coordinated quarterly sampling at the landfill in accordance with Minnesota Solid Waste Rules.

- Hydrogeologist for the Northeast Otter Tail Sanitary Landfill, New York Mills, Minnesota. Authored a Groundwater Monitoring System Work Plan in accordance with Minnesota Solid Waste Rules.

- Project Geologist for monitoring well installations and soil boring investigation, Laidlaw Sanitary Landfill, Terre Haute, Indiana.

- Project Geologist for geophysical and subsurface investigation at an archaeological site in Keokuk, Iowa.

- Project Geologist
  Designed a corrective action plan for an underground storage tank site, Bingham Lake, Minnesota.

- Project Geologist for the Motley By-Pass Superfund Site, Minneapolis, Minnesota. Performed hydrogeologic investigation to delineate the extent of contamination.

- Performed numerous Phase 1 site assessments for real estate transactions.

PUBLICATIONS

Dennen, A. L., *et al.,* 1991, An Investigation of Loess Craters on the Bluffs of the Mississippi River, Northwestern Tennessee: Geological Society of America Abstracts with Programs, vol. 3, no. 4.

Doran, F. J., Dennen, A. L., *et al.,* 1992, Investigation and Remediation of a Superfund Site in Minneapolis, Minnesota: Geological Society of America Abstracts with Programs, vol. 24, no. 3.

DOUGLAS C. PETTY

EDUCATION:

B.S., Industrial Hygiene, University of North Alabama, 1991

RELEVANT EXPERIENCE:

- Conducted industrial hygiene surveys for various industrial facilities.
- Knowledge of DOT and UN rules and regulations regarding the containerization and international shipping of hazardous and special materials.
- Performed hazardous waste determinations for materials stored and shipped from Naval Station Roosevelt Roads, Puerto Rico.
- Utilized NFPA and HMIS criteria for classification of commonly used chemical substances.
- Developed a new Material Safety Data Sheet (MSDS) program for a major chemical formula blender and packager.
- Developed health and safety plans for Naval Air Station (NAS) Memphis, NAS Kingsville, NAS Corpus Christi, and NAS Pensacola.
- Knowledge of USEPA Hazard Ranking System for NPL determination at various military installations.

HENRY H. BEIRO, SENIOR GEOLOGIST

EDUCATION: B.S., Geology, 1981
B.S., Biology, 1980

CERTIFICATION: 1989, Professional Geologist TN0990

EXPERIENCE:

- Completed a $1.5 million project at the Y-12 weapons plant in Oak Ridge, TN involving soil borings, groundwater well installation, and sampling.

- Reviewed and wrote Quality Assurance/Quality Control documents, work plans and field sampling plans for military installations in the U.S. with Martin Marietta Energy System's Hazardous Waste Remedial Action Program (HAZWRAP).

- Designed the Tuskegee, AL Landfill, installed monitoring wells and trained operators.

- Conducted and managed field gas chromatography for soil gas investigations. The most recent was Phelps Collins ANG, Alpena, MI, in October 1991.

- Managed and supervised core drilling, rotary drilling, auger drilling and churn drilling operations at various sites throughout southeast.

JAMES N. SPEAKMAN, Ph.D., P.E.
VICE PRESIDENT; ENGINEERING

EDUCATION: Ph.D., Environmental and Water Resources Engineering, Vanderbilt University/1971
M.S., Sanitary Engineering, Vanderbilt University/1968
B.S., Civil Engineering, Tennessee Technological University/1966

EXPERIENCE:

- Program Manager for Southern Division Naval Facilities Engineering Command Comprehensive Long-term Environmental Action, Navy (CLEAN) District II environmental services contract. The CLEAN District II contract encompasses DOD implementation of CERCLA; compliance with RCRA; underground storage tank program implementation; asbestos abatement; oil and hazardous substance spill contingency planning; and spill response management. The program has a 10-year duration and a $100 million maximum budget.

