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FINAL QUALITY ASSURANCE/QUALITY CONTROL PLAN ENVIRONMENTAL SAMPLING
AND ANALYSIS WITH TRANSMITTAL LETTER KANSAS CITY MO
2/1/1996
TAPANAM ASSOCIATES



AIR FORCE BASE CONVERSION AGENCY

RICHARDS-GEBAUR AFB, MISSOURI

CAMERA COPY

QA/QC PLAN

ENVIRONMENTAL SAMPLING & ANALYSIS

FINAL SUBMITTAL

February 1996

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February 28, 1996

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**RE: Final QA/QC Plan for Environmental Sampling and Analysis, Richards Gebaur
Air Force Base, Belton, MO
Contract No. F41622-96-A-6503
Task: OIT**

Dear Ms. Valade:

Enclosed are two (2) copies of the Final QA/QC plan for all tasks listed in Task Table A and B included in the Statement of Work dated January 2, 1996.

Should you have any questions, please contact Eric Gorman or myself at (816) 444-5917.

Sincerely,

TapanAm Associates, Inc.

Siva Sivalingam, Ph.D.
Project Scientist



AIR FORCE BASE CONVERSION AGENCY

RICHARDS-GEBAUR AFB, MISSOURI

QA/QC PLAN

ENVIRONMENTAL SAMPLING & ANALYSIS

FINAL SUBMITTAL

February 1996

PROJECT # F41622-96-A-6503

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Section III - Laboratory QA/QC Plan

DRAFT

FIELD SAMPLING PLAN

RICHARDS-GEBEUR AIR FORCE BASE

F41622-96-A-6503

**PREPARED FOR
AIR FORCE BASE CONVERSION AGENCY
RICHARDS-GEBEUR AFB, MISSOURI**

February 1996

**PREPARED BY
TapanAm Associates, INC.**

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LIST OF ACRONYMS

AFB	- Air Force Base
AFBCA	- Air Force Base Conversion Agency
AFCEE	- Air Force Center for Environmental Excellence
BTEX	- Benzene, Toluene, Ethylbenzene, and Xylenes
CME	- Central Mine Equipment
DRO	- Diesel Range Organics
FID	- Flame Ionization Detector
FS	- Feasibility Study
FSP	- Field Sampling Plan
GC	- Gas Chromatograph
GRO	- Gasoline Range Organics
GSA	- General Services Administration
HSP	- Health & Safety Plan
HTW	- Hazardous and Toxic Waste
MS	- Matrix Spike
MSD	- Matrix Spike Duplicate
MSHA	- Mine Safety and Health Administration
NIOSH	- National Institute for Occupational Safety and Health
NTU	- Nephelometric Turbidity Units
NX	- "x" Design Flush Coupled Casing, 3-1/2" outside diameter, 3-3/16" inside diameter
PA	- Preliminary Assessment
PAH	- Polynuclear Aromatic Hydrocarbons
PCB	- Polychlorinated Biphenyl
PEST	- Pesticide
PID	- Photoionization Detector
PVC	- Poly Vinyl Chloride
QA/QC	- Quality Assurance/Quality Control
RA	- Remedial Action
RCRA	- Resource Conservation and Recovery Act
SI	- Site Investigation
SOW	- Statement of Work
SVOC	- Semi-Volatile Organic Compounds
TO	- Task Order
TRPH	- Total Recoverable Petroleum Hydrocarbons
TVH	- Total Volatile Hydrocarbons
USCS	- Unified Soil Classification System
VOC	- Volatile Organic Compound

1.0 INTRODUCTION

The Statement of Work (SOW) for this Environmental Sampling and Analysis Blanket Purchase Agreement includes the sampling and laboratory analysis of soil, surface water, ground water, suspected hazardous waste, and other environmental media and the installation and development of monitoring wells for Operation Location Q. This Field Sampling Plan (FSP) has been prepared by TapanAm Associates on behalf of the Air Force Base Conversion Agency (AFBCA) and Richards-Gebaur AFB (Figure 1) to provide sampling and laboratory analysis to confirm or refute the presence of harmful levels of contamination. Work assignments will be designated as a "Call" and will be assigned by the AFBCA.

2.0 SAMPLE DESIGNATION

A unique sample numbering system will be used to identify each sample for chemical or field screening analysis. In addition, this sample numbering system will be used to identify trip blanks, field blanks, equipment blanks, field duplicates and QA/QC. Each unique sample number will consist of the components described below.

The first 3-letter designation will be used to identify the Richards-Gebaur Delivery Order 0003 field samples:

TO1 = Richards-Gebaur Task Order 001

Each sample type will be identified by an alpha-code. The alpha codes for sample type are as follows:

SS = soil sample

GW = ground water sample

Each sample location will be identified by an alpha-code corresponding to the sample type, followed by a 2- or 3-alpha/digit sample location number, as appropriate. The alpha codes are as follows:

SB = soil boring

MW = monitoring well

EP = excavation pit

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The following codes will be used for QC samples and will be added as prefixes to the sample location:

TB = trip blank

FB = field blank

EB = equipment blank

MS/MSD = matrix spike/matrix spike duplicate

NOTE: Field duplicates will be utilized but not identified to the laboratory.

The sampling depths will be designated by the depth of the bottom of the 2-foot sample interval (i.e., a soil sample from a depth of 8 to 10 feet would be given the suffix "10").

The following are examples of the sample numbering to be used during the project:

TO1-GW-MW07: Richards-Gebaur T.O. 001, ground water sample collected from monitoring well 07.

TO1-SS-SB03-10': Richards-Gebaur T.O. 001, soil sample collected from soil boring 3 at a depth interval of 8 to 10 feet

TO1-SS-MW03-4': Richards-Gebaur T.O. 001, soil sample collected from monitoring well 03 during installation at a depth interval of 2 to 4 feet.

A field sketch showing sample locations on site will be drawn following sample collection.

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3.0 SAMPLING EQUIPMENT AND PROCEDURES

3.1 SURFACE SOIL SAMPLING METHODS (CONFIRMATORY & MONITORING)

Soils will be collected through the use of hand augers and trowels. The surface soils to be sampled must be exposed prior to sample acquisition. If the sample is to represent a discrete interval of 6 inches or greater depth, the overlying soils may be removed with a shovel or hand auger.

3.1.1 Hand Auger

In general, hand-operated augers are useful for sampling all types of soil/sediments except cohesionless materials below the water table and hard or cemented soil/sediment. Hand auger samples will be collected as follows:

1. Attach the auger bit to a hand auger extension, and further attach the "T" handle to the auger extension.
2. Begin auguring, periodically removing accumulated soil/sediment.
3. After reaching the desired depth, slowly and carefully remove auger from boring and fill volatile organic compound (VOC), total recoverable petroleum hydrocarbon (TRPH), and semi-volatile organic compound (SVOC) sampling jars immediately.
4. Remove soil cuttings and place into a precleaned stainless steel bowl.
5. Care should be taken to avoid scraping the borehole sides if sampling different interval is desired. If the wall of borehole collapses, an adjacent hole can be dug for the next deeper sampling interval.
6. Homogenize the soil as applicable, place the sample in the appropriate containers, and cap.

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3.1.2 Scoop/Spade/Shovel

This is the simplest and most direct method of collecting soils. This method can be used in most soil/sediment types but is limited somewhat to sampling near the surface.

1. Carefully remove the top layer of soil/sediment to the desired sample depth with a precleaned spade.
2. Using a precleaned stainless steel scoop or trowel, remove and discard a thin layer of soil/sediment from the area that comes in contact with the shovel.
3. Transfer the sample into an appropriate sample bottle with a stainless steel spatula, spoon, or equivalent and cap container.

3.2 **SEDIMENT SAMPLING**

The water content of the sediment may vary greatly. Likewise, the sediments themselves may range from very soft to dense. It may be necessary to use a variety of equipment to obtain the required samples, even at a single site. Equipment may include:

1. Stainless steel, Polytetrafluoroethylene (PTFE), or PTFE-lined sampling tray or bowl.
2. Stainless steel or PTFE dip sampler, scoops, trowels, spoons, ladles.
3. PVC pipe, 2 in. diameter.
4. Hand core sediment sampler, liners (optional) and extensions.
5. Sample bottles.

3.2.1 Hand Corer

The following steps will be used in collecting a sediment sample using a hand corer:

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1. Ensure that the corers and (optional) liners are properly cleaned.
2. Force the corer into the sediment with a smooth continuous motion to a depth of approximately 9 inches.
3. Twist the corer to detach the sample; then withdraw the corer in a single smooth motion.
4. Remove top of corer and decant excess water.
5. Remove the nosepiece and deposit the sample onto a stainless steel, PTFE or PTFE-lined tray.
6. Transfer the sample into sample containers using a stainless steel laboratory spoon (or equivalent device). The transfer equipment may be disposable to avoid decontamination costs, and the risk of cross-contamination.
7. The top 6 inches of the core will be sampled into 3 separate containers - 2 inches per container - to ensure that an accurate chronology of contamination can be determined. If specific data quality objectives mandate, the sample may be homogenized in bowl using sampling spoon, then samples will be placed in containers, preserved (as required), and packed on ice.

3.2.2 Scoop/Trowel/Spoon/Ladle

The following steps will be used to collect a sediment sample using the above devices:

1. Insert the sampling device into the material at the selected point and slowly remove the sample. Care should be taken to retain as much of the clay component as possible.
2. Transfer the sample into the appropriate container, add preservative as required, cap the container, and place in ice chest.

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3.3 SUBSURFACE SAMPLING METHODS

Drilling will be performed using a truck-mounted drill rig or direct-push hydraulic probe unit. The choice of drilling methods is influenced by two main factors: (1) the need to minimize the introduction of foreign materials that may influence the results of analysis and (2) the need to penetrate diverse geologic materials.

In unconsolidated materials, environmental borings will be advanced using 3 1/4-inch to 6 1/4-inch inside diameter hollow stem augers or 2-inch diameter probe rods. Soil samples will be collected utilizing a split-spoon sampler, CME (Central Mine Equipment) continuous sampler, or Geoprobe Systems® Probe-Drive System sampler. In the event that a continuous or semi-continuous rock unit is encountered, mud rotary drilling may be employed to advance a boring for the installation of a monitoring well. Rock samples will be collected using an NX size core barrel. No oils or lubricants will be used on the drill rods, augers, sampling, or other equipment used in drilling and sampling the borings.

Each boring will be continuously sampled and logged by an experienced TapanAm field geologist or engineer using a standard Hazardous and Toxic Waste (HTW) Drilling Log, MRK Form 55 (June 1989), as shown in Figure 2. Information recorded on this log will include boring location, drilling and sampling method, sampling interval, and sample descriptions using the Unified Soil Classification System (USCS). A copy of the USCS is shown in Figure 3. Unusual characteristics observed during drilling activities, such as discoloration of soil, odors, or air monitoring results, will also be noted on the drilling log.

A description of the borehole drilling, sampling, and logging methods and procedures is presented below.

3.3.1 Description of Drilling Methods

3.3.1.1 Hollow-Stem Auger Drilling

Hollow-stem auger drilling uses interconnected hollow auger flights equipped with a cutting head. The screw action of the augers as they are rotated and pressed into the ground pulls the soil cuttings to the surface. The bottom of the auger flight is fitted with a pilot bit and center

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plug attached to drill rods which prevent material from entering the augers during drilling. Soil samples are collected by removing the drill rods and center plug and advancing a sampler ahead of the augers.

3.3.1.2 Air Rotary Drilling

Air rotary drilling uses a rotating rock bit attached to drill rods. Air is used as the drilling fluid to lift rock cuttings from the borehole as the bit is advanced. Equipment needed for air rotary include a large air compressor, a swivel hose assembly connected to the top of the drill pipe or kelly, and a rock bit (i.e., tricone, roller type). Air is forced down through the center of the drill pipe and exits through small openings at the bottom of the drill bit. The cuttings are lifted along the annular space of the borehole, forced out the top of the borehole, and deposited on the surface. Air rotary allows cuttings to be removed rapidly, increases penetration rates, and extreme cold does not impede drilling operations. This method of drilling can only be performed in consolidated or semi-consolidated materials.

3.3.1.3 Direct-Push Drilling

Direct-push drilling uses a hydraulic probe unit to advance ¾-inch to 2-inch diameter probe rods. Soil or ground water samples are collected using specialized samplers attached to the rods and advanced to the desired sampling depth.

3.3.1.4 Borehole Abandonment

The borings will be backfilled to the ground surface upon their completion. The purpose of the backfilling procedure is to prevent foreign materials from entering the boring and possibly contaminating the ground water, and to prevent cross contamination between two separate water bearing zones.

The boreholes will be backfilled with a 94 parts Portland cement and 3 parts bentonite (94:3 cement-bentonite grout) via a tremie pipe to within two feet of the ground surface. The top two feet of the borehole will then be backfilled with soil. These procedures are in accordance with Missouri Department of Natural resources (MDNR) Test Hole Construction and Plugging Code 10 CSR 23-6.050.

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In areas where the boreholes penetrate existing pavement (concrete or asphalt), the backfilled boreholes will be capped with like material. The caps will have a minimum thickness equivalent to the surrounding concrete or asphalt.

3.3.1.5 Plugging of Monitoring Wells

The purpose of well abandonment is to eliminate vertical fluid migration along the borehole. Therefore, the preferred method of abandonment is the removal of the protective casing, riser pipe, and well screen, if possible. MDNR approval must be obtained to leave the casing in place.

The borehole will be backfilled with a 94:3 cement-bentonite grout via a tremie pipe to within two feet of the ground surface. The top two feet of the borehole will then be backfilled with soil. In areas where the boreholes penetrate existing pavement (concrete or asphalt), the backfilled boreholes will be capped with like material. The caps will have a minimum thickness equivalent to the surrounding concrete or asphalt.

If the borehole begins to collapse when removing the casing, the grout must be simultaneously emplaced while the casing is removed. When casing removal is not required or is not possible, then a three foot deep hole must be dug around the casing and the riser pipe cut off at that depth. These procedures are in accordance with MDNR Plugging of Monitoring Well Code 10 CSR 23-4.080.

The plugging or excavation of all monitoring wells must be reported on registration form supplied by MDNR. These forms, along with the fee, must be submitted within 60 days of the plugging.

3.3.2 Installation of Surface Casing

In the event that a surfacing casing is to be installed prior to completing a boring to the planned depth, the borehole will be overdrilled and an 8-, 10- or 12-inch PVC surface casing installed. Hollow stem auger or air rotary drilling will be used to overdrill the borehole for surface casing installation.

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The surface casing will be installed by filling the bottom one foot of the boring with grout, inserting the casing, and tremie grouting the annular space outside the casing. The grout will consist of approximately 94:3 cement-bentonite grout and will be placed in the annulus around the surface casing from the bottom to the surface using a side discharge tremie pipe. No more than 6.0 gallons of water per sack of cement will be used. The grout will be allowed to set for at least 48 hours and accumulated water removed using a sand pump prior to advancing the boring to the required depth.

3.3.3 Description of Sampling Methods

3.3.3.1 Soil Sampling

Many different types of soil samplers are available, and several different samplers may be used in a single boring. The type of sampler used will depend on the subsurface conditions and the sophistication of analyses required for the proposed laboratory testing program. A description of the types of sampling tools and procedures is provided in the following Sections.

Standard Split-Spoon Sampler -A split-spoon sampler is so named because the main section of the sampler consists of a section of pipe that splits into two pieces along the axis of the pipe. A driving shoe and waste barrel screwed to the ends hold the split sections together during driving. A diagram of the split-spoon sampler is shown in Figure 4, Split Spoon Sampler.

To collect a soil sample, a split-spoon sampler (outside diameter 2.0 inches; inside diameter 1.375 inches) is attached to 1 $\frac{5}{8}$ -inch "A" rod or larger drill rods. A soil sample is then obtained by driving the sampler into the soil. The sampler is driven by a 140-pound hammer free-falling a distance of 30 inches onto a collar on the drill rods. The sampler is driven a total of 24 inches into the undisturbed soil. The sampler, containing a soil sample, is then removed from the borehole. The end connections are removed and the split portion is pried open to reveal the sample. The sample is then identified and placed in airtight storage containers. Aids for sample retention, including catchers, spring or gravity traps (in the lower end), and check valves (in the top end) may be incorporated in a split-spoon sampler.

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CME Continuous Sampler - The CME continuous sampler is a 2½-inch I.D. five foot long split barrel. The sampler is attached to non-rotating drill rods mounted inside the hollow stem augers and is advanced along with the augers. The split barrel extends below the hollow stem augers and collects a relatively undisturbed soil core as the augers are advanced. A pilot bit and center plug are not used with the continuous sampler.

After advancing through the interval to be sampled, the split barrel is removed from the borehole without removing the hollow stem augers. The split barrel may be reused after emptying the soil core, or a different split barrel may be utilized so that drilling and sampling operations may continue. If soil sample recovery falls below 70%, the field engineer/geologist will switch to standard split spoon sampling.

Geoprobe Systems®Probe-Drive Soil Sampling System

The Probe-Drive System is a unique soil sampling system designed for use with the Geoprobe® hydraulic probe unit. Unlike split-spoon samplers, the Probe-Drive sampler remains completely sealed by a piston tip at the end of the sample tube while it is pushed or driven to the desired sampling depth. A piston stop-pin at the opposite end of the sampler is then removed, enabling the piston to retract into the sample tube while the sampler is driven to collect a sample.

Sampling Guidelines - Since the sampling techniques used in the field can influence the laboratory test results, the engineering analyses, and the validity of the resulting recommendations, care must be used to maintain sampling consistency from borehole to borehole. The following guidelines will be observed during sampling:

1. If the boring is too small, material from the side wall may be scraped into the sampler; therefore, the drill bit will be of sufficient size to allow free passage of the sampler as it is lowered to the bottom of the borehole.
2. The hole must be drilled to the last depth sampled before the next sample is taken. When the hole is open, the bit of the sampler (without predriving) will be at the last depth drilled. If a cave-in has occurred and/or cuttings have settled to the bottom of the borehole, this extraneous material will be removed by capturing it in the sampler and a clean sampler will be used to take the sample.

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3. The hole will not be drilled to the sample depth and left overnight before the next sample is taken. This procedure will be followed: (1) take a sample at the end of the day; and (2) at the beginning of the next day, drill to the next sample interval and take a sample. This procedure helps eliminate pressure release expansion of samples.
4. The geologist or field engineer will note the length of rods and bit or sampler in the hole. The boring depth is then calculated by subtracting the amount of "stick-up" from the ground surface from this total length. By constantly knowing the depth of the borehole, the depth of lithologic change indicated by changes in drill speed, the color of the drilling fluid, or other indications of strata change may be noted and properly logged.

3.3.3.2 Rock Coring

If bedrock is encountered in a soil boring before the planned depth of the boring is reached, the bedrock will be cored to the planned depth of the boring. If bedrock is encountered in a monitoring well boring, the rock will be cored to a depth of five feet below the static water level and the monitoring well installed.

To obtain a rock core, a carbide- or diamond-tipped bit is attached to the lower end of a core barrel. As the bit cuts deeper, the formation sample moves up the inside of the core tube. The rock coring will be performed using an NX size double tube core barrel. The double tube core barrel consists of an inner tube core recovery barrel and an outer barrel with a diamond bit. Potable water from the approved site source will be used as the drilling fluid during rock coring.

3.2.4 Sample Labeling

Improper sample labeling can result in misleading laboratory data. Therefore, each soil sample jar lid will be initially labeled with the sample identification, collection date and time, and sample depth using indelible ink.

Complete information for each sample selected for analysis will be written by the field geologist/engineer on a label affixed to the sample jars. The label information will include:

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(1) job number, (2) owner, (3) location, (4) boring (5) sample number, (6) depth, (7) date and time, (8) collector, and (9) comments. A description of the sample numbering designation system is provided in Section 2.0 of this FSP. Bottles for water samples will be labeled immediately after sample collection and after they have been wiped dry.

3.3.5 Sample Logging

The borehole logs will contain a detailed description of the soil strata encountered and pertinent information regarding drilling operations and estimated soil properties. Field sample data will be recorded in a bound log.

3.3.6 Soil Sampling for Chemical Analyses

The following procedures will be followed during soil sampling:

1. Set up the decontamination area, sample preparation area, and support area near the borehole location.
2. Decontaminate all equipment, samplers, and tools that will come in contact with the soil sample.
3. Inform the driller of the sample interval(s) for the borehole and oversee the sampling process.
4. Prepare and label the sample containers. Label the containers with the location, depth, date, and time of sampling.
5. Have the driller prepare the sampler for opening, but do not allow the driller to completely open the sampler.
6. Open the sampler slowly while it is lying on a clean sheet of plastic. As the sampler is being opened, the surface of the core should be screened with the photo ionization detector/flame ionization detector (PID/FID), with the probe of the instrument about one inch from the sample. Record the instrument readings in the log book.

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7. Obtain grab soil samples for VOC and TRPH diesel range organics and gasoline range organics (DRO and GRO) analyses first and place into 4-ounce septa sample jars. Completely fill the sample jars to minimize the loss of volatiles.
8. Sample for SVOC analysis will then be collected and placed into 4-ounce septa sample jars. Sample for RCRA metals will be collected last and placed into 8-ounce sample jars.
9. Log the core, recording percent recovery; color; texture; clay, sand, and gravel content; and other notable characteristics on the boring log. Sketch the confirmatory sample location.
10. Perform head space analyses on the soils. Record this reading in the appropriate place on the borehole log and in the field log book.
11. Deposit soil cuttings, wastewater, and waste generated during the decontamination process into 55-gallon 17 E/H steel drums for disposal after the results of chemical analyses of the soil and ground water at that location are known.

3.3.7 Sample Head Space Analysis

Head space analysis will be performed on each soil sample interval to provide information on volatile organic constituents in the soils. Soil samples will be collected continuously at 2-foot intervals to the depth of each borehole or to top of bedrock. A portion of the sample will immediately be collected for VOC analysis. Temporary labels will be placed on the sample containers and the containers placed into an iced cooler.

A portion of the remaining sample then will be placed loosely into a clean 16-ounce jar until it is approximately half full, the jar opening covered with aluminum foil, and the jar capped. The jar will be marked with the same identification number as the filled jars containing the portion of the sample for possible laboratory analysis, and placed in a warm location.

After a period of at least 15 minutes, the cap will be removed and the probe of a PID or FID will be pushed through the aluminum foil. The initial highest meter response will be recorded

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as the head space concentration. PID/FID instrument calibration will be checked at least every 10 analyses or daily, whichever is more stringent.

3.4 GROUND WATER SAMPLING

3.4.1 Monitoring Well Installation and Ground Water Sampling

3.4.1.1 Monitoring Well Drilling

The monitoring well borings will be advanced to a depth of approximately six feet below the first water-bearing zone using 6¼-inch inside diameter hollow-stem augers. A description of the hollow-stem auger drilling method is provided in Section 3.3.1. Each boring will be sampled and logged as described in Section 3.3.

If heaving sands are encountered or available information indicates the potential for heaving sands at a site, the monitoring well boring may be pre-drilled and sampled using smaller diameter augers. The boring will then be overdrilled to the planned depth of the monitoring well using 6¼-inch I.D. augers fitted with a PVC plug. The PVC plug will be knocked out prior to installation of the well. If necessary, non-chlorinated potable water will be introduced into the boring via the augers to increase hydraulic head and minimize the inflow and bridging of sand within the bottom of the augers. The potable water will be obtained from an approved source.

If bedrock is encountered prior to the planned depth for completion of the monitoring well boring, rock coring will be performed. Construction of monitoring wells is discussed in the following section.