- Manager of Remedial Investigation/Feasibility Study (RI/FS) projects at National Priorities List (NPL) sites in Olaton and Island, KY. Each site is being investigated for potential air, surface water, soil and groundwater releases of ammonia, metals, sulfides, ammonia and chlorides.

- Directed numerous environmental compliance projects involving multi-disciplinary teams. He has managed six open-end hazardous waste compliance contracts for the U.S. Navy, involving more than 40 installations in eight states, Puerto Rico, Bermuda and Iceland. Those contracts included preparation of facility closure plans; sampling, analysis and data interpretation; negotiating "how clean is clean" with regulatory officials; closure plans and specifications and closure certifications.

- Performed audits of air emissions, industrial discharges, hazardous wastes, underground storage tanks and hazard communication programs for steel mills, aluminum mills and recycling plants, auto parts fabricators, concrete plants,

aggregate mines, plastic formulation plants, forges and wood milling plants.

- Directed underground tank assessment/closure/removal/remediation projects involving benzene, acetone, toluene, naphtha, and petroleum fuels at Santa Ana, CA; Charleston, SC; Lawrenceburg and Memphis, TN; Dallas, TX, and Yorktown, VA. Projects involved removal of as many as six tanks concurrently.

- Author and certifying engineer for numerous oil spill prevention, control and countermeasures plans, including US Navy installations in KY, OH, MS, TN, VA, Bermuda, Iceland and Puerto Rico.

- Certifying engineer of record for closure of hazardous waste facilities, including container storage, treatment and storage tanks and surface impoundments in FL, GA, SC and TN. Prepared RCRA closure plans for additional facility types, including landfills, land treatment units and "miscellaneous units" (specifically military ordnance open burning/open detonation units).

- Manager of PCB spill investigation, Work/Sampling/Health and Safety Plan preparation, negotiations with TN Department of Health and Environment, and supervision of remediation.

- Site manager, field investigation, immediate removal, groundwater monitoring at inactive HW Lagoon, Western MI.

- Author of Feasibility Study Work Plan for American Creosote Works NPL site in Jackson, TN.

- Supervised preparation of engineering plans and specifications for closure of hazardous waste surface impoundments at NAS Jacksonville (2 units), and NAS Pensacola (3units) and MCLB Albany, GA. Closure-in-place designs included various cap configurations, meeting or exceeding USEPA impermeability requirements, while providing for subsequent beneficial use of the surfaces.

- Directed RCRA permanent status permit applications for the Lake City (Independence, MO), Holston (Kingsport, TN) and Milan, TN Army Ammunition Plants. Those facilities operate multiple hazardous waste storage and treatment facilities, i.e. container storage buildings, surface impoundments, waste piles, incinerators, and treatment units; handling corrosive, reactive (including explosive), ignitable and toxic hazardous wastes.

PAUL V. STODDARD, P.G., VICE PRESIDENT, GEOLOGICAL SERVICES

EDUCATION: M.S., Geology, Memphis State University/1983
B.S., Geology, Memphis State University/1982
B.S., Biology, Memphis State University/1980

CERTIFICATION: Professional Geologist, **TN,** AR
Pending Certification, IN

EXPERIENCE:

- Generated stratigraphic correlations of upper Cretaceous and Tertiary trends of South Texas with concentration in the Wilcox and Frio formations. Responsible for regional correlations of E-logs, preparation of stratigraphic cross sections, development and updating of structure maps, and well spotting and digitizing.

- Conducted remedial site investigations at facilities for sites with potential soil and/or groundwater contamination involving chlorinated hydrocarbons and petroleum products. Field assessments included in-situ monitoring of organic vapors utilizing an organic vapor detector and/or a scanning infrared spectrophotometer.

- Responsible for the design and implementation of **"preconveyance"** investigations to determine potential soil contamination at various sites being considered for commercial development.

- Field supervision and implementation of closure plans for hazardous waste facilities at Charleston, SC Naval Shipyard. Tasks included removal of hazardous waste inventories, decontamination of tanks, confirmation sampling of decontamination solutions and soil sampling at container storage compounds operated by the Shipyard and Defense Reutilization and Marketing Office.

- Sampled groundwater monitoring wells for hazardous substance contamination and supervised well drillers at Shelby County, TN state Superfund site.

- Conducted a study of pesticide contamination in groundwater at an industrial facility in Missouri. Field Investigation included soil boring, monitoring well installation, determination of hydraulic gradients, data reduction and analyses, and report generation.

- Implemented groundwater investigation for photosensitive hazardous substance contamination at NPL site.

- Field supervision and implementation of closure plans for removal of underground waste oil storage tanks and investigation of potential contamination in soils and groundwater from tank releases.

- Field supervision and implementation of sampling plan for hazardous waste facilities at NAS Memphis. The task included Level B inspection of former waste plating treatment storm sewer, and the sampling of soils associated with defective joints. The task also included the sampling of a former salvage yard for petroleum hydrocarbons and lead.

- Field supervision and implementation of underground storage tank removal investigations. Field assessments include soil sampling and/or monitoring well installation with groundwater sampling and assessment of hydrogeologic conditions.

- Project geologist for interior survey of 120-acre underground room and pillar mine. Additional tasks included surface investigation of karst features, monitoring well installation, interpretation of borehole geophysics, and subsequent groundwater sampling and data reduction.

- Project geologist for hydrogeologic assessment of RCRA facilities. Tasks include site selection and installation of groundwater monitoring networks, slug tests, and subsequent data evaluation.

- Project geologist for MCAS Cherry Point, RFI. Tasks included design and implementation of Field Sampling Plan, including soil borings, monitoring well installation, groundwater sampling, slug tests, hydrogeologic characterization — including diurnal and tidal influences on the shallow aquifer and subsequent data reduction and report generation.

- Project geologist for hydrogeologic assessment of a proposed Tennessee state Superfund site. Investigation included step drawdown and constant rate aquifer pump test for design and installation of a groundwater treatment system.

- Project manager for CERCLA Remedial Investigation/Feasibility Study for a former pesticide manufacturing facility. Tasks included the design and implementation of geologic/hydrogeologic assessment for volatile organic and pesticide contamination, subsequent data reduction, and report generation. Project management included cost tracking, scheduling, and continued regulatory compliance (i.e. Administrative Order).

- Project manager for CERCLA RI/FS for an industrial manufacturing facility. Related tasks included the design and implementation of geologic/hydrogeologic assessments for chlorinated solvent contamination in soils and groundwater. Investigation included monitoring well installations, soil vapor extraction, borehole and surface geophysics, and a constant rate aquifer pump test, with subsequent data reduction and interpretation and RI report generation. Project management duties included community relations, maintaining data quality objectives, scheduling, waste disposal, and meeting Administrative Order Requirements.

- Supervised and directed remedial investigations (RI) per CERCLA requirements at multiple NPL sites, Region III, Region IV, Region V and Region VI. Investigations included, but were not limited to, soil analyses, monitor and recovery well installation, sampling and analyses of groundwater, aquifer tests, borehole geophysics, surface geophysics and data interpretation, including preparation and presentation of assessment reports.

BRIAN E. CALDWELL, SENIOR HYDROGEOLOGIST

EDUCATION: M. S. Geology, Florida State University, 1992
B. S. Geology, Florida State University, 1984
B. A. Psychology, Mars Hill College, 1978

CERTIFICATIONS: Professional Geologist, State of Florida, License No. 1330
Professional Geologist, State of Tennessee, Reg. No. TN1937
Professional Geologist, State of Wyoming, (Pending Bd. Approval)

RELEVANT EXPERIENCE:

Project Management:

- Project manager for transition of NAS Pensacola Superfund project from previous consultants to E/A&H.