3.4.1.2 Monitoring Well Construction and Completion

Overburden Wells: Overburden monitoring well borings will be advanced approximately six feet below the top of the upper water bearing zone. The monitoring wells will be constructed of 2-inch diameter Schedule 40 PVC riser pipe and screen (0.010 inch slot) with threaded joints (glued joints will not be used). The riser and a 10-foot section of screen will be installed through the augers with the screen set to straddle the water table. The augers will then be

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extracted as a filter pack consisting 20/40-grade silica sand is tremied from the bottom of the borehole to approximately 2 feet above the screened section. If the well screen and riser are installed in an open borehole, two centralizers will be placed on the well riser pipe (not on well screen) to keep the screen and riser centered in the borehole during completion of the well. A bentonite seal with a minimum thickness of 2 feet will be placed above the sand pack. The bentonite seal will consist of a high solids bentonite slurry and will be placed using a side discharge tremie pipe. The slurry will be allowed to set up per manufactures specifications prior to placing the cement-bentonite grout. A 94:3 cement-bentonite grout will be tremied in the annulus around the well casing from the top of the bentonite seal to the surface. No more than 6.0 gallons of water per sack of cement will be used. During installation, the depths of the well, sand pack and bentonite seal will be verified using a weighted tape. The monitoring wells will be capped and locked during any delays of field activities.

If the stable ground water level is at less than 12 feet, the well completion will be modified as follows:

1. Top of screen shall be at a minimum depth of 7.5 feet.
2. Filter pack sand shall extent approximately one foot above the screened section.
3. A bentonite seal consisting of a minimum 2 feet high solids bentonite slurry will be poured on top of the filter pack. The slurry will be allowed to set up for 8 hours prior to placing a minimum one foot cement-bentonite grout.
4. The remainder of the installation will be completed as described above.

If the stable ground water level is less than 8.5 feet or other conditions encountered prevent completion as specified above, AFBCA will be consulted on well completion specifics.

A protective steel collar with weep holes and a locking cap will be cemented in place over the PVC casing to prevent damage to the well. The protective collar will be seated in a concrete surface pad with approximate dimensions of 4 feet by 4 feet (or 48 inch diameter) by 4 inches thick. Three 2-inch diameter steel pipes, rising to a height approximately equal to the well, will be installed in concrete to further protect the well. The protective casing and the steel pipes will be primed and painted brown. Reflection tape will be placed on the painted pipes.

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Alternately, a flush mount protective cover may be installed. Information regarding the construction of each monitoring well will be recorded on a TapanAm Monitoring Well Information Sheet as shown in Figure 5.

Bedrock Wells: If a monitoring well boring reaches bedrock prior to encountering ground water, the rock will be cored and overdrilled to two feet below the bedrock surface and an 8-, 10- or 12-inch PVC surface casing installed. Hollow stem auger or air rotary drilling will be used to overdrill the borehole for surface casing installation. The surface casing will be installed as described in Section 3.3.2.

The boring will then be advanced at least five feet below the depth where static ground water is first encountered in the bedrock. The monitoring well will be constructed of 2-inch diameter Schedule 40 PVC riser pipe and 10-foot screen (0.010 inch slot) with threaded joints (glued joints will not be used). A filter pack consisting 20/40-grade silica sand will be tremied from the bottom of the borehole to approximately 2 feet above the screened section. A bentonite seal with a minimum thickness of 2 feet will be placed above the sand pack. The bentonite seal will consist of bentonite pellets if the seal is within the saturated zone. A high solids bentonite slurry shall be used in place of the pellets if the seal is placed above the saturated zone.

Dependent upon site requirements and subsurface conditions, certain wells may be installed uncased (without a well screen) within the bedrock. The boring will be cored and overdrilled to two feet below the bedrock surface and a 4-inch PVC casing installed. The boring will then be cored a minimum of 10 feet into bedrock and completed as an uncased well.

3.4.1.3 Monitoring Well Development

Monitoring wells will be developed no sooner than 48-hours after grouting is completed. During development, each well will be mechanically surged for 15 minutes followed by the purging of a minimum of three well casing volumes. Surging and purging will continue until the Ph, temperature, conductivity, and turbidity are stabilized; artifact-free formation water can be obtained; or for a maximum of 4 hours. To perform the stabilization test, the pH, temperature, specific conductance, and turbidity of the development water will be monitored. The wells will be considered developed when readings remain stable within plus or minus 10

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percent between three consecutive measurements and the turbidity remains within a 10 nephelometric turbidity unit (NTUs) range for at least 30 minutes. An example of the form to be filled out during well development is shown in Figure 6.

A minimum of three well volumes will be purged from each well during development. In addition, three times the volume of any water introduced into the well borehole during drilling and monitoring well installation (i.e. water loss during coring) will also be removed during development.

3.4.1.4 Monitoring Well Sampling

A minimum of one week will be allowed for a well to recover after development prior to collecting a ground water sample. The well will be sampled when a minimum of three well casing volumes have been purged from the well and ground water parameters remain stable within plus or minus 10 percent between three consecutive readings. An example of the form to be filled out during the stabilization test is shown in Figure 7.

Ground water samples will be collected using dedicated disposable Teflon[®] bailers, or by using either a submersible or bladder pump. The ground water samples will be transferred directly to laboratory-supplied sample containers. Containers for VOC samples will be filled such that no head space remains. Turbulence will be minimized during the transfer to prevent the loss of volatile organics. The sample containers will be labeled appropriately, stored in a cooler containing ice, and kept at approximately 4°C during storage and shipment to the laboratory. A full description of sampling procedures follows:

Sample Collection Using a Teflon[®] Bailer: The following steps will be used when collecting a groundwater sample using a Teflon[®] bailer:

1. On arrival at the well head, remove the locking and protective cap. Measure depth-to-water with an electric measurement tape (e- tape), and depth to bottom of the well to the nearest 0.01 foot, and record the values in the field logbook. An oil water interface probe will be used to check for immiscible hydrocarbons (gasoline, diesel, etc.) floating on the surface in the borehole at the time the water level measurements are made.

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2. Calculate the volume of water in the well to include the water in the filter pack. Begin purging the well and measure and record the temperature, pH, conductivity, and turbidity of each borehole volume during purging. The sample may be collected after three borehole volumes have been removed and the temperature, pH, and conductivity have stabilized within 10 percent for two consecutive readings. If these parameters do not stabilize the sample shall be taken after six borehole volumes have been removed. The sample will be collected after the water level has recovered to 80 percent of its static level or 16 hours after completion of purging, whichever occurs first. If a well is purged dry before three well volumes have been removed, the sample will be collected as soon as enough fluid has reentered the well.
3. To collect the sample, slowly lower the bailer into the water. Do not drop the bailer into the well as this may cause degassing of volatile organics. Allow about 30 seconds for the sample tube to fill. Slowly raise the Teflon® bailer to the surface.
4. Unscrew the cap of the sample container, being careful not to touch the lip of the bottle or the inside of the Teflon® liner. Avoid touching the mouth of the Teflon® bailer.
5. Unclasp the Teflon® bailer.
6. Pour the water from the bailer into the sample container slowly to prevent trapping any air bubbles (VOC Samples). Avoid splashing or agitating the water while the sample container is being filled.

Sample Collection Using a Submersible Pump: The following steps will be used to collect a sample from a well using a submersible pump.

1. On arrival at the well head, remove the locking and protective cap. Measure depth-to-water and total depth of the well and calculate purge volume. Check for immiscible hydrocarbons as described above.
2. Slowly lower the submersible pump into the well.

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3. Turn the power switch on to begin the pumping process. Do not handle energized power cords. If the pump does not work properly, as indicated by a lack of air flow out the discharge hose or by generator "lug" down, turn the switch off immediately. Wait a few seconds, then turn the switch on and off several times rapidly, finally pausing in the ON position to determine if the pump has started to function properly. Repeat this several times. If the sample pump still doesn't work, it needs repair. If the breakers or fuses on the generator disengage, an electrical short in the system is indicated, and repair is needed. Record problems in the field logbook.
4. Pump for a minimum of three well volumes, and check for stabilization of the pH, temperature, and specific conductivity. The sample may be collected after three borehole volumes have been removed and the temperature, pH, and conductivity have stabilized within 10 percent for two consecutive readings. If these parameters do not stabilize the sample shall be taken after six borehole volumes have been removed.
5. The sample will be collected after the water level has recovered to 80 percent of its static level or 16 hours after completion of purging, whichever occurs first. Measure the pH, temperature, and specific conductivity of the discharged water and record in the field logbook.

NOTE: If the well pumps dry while purging, it does not generally mean that a sample cannot be collected. A sample can still be obtained by following these steps after all other steps have been completed:

- a. When the well pumps dry, turn off the pump.
- b. Wait for the well to recharge sufficiently to draw a sample.
- c. Measure the depth-to-water using the electrical tape. Make sure that the water level is above the pump intake.
- d. Turn the pump on.

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- e. Measure pH, temperature, and specific conductivity once and record on the Ground Water Sample Report.
 - f. Collect the samples that are designated for collection with the pump.
 - g. The well may pump dry during the collection of samples. If this occurs, repeat steps (a.) through (d.) before collecting the remaining samples.
 - h. If sufficient water to sample has not recharged into well after a total of 16 hours, report the problem in the field logbook and discontinue sampling at that well.
6. Place sample numbers or description of the samples on the Ground Water Sample Report or field logbook as required and Chain of Custody forms.
 7. Fill the appropriate sample containers.

3.4.2 Ground Water Level Measurement

Following development of the monitoring wells, the water level in each well will be allowed to stabilize for a minimum of 48-hours prior to collecting a water level measurement. The depth to ground water and/or free product in each well will be measured to the nearest 0.01 foot using an oil/water interface probe or other electronic water level meter.

If free product is encountered in a monitoring well, the depth to the air/free product interface will be recorded to the nearest 0.01 foot from the top of the PVC casing. The probe will be advanced through the free product until the free product/water interface is encountered. This depth will be recorded to the nearest 0.01 foot from the top of the PVC casing. The thickness of free product will be determined by subtracting the depth to the air/free product interface from the depth to the free product/water interface. If only ground water is encountered, the depth to the air/ground water interface will be recorded to the nearest 0.01 foot from the top of the PVC casing.

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Water level/free product level measurements from the existing and newly installed monitoring wells on site will be obtained on the same day. If free product is detected in a monitoring well, this information will be provided to the Richards-Gebaur site representative personnel so that MDNR can be notified in accordance with MDNR and Federal regulations.

3.4.3 In Situ Ground Water Sampling

A Geoprobe Systems® or similar hydraulic probe unit is used to advance a slotted well point or specialized screen sampler fitted with an expendable drive point to the desired sampling depth. At the desired sampling depth the probe rod chain is withdrawn six inches from the expendable point to allow water to enter. A length of 3/8-inch Teflon tubing equipped with a stainless steel ball valve is then inserted into the probe rods from the ground surface to the bottom of the rods. The tubing is oscillated up and down to obtain a ground water sample.

3.5 SURFACE WATER SAMPLING

In general, and especially in areas where directly submerging the sample container is not feasible, the use of a sampling device, either disposable or constructed of a non-reactive material such as glass, stainless steel, or Teflon, is the most prudent method. A transfer device can be utilized in most sampling situations except where aeration must be eliminated (samples for volatile organic analysis) or where significant material may be lost due to adhesion to the surface of the transfer container. The device should have a capacity of at least 500 ml, if possible, to minimize the number of times the liquid must be disturbed, thus reducing agitation of any sediment layers.

A dipper, beaker with pour spout and handle, ladle, ice scooper, pond sampler, or other container constructed of inert material, such as stainless steel or Teflon, can be used to transfer water from the source to a sample bottle. This prevents unnecessary contamination of the outer surface of the sample bottle that would otherwise result from direct immersion of the sample bottle in the liquid. Use of this device also prevents the technician from having to physically contact the water. Depending upon the sampling application, the transfer vessel can be either disposed or reused. If reused, the vessel will be thoroughly decontaminated prior to sampling a different source.

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When surface water sampling is done in conjunction with sediment sampling, the water samples should be collected first.

3.5.1 Dipper or Other Suitable Sampling Devices

The following steps will be used to collect a surface water sample using the above mentioned devices.