- Project Field Manager for characterization of 37 hazardous waste sites at NAS Pensacola, under the Navy's Installation Restoration Program.

- Managed installation and quarterly sampling of monitoring well network at a United States Naval industrial waste treatment facility in Pensacola, Florida, to ensure compliance with RCRA.

- Project Manager for three investigation/remediation environmental sites at Fort McClellan, Alabama, and 10 environmental sites at Tyndall Air Force Base, Florida.

- Managed design, installation, and sampling of a complex multiaquifer groundwater monitoring network and site assessment of a chlorinated semivolatile hazardous waste site in Warrington, Florida.

- Contributing author on RI/FS work plans for all hazardous waste sites at NAS Pensacola; principal author for RI/FS work plan for Operable Unit 10.

Hydrogeology:

- Geochemical and statistical analysis of water-quality data collected for statewide Ambient Groundwater Monitoring Program.

- Statistical and mathematical analysis of hydrogeologic and geological data with computer simulations of single and multiaquifer groundwater systems.

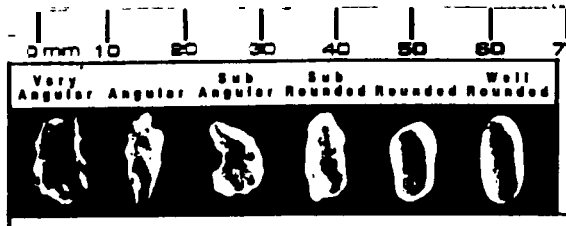
- Supervised design, installation, and sampling of monitoring well networks and conducted single and multi-well aquifer tests.

- Geophysical surveys using magnetometer, ground-penetrating radar, electromagnetic conductivity, and seismic refraction methods.

- Incorporated hydrogeological field and analytical results to site characterizations.

APPENDIX R

USCS SUMMARY



Geotechnical Gauge

Manuf. by:
W.F. McCollough
3101 Elkridge Ct.
Beltsville, MD 20705

MAJOR DIVISIONS			GRAPHIC SYMBOL	LETTER SYMBOL	TYPICAL DESCRIPTIONS	
COARSE GRAINED SOILS MORE THAN 50% OF MATERIAL IS LARGER THAN NO. 200 SIEVE SIZE	GRAVEL AND GRAVELLY SOILS MORE THAN 50% OF COARSE FRACTION PASSING NO. 4 SIEVE	CLEAN GRAVELS (LITTLE OR NO FINES)		GW	WELL - GRADED GRAVELS, GRAVEL - SAND MIXTURES, LITTLE OR NO FINES.	Light Brown
				GP	POORLY - GRADED GRAVELS, GRAVEL - SAND MIXTURES, LITTLE OR NO FINES.	
		GRAVELS WITH FINES (APPRECIABLE AMOUNT OF FINES)		GM	SILTY GRAVELS, GRAVEL - SAND - SILT MIXTURES.	Yellowish Orange
				GC	CLAYEY GRAVELS, GRAVEL - SAND - CLAY MIXTURES.	
	SAND AND SANDY SOILS MORE THAN 50% OF COARSE FRACTION PASSING NO. 4 SIEVE	CLEAN SAND (LITTLE OR NO FINES)		SW	WELL - GRADED SANDS, GRAVELLY SANDS, LITTLE OR NO FINES.	
				SP	POORLY - GRADED SANDS, GRAVELLY SANDS, LITTLE OR NO FINES.	Greenish Gray
		SANDS WITH FINES (APPRECIABLE AMOUNT OF FINES)		SM	SILTY - SANDS, SAND - SILT MIXTURES.	
				SC	CLAYEY SANDS, SAND - CLAY MIXTURES.	
FINE GRAINED SOILS MORE THAN 50% OF MATERIAL IS SMALLER THAN NO. 200 SIEVE SIZE	SILTS AND CLAYS	LIQUID LIMIT LESS THAN 50		ML	INORGANIC SILTS AND VERY FINE SANDS, ROCK FLOUR, SILTY OR CLAYEY FINE SANDS OR CLAYEY SILTS WITH SLIGHT PLASTICITY.	Olive Gray
				CL	INORGANIC CLAYS OF LOW TO MEDIUM PLASTICITY, GRAVELLY CLAYS, SANDY CLAYS, SILTY CLAYS, LEAN CLAYS.	
				OL	ORGANIC SILTS AND ORGANIC SILTY CLAYS OF LOW PLASTICITY.	
	SILTS AND CLAYS	LIQUID LIMIT GREATER THAN 50		MH	INORGANIC SILTS, MICACEOUS OR DIATOMACEOUS FINE SAND OR SILTY SOILS.	Light Gray
				CH	INORGANIC CLAYS OF HIGH PLASTICITY, FAT CLAYS.	
				OH	ORGANIC CLAYS OF MEDIUM TO HIGH PLASTICITY, ORGANIC SILTS.	
HIGHLY ORGANIC SOILS				PT	PEAT, HUMUS, SWAMP SOILS WITH HIGH ORGANIC CONTENTS.	Dark Gray