1. Submerge a pre-cleaned stainless steel dipper or other suitable device with minimal surface disturbance. The mouth of the container should be facing upstream. The sampler, if wading, should remain downstream of the sample collection point. In addition, downstream samples should be collected prior to upstream samples. Care should be taken not to disturb bottom sediments. Allow the device to fill slowly and continuously.
2. Retrieve the dipper/device from the surface water with minimal disturbance.
3. Obtain water quality measurements as required.
4. Repeat steps 1 and 2 above, obtaining a new aliquot of the material to be sampled.
5. Samples for organics analyses should be collected first, followed by samples for other analyses. Remove the cap from the sample bottle and slightly tilt the mouth of the bottle below the dipper/device.
6. Empty the dipper/device slowly, allowing the sample stream to flow gently down the inside of the bottle with minimal entry turbulence.
7. Continue delivery of the sample. In general, for samples to be analyzed for organic constituents, the container should be completely filled with no air bubbles present. For other samples, fill the container almost completely, leaving adequate space to allow for expansion.

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3.5.2 Peristaltic Pump

If a medical grade silicone tubing is used in the peristaltic pump, the system is suitable for sampling almost any parameter including most organics. Some volatile stripping, however, may occur, and though the system may have a high flow rate, some material may adhere to the tubing. Therefore, pumping methods should be avoided for sampling volatile organics or oil and grease. It is necessary in most situations to change both the Teflon suction line and the silicon pump tubing between sample locations to avoid cross-contamination. This requires maintaining a sufficiently large stock of material to avoid having to clean the tubing in the field. Teflon tubing may be used as an effective substitute for that supplied with the automatic liquid waste samplers such as the ISCO Model 2100 and Manning Models S-3000 and S-4000. When sampling a liquid stream which exhibits a considerable flow rate, it may be necessary to weight the bottom of the suction line. The procedure for sample collection using a peristaltic pump are as follows:

1. Install clean, medical-grade silicone tubing in the pump head, according to the manufacturer's instruction. Allow sufficient tubing on the discharge side to facilitate convenient dispensation of liquid into sample bottles and only enough on the suction end for attachment to the intake line. This practice will minimize sample contact with the silicone pump tubing.
2. Select the length of suction intake tubing necessary to reach the required sample depth and attach to the intake side of the pump tubing. Heavy-wall Teflon, of a diameter equal to the required pump tubing, suits most applications. (Heavier wall will allow for a slightly greater lateral reach.)
3. If possible, allow several liters of sample to pass through the system, before actual sample collection. Collect this purge volume and then return it to the source after the sample aliquot has been withdrawn.
4. Obtain water quality measurements as required.

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5. Samples for organics analyses should be collected first, followed by samples for other analyses. Remove the cap from the sample bottle and slightly tilt the mouth of the bottle toward the end of the discharge tubing.
6. Fill the necessary sample bottles by allowing the pump discharge to flow gently down the inside of the bottle with minimal entry turbulence. In general, for samples to be analyzed for organic constituents, the container should be completely filled with no air bubbles present. For other samples, fill the container almost completely, leaving adequate space to allow for expansion. Cap each bottle as filled.

3.6 DECONTAMINATION

3.6.1 Personnel

Persons working on the site shall undergo decontamination before leaving the site. In most instances, removal of protective clothing will suffice for decontamination. Facilities for storage of reusable protective clothing and for the disposal of clothing contaminated beyond reuse will be constructed or placed on site. Facilities for decontaminating hands, boots, and gloves, consisting of a detergent wash and water rinse will also be provided.

Decontamination of personnel and miscellaneous small tools will be in accordance with the Site Health and Safety Plan.

3.6.2 Equipment

Precautions will be taken to prevent the potential transfer of contamination from one boring location to another during the field activities. Equipment used to advance and sample soil borings will be decontaminated prior to use at each boring location. This equipment includes but is not limited to the drill rig, augers, drill rods, soil and ground water samplers, and pumps. The following sections describe the decontamination procedures that will be used at the site.

3.6.2.1 Drilling and Soil Sampling Equipment

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The drill rig and other surface drilling equipment will be decontaminated prior to use at each site. Downhole equipment including reusable casing augers, rods, bits and related equipment will be decontaminated between the sampling locations. The procedure for decontaminating equipment will consist of manually scraping visible soil and mud from the equipment, applying a high-pressure low-volume hot water and Alconox® wash, and rinsing with potable water.

Soil samplers used to obtain soil samples for chemical or geotechnical analyses, along with head space analysis jars, knives, stainless steel trowels, spoons, and mixing bowls, will be decontaminated after each use according to the following procedure:

1. Scrub off a majority of the soil using potable water. Sampling equipment may also be washed by performing a high-pressure low-volume hot water and Alconox® wash of the disassembled parts.
2. Wash with a mixture of potable water and Alconox® detergent;
3. Rinse three times with potable water.
4. Rinse with a laboratory certified Type II Reagent-Grade Water.
5. Rinse equipment with pesticide-grade methanol.
6. Rinse equipment with pesticide-grade hexane.
7. Air dry on a clean elevated surface and then wrap in aluminum foil if not used immediately.

To facilitate the decontamination process, decontamination zones will be constructed. The decontamination zone for the soil samplers, water sampling tools, and miscellaneous small tools will be established near each borehole. The decontamination area will consist of a low-lying area covered with a 6-mil polyethylene sheet and several buckets, one dedicated to each decontamination step. At the completion of decontamination procedures at each boring, the debris will be enclosed in the polyethylene sheet and deposited into 55-gallon type 17 E/H drums for later disposal. The decontamination zone for the rear end of the drill rig, augers, rods, and other large items of equipment used during drilling and sampling will be established in the general vicinity of each sampling area. A low-lying area sloped toward a collection basin, will be covered with a 6-mil polyethylene sheet to collect the water and solid wastes generated during cleaning of the equipment. The water and the debris will be placed into 55-gallon 17 E/H drums for later disposal.

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3.6.2.2 Monitoring Well Development, Sampling, and Monitoring Equipment

Equipment used to surge and purge the monitoring wells during development and the probe used for water level measurements will be decontaminated using the following procedure prior to placement into a well:

1. Wash with a potable water and Alconox® detergent solution; and
2. Rinse three times with potable water followed by laboratory certified Type II Reagent-Grade Water wash.
3. Methanol and hexane may be used if particularly oily samples are encountered.

The instruments used to monitor development water during stabilization tests (i.e. pH, temperature, conductance, and turbidity meter) will be triple rinsed with distilled water only to minimize variance from calibration standards. If obvious signs of contamination remain, the instruments will be decontaminated, as described above, and then recalibrated.

Specific procedures for decontaminating the downhole pump and tubing follows:

1. Immerse the pump and tubing into a container of Alconox® detergent solution and pump a minimum of three pump and tubing volumes of the solution through the pump and tubing.
2. Immerse the pump and tubing into a container of distilled water. Pump a minimum of three pump and tubing volumes of distilled water through the pump and tubing.
3. Remove the decontaminated pump and tubing and place into a clean plastic bag/container for transport.

The outside of the pump will be decontaminated by washing with an Alconox® and water solution and triple rinsing with laboratory certified Type II Reagent-Grade Water.

Water samples from the developed wells will be obtained using one-time use dedicated Teflon® disposable bailers and nylon rope. A Teflon® bottom discharge device will be used to transfer the water sample from the bailer to the appropriate laboratory container(s).

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4.0 ENVIRONMENTAL SAMPLE HANDLING AND PACKING

4.1 SAMPLE CONTAINERS

Sample containers will be provided by the analytical laboratory. The containers will be either high density polyethylene or glass with Teflon®-lined lids and will be pretreated with preservatives as applicable. The type of container, minimum sample volume, preservation method, and holding time for each analytical method that will be used for this project are listed in Table 1.

4.2 SAMPLE HANDLING AND DECONTAMINATION

After sample collection in the field, the exterior of the sample containers will be decontaminated if gross contamination is present. The sample containers will be handled with gloves until decontaminated with a detergent wash and water rinse. Care will be taken to avoid damaging the temporary labeling during decontamination. After decontamination, permanent labels will be placed on clean sample container exteriors.

The sample containers will be well-cushioned with packing materials when they are placed in the insulated cooling chests for transportation to the laboratory. Care will be taken to seal bottle caps tightly. The samples will be shipped via overnight carrier to the laboratory to arrive no later than 48 hours after the time sampled.

4.3 PROCEDURES FOR PACKING AND SHIPPING LOW CONCENTRATION SAMPLES

Samples will be packaged as follows:

- Use water-proof metal (or equivalent strength plastic) ice chests or coolers only.

- After determination of specific samples to be submitted and filling out the pertinent information on the sample label and tag, put the label on the bottle or vial prior to packing.

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- Place about 3 inches of inert cushioning material such as vermiculite in the bottom of the cooler.
- Enclose the bottles in clear plastic bags through which sample tags and labels are visible, and seal the bag. Place bottles upright in the cooler in such a way that they do not touch and will not touch during shipment.
- Place bubble wrap and/or packing material around and among the sample bottles.
- Add sufficient ice (double bagged) between and on top of the samples to cool them and keep them at approximately 4°C until received by the analytical laboratory.
- Fill cooler with cushioning material.
- Put paperwork (Chain-of-Custody Record) in a waterproof plastic bag and tape it with duct tape to the inside lid of the cooler.
- Tape the drain of the cooler shut with duct tape.
- Secure lid by wrapping the cooler completely with strapping, duct or clear shipping tape at a minimum of two locations. Do not cover any labels.
- Attach completed shipping label to top of the cooler.
- Label "This Side Up" on the top of the cooler, "Up" with arrow denoting direction on all four sides, and "Fragile" on at least two sides.
- Affix numbered and signed custody seals on front right, and back left of cooler. Cover seals with wide, clear tape.

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4.4 PROCEDURES FOR PACKING AND SHIPPING MEDIUM CONCENTRATION SAMPLES

An effort will be made to identify samples suspected of having elevated contaminant concentrations based on field observations and screening test. These samples will be segregated and packed in a separate container to the extent allowed by prevailing field conditions. Medium concentration samples will be packed in the same manner as described in Section 5.3 for low concentration samples.

4.5 CHAIN-OF CUSTODY RECORDS

As part of the sampling plan, Chain-of-Custody protocols will be established to provide documentation that samples were handled by authorized individuals as a means to maintain sample integrity. The Chain-of-Custody form will contain the following information:

- Sample identification number;
- Date, time, and depth of sample collection;
- Sample type (e.g. soil);
- Type and number of container;
- Requested analyses;
- Field notes and laboratory notes;
- Project name and location;
- Name of collector;
- Laboratory name and contact person; and
- Signature of persons relinquishing or receiving samples.

A sample Chain-of-Custody form to be used during this investigation is illustrated in Figure 8, TapanAm Chain-of-Custody Form. The field sampler is personally responsible for the care and initiation of custody of the samples collected until they are transferred to the Sample Coordinator.

- Sample containers will be labeled/tagged with the sample numbers and locations. The date, time sampled, analyses to be performed and sample collector's signature also will be entered on the tag.

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Sample labels will be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a field logbook notation would explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.

Each sample container cap also will have an adhesive label on it indicating the sample number in the event the tag on the sample container becomes loose, or lids are inadvertently placed on the wrong sample container.

The Field Team Leader will review the field activities to determine whether proper custody procedures were followed during the field work, assess whether proper documentation was filled out, and decide if additional samples are required.

Chain-of-Custody records will be maintained for each laboratory sample. At the end of each day on which samples are obtained, and prior to the transfer of the samples off-site, Chain-of-Custody documentation will be completed for each sample. Information on the Chain-of-Custody form will be verified to ensure that the information is consistent with the information on the container labels and in the field log book.

Upon receipt of the sample cooler at the laboratory, the laboratory custodian will break the shipping container seal, inspect the condition of the samples, and sign the Chain-of-Custody form to document receipt of the sample containers. Information on the Chain-of-Custody form will be verified to ensure that the information is consistent with the information on the container labels. If the sample containers appear to have been opened or tampered with, this should be noted by the person receiving the samples under the section entitled "Remarks." The completed Chain-of-Custody records will be included with the analytical report prepared by the laboratory.

5.0 FIELD EQUIPMENT AND MEASUREMENTS

TapanAm field personnel will assemble the required field equipment prior to mobilization to the site. At this time, the equipment will be checked to ensure that it is in proper working order, and required maintenance will be performed. Tools and equipment that may be needed

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for field maintenance will be assembled at this time, and pertinent sections of the manuals will be photocopied for reference in the field.

Personnel will become familiar with the calibration of the instruments, as outlined in the respective manuals, and will make the calibrations that can be made at that time. Pertinent sections of the respective manuals will be photocopied for reference in the field, and the equipment that will be necessary for field calibration of the instruments, such as buffer solutions and calibration gases, will be assembled for mobilization to the site.

The following parameters will be measured on site during the field investigation:

- USCS soil and sediment classification, including color (Munsell), consistency, structure, mottling, layering, lenses, fractures, organic matter or voids.
- PID screening of VOCs.
- Ground water temperature, pH, turbidity, and conductivity.

Descriptions of the field equipment to be utilized in the field investigation are presented in the following sections.

5.1 PHOTOIONIZATION DETECTOR/FLAME IONIZATION DETECTOR

A PID or FID is a quantitative instrument that measures the total concentration of numerous organic vapors in air. The instrument to be utilized during the field investigation is an HNu PI101 photoionization detector or equivalent. The HNu is battery operated and lightweight, making it very useful in field monitoring projects. The instrument is calibrated by introducing pressurized gas from a cylinder with a known organic vapor concentration into the detector. Once the reading has stabilized, the display of the instrument is adjusted to match the known concentration. A calibration of this type is performed each day prior to using the instrument.

If the output differs greatly from the known concentration of the calibration gas, the initial procedure to remedy the problem is a thorough cleaning of the instrument. The cleaning process normally removes foreign materials (i.e., dust, moisture) that affect the calibration of

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the instrument. If this procedure does not rectify the problem, further troubleshooting is performed until the problem is resolved. If the problem cannot be resolved by the field personnel, the instrument will be returned to the manufacturer for repair and a replacement unit shipped to the site immediately. The manufacturer's manual will accompany the instrument.

The Hnu PI101 detector must be kept clean for accurate operation. Foreign materials can be rinsed or wiped off, or blown out of the detector. The cord between the analyzer and the recorder should not be wound tightly, and will be inspected visually for integrity before going into the field. A new cord will be ordered from the manufacturer if problems are found. A battery check indicator is included on the equipment and will be checked prior to going into the field and prior to use. The batteries will be fully charged each night. The analyzer, probe, and meter will be packed securely and handled so as to minimize the risk of damage.

5.2 CONDUCTANCE, TEMPERATURE, AND PH METER

A HyDAC conductance, temperature, and pH meter, or equivalent, will be used at the site. The unit has the following detection ranges: conductance 0 to 20,000 $\mu\text{mhos/cm}$; temperature 0 to 160°F; and pH 0 to 14. Conductance and temperature are factory calibrated: however, conductance will be checked against a standard solution of known conductance each day and recalibrated, if necessary.

The pH will be calibrated prior to each use by immersing the pH electrode in a pH 7.0 buffered solution. The electrode is then placed in a pH 4.0 or 10.0 buffered solution and the "SLOPE" potentiometer on the tester adjusted to display the value of the buffer solution chosen. The meter and probes will be packed in a protective case for transport. The probes must be kept clean, and will be rinsed with distilled water after each use. The buffer solutions used in calibration will be packed with the meter.

5.3 OIL/WATER INTERFACE PROBE

The oil water interface probe to be used will be an ORS® brand interface probe or equivalent. This unit gives a single tone when its probe interfaces with hydrocarbons and an intermittent tone when it reaches water. Depth is read directly off the tape. Field calibrating will entail measurement between wire marks with an accurate tape measure to help ensure length validity.

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5.4 NEPHELOMETRIC TURBIDITY METER

An HF Scientific DRT nephelometric turbidity meter or equivalent will be used during monitoring well development and sampling. The unit is battery operated and will be fully charged prior to all pertinent field operations. Calibration and other pertinent maintenance and operations information provided in the operator's manual will accompany the unit to the field.

5.5 DECONTAMINATION SUPPLIES

The decontamination wash solutions will consist of Alconox[®] detergent and potable water, distilled water, and hexane/methanol in accordance with procedures suggested by the Agency. Other supplies will include buckets, tubs, and brushes. The decontamination supplies will be transported in sealed unbreakable containers. The containers will be inspected visually for leaks or contamination prior to each use.

5.6 RESPIRATORS, CARTRIDGES, AND FILTERS

Air purifying filter/cartridge respirators will be donned by sampling personnel if field situations warrant. The respirators will be fitted with appropriate compatible cartridges meeting NIOSH (National Institute for Occupational Safety and Health) criteria for removal of organic vapors, dusts, and mists. These cartridges are NIOSH and MSHA (Mine Safety and Health Administration)-approved. The cartridge is approved for use in atmospheres containing at least 19.5 percent oxygen and less than 0.1 percent organic vapors by volume.

5.7 LOCKS

Padlocks will be placed on each monitor well to discourage tampering and vandalism. The locks will be purchased from a locksmith supplier or hardware store and will be performance tested at the time of purchase and when placed on a well. The locks will be keyed alike to avoid the possibility of confusion among keys. Three sets of keys will be provided to the Richards-Gebaur personnel.

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5.8 ELECTRICAL GENERATOR

A portable gasoline powered electrical generator with a minimum 3,500 watt capacity will be used to provide on-site power for the submersible pump. Care will be taken during the fueling of the generator to prevent the spillage of gasoline at the site.

6.0 QUALITY ASSURANCE/QUALITY CONTROL

The objective of the Quality Assurance/Quality Control (QA/QC) program is to demonstrate that the data produced are scientifically valid, defensible, and of known precision and accuracy. QC will be maintained in the field by adhering to the field procedures outlined in the Field Sampling Plan; by properly and fully documenting sample information on chain-of-custody forms; by maintaining field logs documenting field activities; and by the collection of QC samples. The QC samples will be analyzed to assess laboratory performance and to assess the possibility of cross-contamination.

6.1 QUALITY ASSURANCE/QUALITY CONTROL SAMPLES

As part of the Quality Control program, QC samples will be prepared and collected to provide data for the subsequent review, interpretation, and validation of the analytical data. Four types of QC samples for soil will be prepared or collected: (1) trip blanks; (2) duplicate (replicate) samples; (3) QA samples, and (4) equipment blanks. The QA/QC samples are discussed in more detail below.

6.1.1 Trip Blanks

Trip blanks will be prepared by the off-site analytical laboratory and shipped to the site with the sample containers. Two trip blank vials will be packed with each shipment of samples submitted to the laboratory for VOC analyses. The trip blanks will be analyzed for VOCs and will be used to assess the possibility of cross-contamination of the samples during shipment to the laboratory.

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6.1.2 Duplicate Samples

Collection of duplicate samples provides for evaluation of the laboratory's performance by comparing analytical results of two samples from the same location. Duplicate samples will be collected at a rate of 10% (1 in 10). Duplicates will be labeled as independent samples so they can not be identified as duplicates by the laboratory (blind duplicates).

6.1.3 QA Samples

QA samples will be collected to allow for an independent evaluation of the analytical data quality. The QA sample(s) will be sent to another laboratory, with the sample being collected by TapanAm personnel as directed by the AFBCA or by an independent third party.

6.1.4 Equipment Blanks

One equipment blank will be taken by the sampling team on each day of sampling. Sampling equipment blanks will be collected immediately after the equipment has been decontaminated. This blank will be analyzed for all laboratory analyses requested for environmental samples collected at the site in that particular day.

6.2 QA/QC SAMPLING PROCEDURES

In order for duplicate sample analysis to be valid, the duplicate samples must be as homogeneous as possible. Duplicate soil samples will be split vertically so that vertical stratification of contaminants will be distributed equally between the samples. Half of the sample (one of the split sides) will be transferred to the regular sample container; the duplicate half will be transferred to the similarly labeled duplicate or split sample container. Stainless steel sampling spoons and knives will be used. Samples will be handled by personnel wearing nitrile gloves to avoid contamination.

Soil collected for duplicate samples, or for samples to be split with third parties (QA samples), will be obtained by consecutively filling additional sets of sample jars using the same sampling equipment. Samples collected for VOC analysis will be placed directly into the sample jars. The sample jars will be completely filled to minimize the loss of volatiles.

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Composite soil samples obtained for TRPH or SVOC analysis will be homogenized prior to filling the sample jars. Moisture content, particle size, and absorption properties of the soils may inhibit complete homogenization. The soil sample to be homogenized will be placed initially in a stainless-steel bowl. After the removal of stones, vegetation, or other debris, the soil will be blended with a stainless-steel sampling trowel or spoon until it appears uniform in color and texture. The samples will then be placed into the sample containers.

Duplicate water samples will be obtained by consecutively filling additional sets of sample jars using the same sampling equipment. Duplicate samples for VOC analysis will be filled first from the same bailer.

7.0 SITE DOCUMENTATION

7.1 FIELD LOG BOOKS

Each TapanAm Field Team member will maintain a personal field log book while on the site. Information recorded in the log book will be written in an objective, factual manner so that persons reading the entries will be able to determine the sequence of events as they occurred in the field. If notes are made in the log book by someone other than the owner of the book, this will be indicated by the writer's signature and date. Information that may be recorded in the field log book include:

- Date and time of entry;
- Sample number;
- Sample description;
- Method of sampling;
- Location of sampling;
- Sketch of sample location;
- Field measurements such as pH, conductivity, temperature, and water level;
- Names and phone numbers of field contacts, drillers, and persons on-site;
- Materials used in well construction;
- Driller's standby and drilling time; and
- Weather and field conditions during drilling and sampling.

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In addition to the above information, the following forms will be used to record detailed data:

- HTW Drilling Log (Figure 2) - used in the field to record detailed sample descriptions and drilling methods;
- Monitoring Well Installation Details (Figure 5) - used to record details of well installation.
- Well Development Log (Figure 6) - used to record details of well development.
- Monitoring Well Sampling Form (Figure 7) - used to stabilize the wells prior to sampling.
- Daily Activity Log (Figure 9) - used to outline daily activities for information of project manager and file records; and

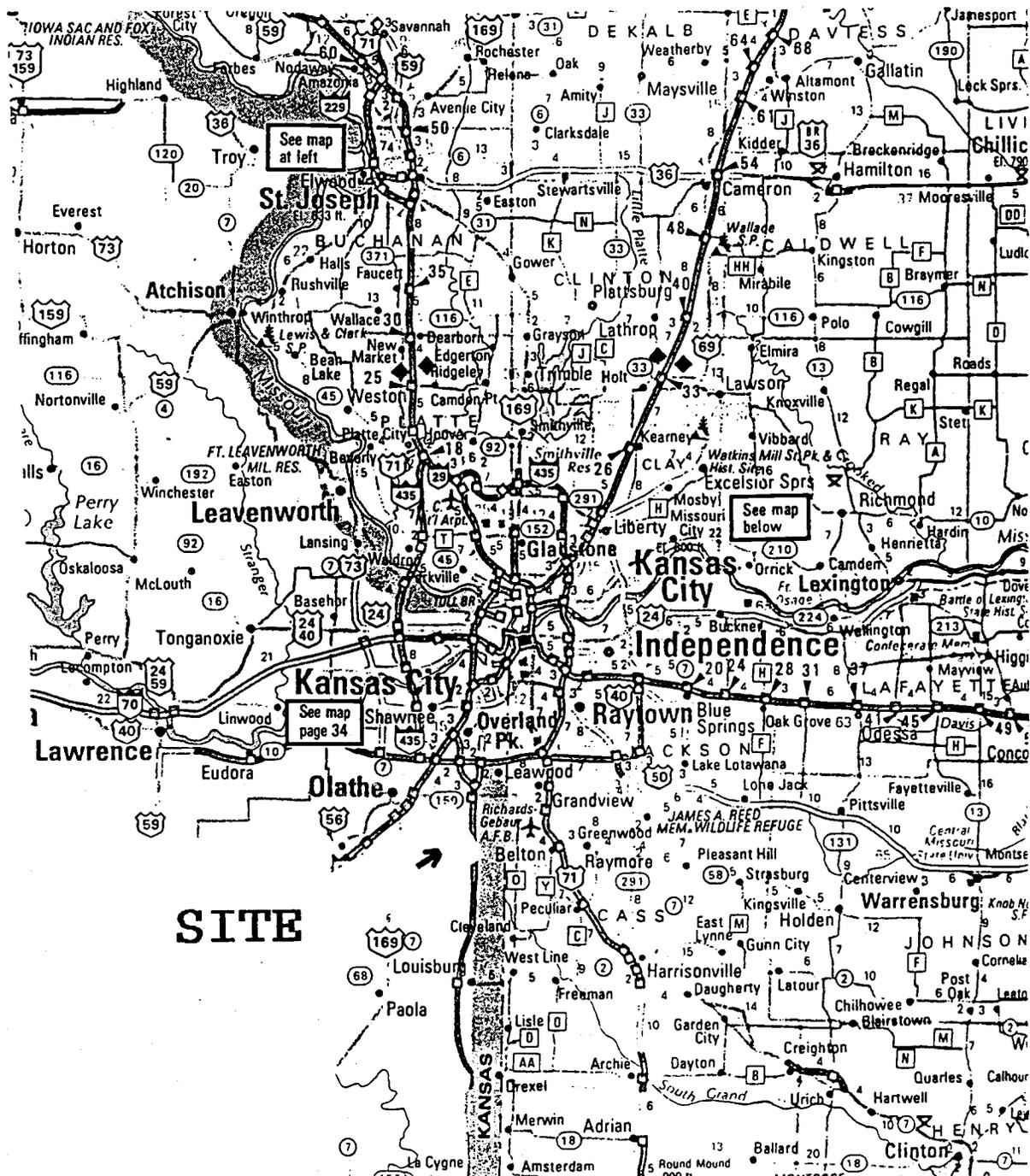
7.2 CORRECTIONS TO DOCUMENTATION

Errors or mistakes in the original field data will be crossed out with a single line, and the person making the correction will initial it. No data will be erased.