Inches 0 1 2 3 4 5 6

CLAY						
CLAY CONSISTENCY	THUMB PENETRATION	SPT, N BLOWS/FT.	Undrained Shear Strength (PSF) TORVANE	Unconfined Compressive Strength q_u Pocket Penetrometer		
VERY SOFT	Easily penetrated several inches by thumb. Exudes between thumb and finger when squeezed in hand.	< 2	250	500		
SOFT	Easily penetrated one inch by thumb. Molded by light finger pressure.	2 - 4	250 - 500	500 - 1000		
MEDIUM STIFF	Can be penetrated over 1/4" by thumb with moderate effort. Molded by strong finger pressure.	4 - 8	500 - 1000	1000 - 2000		
STIFF	Indented about 1/4" by thumb but penetrated only with great effort.	8 - 15	1000 - 2000	2000 - 4000		
VERY STIFF	Readily indented by thumbnail.	15 - 30	2000 - 4000	4000 - 8000		
HARD	Indented with difficulty by thumbnail.	> 30	> 4000	> 8000		

SAND					
SOILTYPE	SPT, N Blows/ft	Relative Density,%	FIELD TEST		
VERY LOOSE SAND	4	0 - 15	Easily penetrated with 1/2" reinforcing rod pushed by hand.		
LOOSE SAND	4 - 10	15 - 35	Easily penetrated with 1/2" reinforcing rod pushed by hand.		
MEDIUM DENSE SAND	10 - 30	35 - 65	Penetrated a foot with 1/2" reinforcing rod driven with 5-lb hammer.		
DENSE SAND	30 - 50	65 - 85	Penetrated a foot with 1/2" reinforcing rod driven with 5-lb hammer.		
VERY DENSE SAND	50	85 - 100	Penetrated only a few inches with 1/2" reinforcing rod driven with 5-lb hammer.		

Unified Soil Classification System (USCS)

	MILLIMETERS	INCHES	SIEVE SIZES
BOULDERS	> 300	> 11.8	
COBBLES	75 - 300	2.9 - 11.8	-
GRAVEL:			
COARSE	75 - 19	2.9 - .75	-
FINE	19 - 4.8	.75 - .19	1/4" - No. 4
SAND:			
COARSE	4.8 - 2.0	.19 - .08	No. 4 - No. 10
MEDIUM	2.0 - .43	.08 - .02	No. 10 - No. 40
FINE	.43 - .08	.02 - .003	No. 40 - No. 200
FINES:			
SILTS	< .08	< .003	No. 200
CLAYS	< .08	< .003	No. 200

9.1 Correspondence - documentation of state involvement