In some circumstances, original documents may be transcribed, making appropriate changes and eliminating errors. In these cases, the successive documents will be dated, numbered as sequential drafts and the originals maintained in the project file.

7.3 SAMPLE TRAFFIC REPORTS

Knowledge of sample status will be maintained through review and evaluation of the TapanAm field geologist's/engineer's reports, discussions with field personnel, and through contact with the analytical laboratory on a periodic basis. In this way, a working knowledge of sample traffic will be available throughout the project.



SITE

FIGURE DESCRIPTION:		SITE LOCATION MAP	
SITE NAME/LOCATION: RICHARDS-GEBAUR AIR FORCE BASE BELTON, MISSOURI		PROJECT NO.: 1211	
 TapanAm Associates, Inc. Engineers • Scientists • Architects 8010 STATE LINE LEANWOOD, KANSAS 66208 (913) 648-5411 FAX (913) 648-0418		FIGURE NO.: 1	

10/11/01 01:26:18 14:55:12

HTW DRILLING LOG						HOLE NO.
1. COMPANY NAME			2. DRILLING SUBCONTRACTOR			SHEET 1 OF SHEETS
3. PROJECT				4. LOCATION		
5. NAME OF DRILLER				6. MANUFACTURER'S DESIGNATION OF DRILL		
7. SIZES AND TYPES OF DRILLING AND SAMPLING EQUIPMENT		8. HOLE LOCATION				9. SURFACE ELEVATION
		10. DATE STARTED		11. DATE COMPLETED		
		12. OVERBURDEN THICKNESS				
13. DEPTH DRILLED INTO ROCK				16. DEPTH TO WATER AND ELAPSED TIME AFTER DRILLING COMPLETED		
14. TOTAL DEPTH OF HOLE				17. OTHER WATER LEVEL MEASUREMENTS (SPECIFY)		
18. GEOTECHNICAL SAMPLES		DISTURBED	UNDISTURBED	19. TOTAL NUMBER OF CORE BOXES		
20. SAMPLES FOR CHEMICAL ANALYSIS		VOC	METALS	OTHER (SPECIFY)	OTHER (SPECIFY)	OTHER (SPECIFY)
22. DEPOSITION OF HOLE		BACKFILLED	MONITORING WELL	OTHER (SPECIFY)	23. SIGNATURE OF INSPECTOR	
ELEV. a.	DEPTH. b.	DESCRIPTION OF MATERIALS c.		FIELD SCREENING RESULTS d.	GEOTECH SAMPLE OR CORE BOX NO. e.	ANALYTICAL SAMPLE NO. f.
						BLOW COUNTS g.
						REMARKS h.

htw1.cdr

PROJECT

HOLE NO.

FIGURE DESCRIPTION: HTW DRILLING LOG	
SITE NAME/LOCATION: RICHARDS-GEBEUR AIR FORCE BASE BELTON, MISSOURI	PROJECT NO.: 1211
TapanAm Associates, Inc. Engineers • Scientists • Architects <small>8010 STATE LANE LEANWOOD, KANSAS 66308 (913) 646-5411 FAX (913) 646-0840</small>	
2	

03-10-2011 12:51:46 PM 1002 01 70 1296 14-36-36

UNIFIED SOIL CLASSIFICATION SCHEME					
MAJOR DIVISIONS			GENERAL DESCRIPTION		
COARSE-GRAINED SOILS MORE THAN HALF IS LARGER THAN NO. 200 SIEVE	GRAVELS MORE THAN HALF COARSE FRACTION IS LARGER THAN NO. 4 SIEVE	CLEAN GRAVELS WITH LITTLE OR NO FINES	GW 	WELL GRADED GRAVELS, GRAVEL-SAND MIXTURES	
		GRAVELS WITH OVER 12% FINES	GP 	POORLY GRADED GRAVELS, GRAVEL-SAND MIXTURES	
		SANDS MORE THAN HALF COARSE FRACTION IS SMALLER THAN NO. 4 SIEVE	CLEAN SANDS WITH LITTLE OR NO FINES	SW 	WELL GRADED SANDS, GRAVELLY SANDS
			SANDS WITH OVER 12% FINES	SP 	POORLY GRADED SANDS, GRAVELLY SANDS
	FINE-GRAINED SOILS MORE THAN HALF IS SMALLER THAN NO. 200 SIEVE	SILTS AND CLAYS LIQUID LIMIT LESS THAN 50%	SILTY GRAVELS, POORLY GRADED GRAVEL-SAND-SILT MIXTURES	GM 	SILTY SANDS, POORLY GRADED SAND-SILT MIXTURES
			CLAYEY GRAVELS, POORLY GRADED GRAVEL-SAND-CLAY MIXTURES	GC 	CLAYEY SANDS, POORLY GRADED SAND-CLAY MIXTURES
			INORGANIC SILTS AND VERY FINE SANDS, ROCK FLOUR, SILTY OR CLAYEY FINE SANDS, CLAYEY SILTS WITH SLIGHT PLASTICITY	ML 	INORGANIC CLAYS OF LOW TO MEDIUM PLASTICITY, GRAVELLY CLAYS, SANDY CLAYS, SILTY CLAYS, LEAN CLAYS
		SILTS AND CLAYS LIQUID LIMIT GREATER THAN 50%	ORGANIC CLAYS AND ORGANIC SILTY CLAYS OF LOW PLASTICITY	CL 	INORGANIC CLAYS OF HIGH PLASTICITY, FAT CLAYS
INORGANIC CLAYS OF MEDIUM TO HIGH PLASTICITY, ORGANIC SILTS	CH 		ORGANIC CLAYS OF MEDIUM TO HIGH PLASTICITY, ORGANIC SILTS		
HIGHLY ORGANIC SOILS	PI 		PEAT AND OTHER HIGHLY ORGANIC SOILS		

FIGURE DESCRIPTION:
 UNIFIED SOIL CLASSIFICATION CHART

SITE NAME/LOCATION: RICHARDS-GEBEUR AIR FORCE BASE BELTON, MISSOURI	PROJECT NO.: 1211
 TapanAm Associates, Inc. Engineers • Scientists • Architects 8010 STATE LINE (913) 640-5411 LEANWOOD, KANSAS 66208 FAX (913) 640-0848	
FIGURE NO.: 3	

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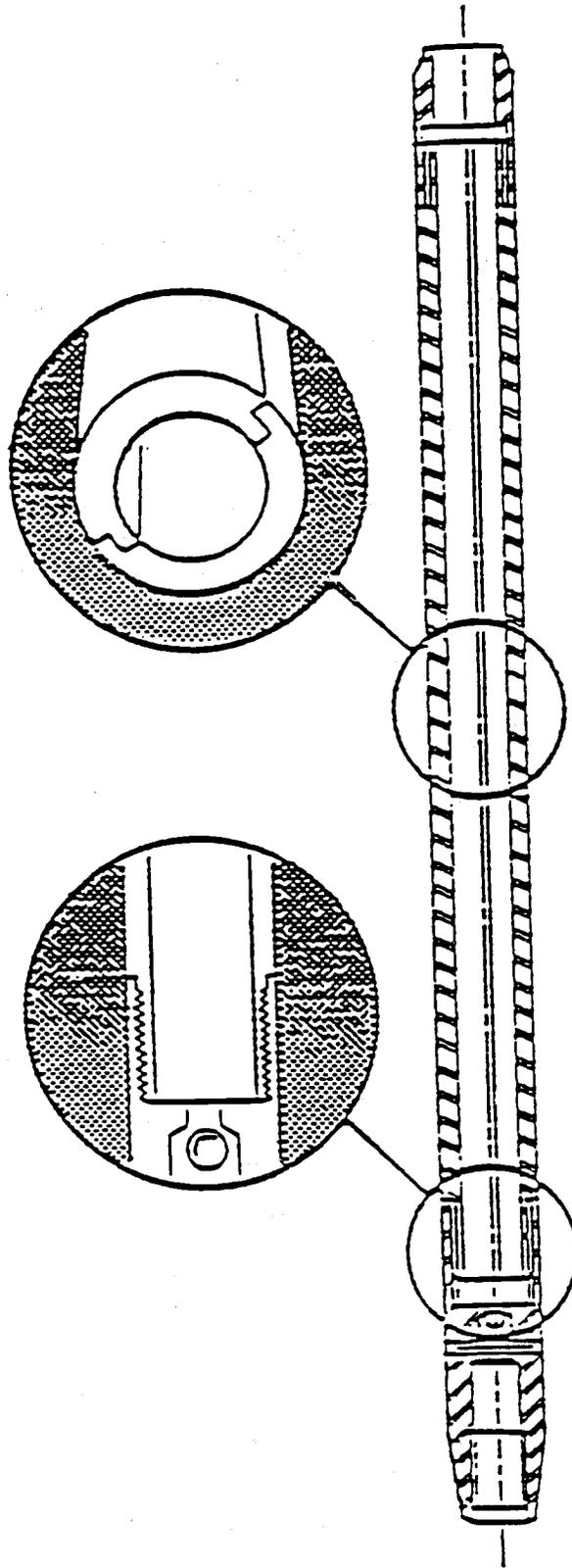


FIGURE DESCRIPTION:	
SPLIT-SPOON SAMPLER	
SITE NAME/LOCATION: RICHARDS-GEBAUR AIR FORCE BASE BELTON, MISSOURI	PROJECT NO.: 1211
 TapanAm Associates, Inc. Engineers • Scientists • Architects 8010 STATE LINE (913) 640-5411	LEANWOOD, KANSAS 66208 FAX (913) 640-0840 <div style="font-size: 2em; font-weight: bold; text-align: center;">4</div>
FIGURE NO.:	

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MONITORING WELL INFORMATION SHEET

Client _____
 Job Number _____
 Boring Number _____
 Location _____
 Coordinates _____
 Ground Surface Elevation _____
 Top of Well Casing Elevation _____
 By _____
 Date _____

- 1) Surface Completion (see top diagram at left).
 Height of well casing above ground _____ feet.
 Locking cap _____ Yes _____ No.
 Bumper posts _____ Yes _____ No.
 Number _____ Size _____
- 2) Concrete pad _____ Yes _____ No.
 Size (length/width/height) _____
- 3) Borehole diameter _____ inches.
- 4) Total length of riser pipe _____ feet.
 Pipe diameter _____ inches.
 Pipe material _____
 Schedule _____
 Centralizers _____ Yes _____ No.
 Number _____
 Depths _____
- 5) Type of upper backfill _____
 Depth to top of upper backfill _____ feet
 below surface .
- 6) Seal Material _____
 Depth to top of seal (if installed) _____ feet
 below surface.
- 7) Type of filter pack material around screen _____
 Filter pack _____ Yes _____ No.
 Depth to top of filter pack _____ feet below surface.
- 8) Length of well screen _____ feet.
 Diameter of screen _____ inches.
 Slot size of screen _____ inches.
 Screen Material _____
 Schedule _____
 Depth to top of well screen _____ feet below surface.
- 9) Sediment trap (solid pipe below slot) _____ feet.
- 10) Type of lower backfill _____
- 11) Depth to ground water _____ feet measured
 on _____ (date).
- 12) Total depth of boring completed at _____ feet
 completed on _____.
 Quantity of non chlorinated water introduced during
 installation _____ gallons.

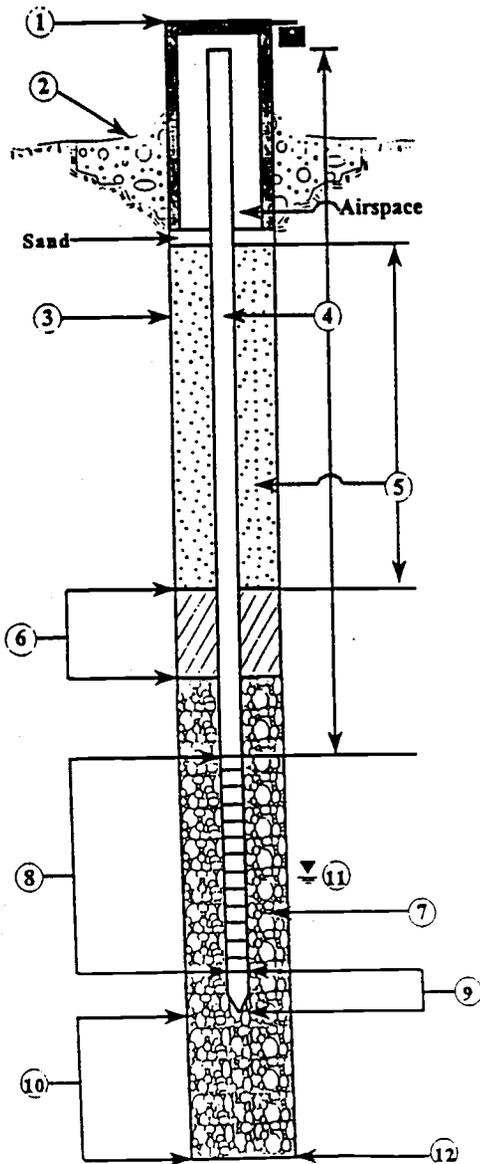
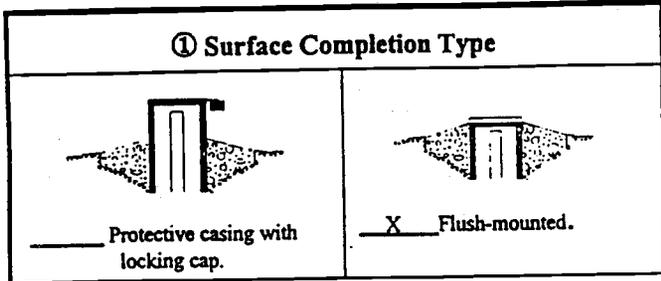


FIGURE DESCRIPTION:
 MONITORING WELL INFORMATION SHEET

SITE NAME/LOCATION: RICHARDS-GEBEUR AIR FORCE BASE BELTON, MISSOURI	PROJECT NO.: 1211
FIGURE NO.: <div style="display: flex; justify-content: space-between; align-items: center;"> <div style="text-align: center;">  TAI TapanAm Associates, Inc. Engineers • Scientists • Architects 8010 STATE LINE (913) 648-5411 </div> <div style="text-align: center;"> LEAWOOD, KANSAS 66208 FAX (913) 648-0648 </div> </div>	

5

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MONITORING WELL SAMPLING FORM

WELL NO. _____ - STABILIZATION TESTS

DATE: _____

Job # 19577-019

PARAMETER	WELL VOLUME EXTRACTED					
	1	2	3			
Time						
Specific conductance (temp. corrected) ±10 μhos/cm						
pH: ± 0.1 pH unit						
Temperature: ±1.0°F						
Turbidity						
Color						
Odor						
Other						

All measurements taken from the north side of the top of riser pipe

Well Diameter (feet) _____

Depth of well (feet) _____

Initial depth to water (feet) _____

Height of water column in riser (feet) _____

Volume of water in riser (gallons) [A] _____

Diameter of filter pack (feet) _____

Height of water in filter pack (feet) _____

Volume of water in borehole (gallons) [B] _____

Volume of water in filter pack (gallons) ¹[C] _____
(B - A) x 0.35

Total volume to be purged (gallons) [D] _____
(A + C)

Final depth to water (feet) _____

Purged dry? Yes _____ No _____

Time start/Time sampled _____ / _____

Product thickness and/or sheen _____ none _____

Comments: _____

¹ "C" is multiplied by 0.35 to account for 35% void space in filter pack.

Formula for Calculating Purge Volume

$$\frac{\pi D^2 h}{4} \times 7.48 = \text{volume in gallons}$$

D = Diameter in feet
 h = Height of water column in feet

Sampler Name(s) (Print): _____

FIGURE DESCRIPTION:
 MONITORING WELL SAMPLING FORM

SITE NAME/LOCATION:
 RICHARDS-GEBEUR AIR FORCE BASE
 BELTON, MISSOURI

PROJECT NO.:
 1211



TapanAm Associates, Inc.
 Engineers • Scientists • Architects
 8010 STATE LINE
 (913) 648-5411 LEANWOOD, KANSAS 66208
 FAX (913) 648-0848

FIGURE NO.:

7

FORM 10/19/96 10/15/98 10/01/03

TABLE 1
RICHARDS-GEBAUR AIR FORCE BASE
Sample Container and Preservation Requirements

Parameter	Reference Method	Container	Minimum Volume	Preservation	Max. Holding Time/Preparation	Max. Holding Time/Analysis
TRPH GRO- soil	SW8015	G	4 oz.	4°C	14 days	14 days
TRPH GRO - water	SW8015	G	2 x 40 mL	4°C, HCL to pH <2	14 days	14 days
TRPH DRO - soil	SW8015	G	8 oz.	4°C	14 days	40 days
TRPH DRO - water	SW8015	GA	1000 mL	4°C	7 days	40 days
PEST/PCB - soil	SW8080	G	100 g	4°C	14 days	40 days
PEST/PCB - water	SW8080	GA	1000 mL	4°C	7 days	40 days
PAH's - soil	SW8100	G	100 g	4°C	14 days	40 days
PAH's - water	SW8100	GA	1000 mL	4°C	7 days	40 days
VOCs - soil	SW8260	G	4 oz.	4°C	14 days	14 days
VOCs - water	SW 8260	G	2 x 40 mL	4°C, HCL to pH <2	14 days	14 days
SVOCs - soil	SW8270	G	4 oz.	4°C	14 days	40 days
SVOCs - water	SW8270	GA	2 x 40 mL	4°C, HCL to pH <2	7 days	40 days
RCRA metals - soil	SW6010	GA	8 oz.	4°C	180 days	180 days
RCRA metals - water	SW6010	P	500 mL	4°C, HNO to pH < 2	180 days	180 days

G Glass wide mouth jar with teflon-lined cap

GA Glass amber wide-mouth bottle with teflon-lined cap

P Plastic, polyethylene bottle with polypropylene cap

QUALITY ASSURANCE/QUALITY CONTROL PLAN

**LEAD BASED PAINT INSPECTION
RADIATION SAFETY**

JANUARY 1996

KINGSTON ENVIRONMENTAL SERVICES

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Quality Assurance/Quality Control Plan Lead Based Paint Inspection Radiation Safety

This document outlines the practices, procedures and methods used by Kingston Environmental Services, Inc. to provide training for its employees in the use of its radioactive lead analyzers. Successful completion of the course will assist the employees in the safe use of the equipment, as well as specific radiation safety practices consistent with the devices and the radioactive material involved.

PROFESSIONAL TRAINING REQUIREMENTS

1. All personnel participating in project activities will have received the 24-Hour Lead Inspector Training from an accredited training facility in accordance with 19 CSR 20-8.010 (1-4) (Missouri Department of Natural Resources). This training will have met or exceeded 19 CSR 20-8.010(6), and the inspector will be Missouri licensed in accordance with 19 CSR 20-8 (Licensing of Lead Inspectors).
2. Personnel will have received the mandatory Princeton Gamma-Tech training regarding the use of the XK-3 XRF Analyzers, principles and practices of radiation safety, and product-specific information to the analyzer used onsite. This training will be provided by the Radiation Safety Officer (RSO) as denoted by the XRF-Analyzer license.

Topics covered in the X-ray Fluorescence training include:

1. Theory of the X-ray Fluorescence and Spectrum Analysis.
2. Theory of the XK-3 and MAP-3 Analyzer.
3. The construction and characteristics of the Cobalt-57 source.
4. Principles and practices of radiation safety, biological hazards, radiation protection and specific XK-3 Warrington and MAP-3 safety practices.
5. Operation of the XK-3, MAP-3 and Warrington.
6. Questions and Discussion.

Following completion of the training program, the trainer will document, in writing, that:

1. The named person has completed the program;
2. Sufficient hands-on and on-the-job training have allowed the named person to demonstrate to the trainer competence in handling and use of the device; and
3. The person is an approved user.

The documentation will become a part of the individual's personnel file as well as having their name added to the list of approved users maintained by the Trainer.

RADIATION SAFETY

Radioactive Material Control

Instruments containing quantities of radioactive material, except those specifically exempted, are subject to regulation by the U.S. Nuclear Regulatory Commission (NRC) or an Agreement State. An "Agreement State" is one which has entered into an agreement with the NRC, in which regulatory authority over the use of certain material within the state has been transferred to the respective state. These agreements provide for a high degree of compatibility between regulatory programs of the state and the NRC. Because of the similarities between the programs of the twenty-seven Agreement States and the NRC, this section will discuss only the NRC program as being exemplary of all programs.

The approach utilized in assuring the safety of a manufactured device containing radioactive material is to regulate the manufacturer. The inherent safety of the device is studied, and conditions for assuring its safety are contained in the specific license under which the device is manufactured and distributed. Additional conditions will be stated in the specific license under which the device is manufactured and distributed. Additional conditions, incident to the use of the device, will be stated in the specific license which must be obtained by the user before he/she can take possession. The following discussion will detail the inherent safety features of the analyzer, the magnitude of the radiation hazard, and the regulatory responsibility of the user.

Worksite Safety and Health

Physical Hazards

Physical Hazards are an inherent risk when working in an unfamiliar environment. Accidents can be avoided by eliminating unsafe conditions and reducing unsafe actions. A high degree of safety awareness is necessary to maximize efficiency and minimize accidents. This degree of safety will be reached with extensive instruction to inspectors from the Site Safety/Project Manager. The following are the various topics which will be conveyed by the Site Safety Manager.

1. Injuries Resulting from Materials and Machine Handling -

The reduction of unsafe work habits and proper use and protection associated with machine operation can eliminate risk of injury to not only operators and handlers, but also to those persons in proximity to those actions.

- 1.a. Cold stress can take the form of frostbite or more seriously, hypothermia. Personnel should wear gloves and thermal work attire to include thermal underwear, socks, and hardhat liners whenever the temperature drops below 35°F. Exposure to below freezing temperatures can cause rapid loss of body temperature and result in hypothermia or frostbite.

- 1.b. The heat stress prevention program consists of physiological monitoring and symptom recognition and treatment. Many factors besides ambient temperature, such as age, weight, physical fitness, and acclimatization can affect the body's temperature control system. For this reason, physiological monitoring is required when ambient temperatures are elevated. The inspector will monitor his/her heart rate by counting the radial pulse over a 30 second period early in the break period. If the heart rate exceeds 110 beats per minute then the work period will be shortened by one third. The rest period will remain the same. If the heart rate is still over 110 at the next break period then cut the work period by one third again. The inspector will also monitor his/her body temperature by measuring oral temperature. This should also be done early in the break period. If the body temperature exceeds 99.6°F, adjustments of work periods will be made.
- 1.c. Injuries related to electrical hazards are a risk associated with work in dated, abandoned facilities and crawl spaces. When maneuvering in areas where electrical hazards are prevalent, be sure to identify electrical lines - damaged or otherwise and avoid them if possible. Again, identifying the hazard and reducing unsafe actions prevent accidents.

2. Radiation Hazards -

The use of an XRF analyzer poses the threat of radiation hazards to not only the operator of the instrument, but also those around the operator. As with any instrument containing radioactive materials, the main considerations are to prevent unnecessary radiation exposure and to insure that the radioactive material remains contained. The radiation hazard associated with the analyzer is minimized by the construction detail; the source is constructed such that significant radiation is emitted in one direction only. Health officials measure radiation in mREM/hr based on the Time of Exposure, Distance, and Shielding between the operator and the radioactive material (TDS). The Cobalt-57 source emits relatively low levels of radiation, though TDS should be optimized whenever possible. (See Medical Surveillance, No. 2 for radiation exposure assessments). **Never pull the trigger while pointing the probe at another person.**

In the event of an accident which is thought to have damaged the source encapsulation, the appropriate authorities should be notified immediately. The source should be handled as little as possible to avoid the spread of contamination until appropriate measurements have been taken. Manufacturers will provide advice and help in the evaluation of further steps to be taken.

Should the use of the analyzer be discontinued or the disposal of the radioactive material become necessary, contact your manufacturer. Disposal of radioactive material must be handled by someone specifically licensed to do so.

Health Surveillance Programs

Medical Surveillance

1. General Medical Examinations

All personnel conducting inspection work will have passed a physical examination within the last year. The examination must comply with the requirements of 29 CFR 1926.58(m). Components of the exam must include a work and medical history questionnaire, pulmonary function testing, a chest roentgenogram at the physician's discretion, and a physical examination directed to the pulmonary and gastrointestinal systems. The results of the medical examinations are maintained in accordance with 1926.158 (n)(3).

2. Lead Exposure Assessment Program

The permissible exposure limit for lead is 50mg/m³ over an 8-hour period (OSHA 29 CFR § 1910.62 (c)). Should respirators be used to limit employee exposure, employee exposure can be considered at the level provided by the protection factor of the respirator for the period of time worn (See No. 4 below on Respiratory Programs). The exposure assessment program will comply with OSHA 29 CFR § 1910.62 (d) for all personnel participating in the inspection.

3. Radiation Exposure Assessment from XRF Analyzer

The surveillance program associated with radiation from the XRF analyzer specifies that all personnel using the instrument wear a dosimetry badge when operating the XRF analyzer. The dosimetry badges, which indicate accumulated radiation exposure are worn for one (1) month and then returned to the supplier for analysis. In turn the supplier issues new badges and returns the dosimetry reading of the old badges. Dosimetry reports enable an organization to track each inspector's exposure over time.

4. Respiratory Protection Program

The use of respirators is required under the following conditions: when an employee's exposure to lead exceeds the PEL; in work situations where engineering controls and work practices are not sufficient to reduce exposures to or below the PEL; when an employee requests a respirator; and an interim protection for employee's performing special tasks. The respiratory protection program is in accordance with OSHA 29 CFR § 1910.62 (f). This program also adheres to regulations stated in OSHA 29 CFR § 1910.134 (b), (d), (e) and (f). The following table illustrates the specific type of respirator needed when working amidst various concentrations of lead aerosols.

TABLE 1 - RESPIRATORY PROTECTION FOR LEAD AEROSOLS

Airborne Concentrations of Lead of Conditions of Use	Required Respirator
Not in excess of 500mg/m ³	½ mask air purifying respirator with high-efficiency filters
	½ mask supplied air respirator operated in demand (negative pressure mode).
Not in excess of 1,250mg/m ³	Loose fitting hood or helmet powered air-purifying respirator with high-efficiency filters.
	Hood or helmet supplied air respirator operated in a continuous-flow mode.
Not in excess of 2,500mg/m ³	Full face piece air purifying respirator with high-efficiency filters.
	Tight fitting powered air purifying respirator with high-efficiency filters.
	Full face piece supplied air respirator operated in demand mode.
	½ mask or full face piece supplied air respirator operated in a continuous flow mode.
	Full face piece self-contained breathing apparatus (SCBA) operated in demand mode.
Not in excess of 50,000 mg/m ³	½ mask supplied air respirator operated in pressure demand or other positive pressure mode.
Not in excess of 100,000mg/m ³	Full face piece supplied air respirator operated in pressure demand or other positive pressure mode.
Greater than 100,000mg/m ³ - unknown concentration	Full face piece SCBA or operated in pressure demand or other positive pressure mode.

5. Personal Protective Equipment Program

Kingston lead inspectors work in total compliance with OSHA 29 CFR § 1910.62 (g). This section describes the specifics of the personal protective equipment program necessary during lead exposure. Personal protective equipment is needed when personnel are exposed to lead above the PEL, without regard to the use of respirators. Full body coveralls, disposable hats, gloves, and eye/face protection are recommended in addition to equipment which applies under OSHA 29 CFR § 1910.133.

TESTING METHODOLOGY

Instrumentation

One of the lead-based-paint analyzers utilized by Kingston is the PGT XK-3 X-ray Fluorescence direct reading instrument manufactured by Princeton Gamma-Tech, Inc., Princeton, New Jersey, is equipped with a 10mCi Cobalt-57 sealed radioactive source. The corrected lead concentration (CLC) is arrived at by subtracting the substrate equivalent lead concentration (SEL) from the apparent lead concentration (ALC). PGT XK-3 X-ray Fluorescence direct reading serial #689 was used for the inspection of Union Station.

X-ray fluorescence instruments display lead content in milligrams per square centimeter (mg/cm^2). Based on Princeton Gamma Tech's Operation Procedures for the direct reading instrument, painted surfaces with lead content greater than or equal to $1.0 \text{ mg}/\text{cm}^2$ are considered positive for the presence of lead. Laboratory results with total lead content greater than 0.5% by weight are considered positive for the presence of lead according to Title X, Lead Based Paint Poisoning Prevention Act.

Inspections consist of testing each building component with every type of paint referenced. According to the XRF manufacturer's guidelines, when testing surfaces for LBP employing a direct read instrument, results less than $0.5 \text{ mg}/\text{cm}^2$ are considered negative for lead content; greater than $1.5 \text{ mg}/\text{cm}^2$ are considered positive for lead content; and $0.5 \text{ mg}/\text{cm}^2$ to $1.5 \text{ mg}/\text{cm}^2$ are inconclusive.

When testing with the direct read instrument, several readings are taken, potentially increasing the precision of the measurement. Consistently three readings are collected to establish a history for a specific paint on a certain component. Once the history of the specific paint is known, fewer readings may be collected when in agreement with established levels. Atomic Absorption Spectroscopic analysis (AAS) is utilized to determine inconclusive lead analysis results of painted surfaces to verify the XRF results.

Negative readings may be identified with the direct read XRF. This type of machine allows this measurement error, whereas, a spectrum analyzer reports all negative readings as zero. However, when the substrate correction is calculated, spectrum analyzer measurements can also be negative since it is possible for the substrate reading to exceed that on the paint.

Calibration

Field quality control procedures are organized to protect from a drift of the XRF's calibration. HUD and NIST suggest that calibration checks against a wood and lead source be conducted at the beginning and end of each testing day. KES, however, checks the calibration of the XRF at the beginning and end of each testing period - meaning whenever the instrument is stopped for a break. If the difference of the averages of each period exceed $0.7 \text{ mg}/\text{cm}^2$ for either the wood or lead calibration, the drift test fails.

Factors Affecting Precision

The XRF's capabilities may be affected by several factors. The manufacturer of the machine suggests not to use the instrument in weather conditions less than 35 degrees F. That is why the field surveys are sometimes rescheduled for a later time if the weather is extremely cold. The machine is kept in warm storage at the end of each testing day and checked for proper functioning prior to reaching the work site.

In addition, the source strength may affect precision of the instrument. As the Cobalt-57 source decreases in strength, the measurement time is increased. The half-life is 273 days of this radioactive isotope. Therefore, KES replaces the source new at least annually.

Also, unusual variability may be caused by changing substrates often. The inspectors collect readings in order of the substrate types. And the first reading after changing substrates is discarded if it is not within the established history of that specific paint component.

Report Production

The results of inspections are arranged in table form. The table includes a list of suspect LBP by room number, color of paint, substrate and component that the paint covers, location, and concentration of lead content. In addition to the table, confirmatory paint chip laboratory results and an estimate of LBP quantities are included. Two sample pages from LBP surveys follow this section.

SAMPLE TABLE FORM GROUPED BY COMPONENT

SUMMARY of RESULTS

Interior and exterior paint surfaces were tested for lead content. A total of 842 individual components were tested with multi-readings. Testing and sampling of paint materials indicated that the following colors of paint of the specific components listed are lead-based:

Component	Paint Color
Metal Access Panel	Black, Egg Shell White, Lime Green, Red Rust, White
Wood Baseboard	Grey
Wood Cabinet	White
Wood Cabinet Door	Mustard Yellow
Wood Cabinet Interior	Black
Plaster Ceiling	Cream, Egg Shell White, Orange, Pumpkin, Tan
Cement Column	Black
Metal Column	Grey
Metal Column Cover	Black, Grey
Metal Column Door	Green, Grey
Metal Door	Black, Blue, Grey
Wood Door	Green, Red

SAMPLE SUMMARY OF RESULTS

TEST #	BUILDING	AREA	COMPONENT	SUBSTRATE	PAINT COLOR	LOCATION	READING #1	READING #2	READING #3	AVG. ALC.	CLC. **
388		5011	ACCESS PANEL	METAL	BLACK	NORTH WALL	0.6	0.5	0.6	0.8	-0.2
388		6011	ACCESS PANEL	METAL	BLACK	NORTH WALL	1.5	0.1	1.0	0.8	0.2
246		3029	ACCESS PANEL	METAL	BLACK	SOUTH WALL	1.2	10.0	1.2	0.8	0.4
376		6024	ACCESS PANEL	METAL	BLACK	SE CORNER	10.0	10.0	10.0	0.8	0.8
574		1057	ACCESS PANEL	METAL	EGG SHELL WHITE	EAST WALL	3.6		3.6	0.8	2.6
588		1064	ACCESS PANEL	METAL	GREY	CENTER ROOM	0.9		0.9	0.8	0.1
194		3037	ACCESS PANEL	METAL	GREY	SOUTH WALL	1.1	1.2	1.4	0.8	0.4
573		1057	ACCESS PANEL	METAL	GREY	EAST WALL	1.4		1.4	0.8	0.8
62		2041	ACCESS PANEL	METAL	LIME GREEN	CENTER EAST WALL	10.0	10.0	10.0	0.8	9.2
79		2028	ACCESS PANEL	METAL	LT CHOCOLATE	NORTH WALL	1.1	1.4	1.4	0.8	0.6
3074		3074	ACCESS PANEL	METAL	LT PEACH GREEN	NW CORNER	1.1	1.1	1.1	0.8	0.3
198		3038	ACCESS PANEL	METAL	LT PEACH GREEN	SOUTH WALL	1.0	1.2	1.2	0.8	0.3
282		3057	ACCESS PANEL	METAL	MUSTARD YELLOW	SE CORNER	1.2	1.2	1.2	0.8	0.4
20		2009	ACCESS PANEL	METAL	PEACH GREEN	SW CORNER	1.4	1.4	1.3	0.8	0.5
135		2075	ACCESS PANEL	METAL	RED FLUOR	SOUTH WALL COLUMN	10.0	10.0	10.0	0.8	9.2
559		1040	ACCESS PANEL	METAL	TAN	SOUTH WALL	1.4	1.0	1.3	0.8	0.5
387		5010	ACCESS PANEL	METAL	VARNISH	NW CORNER	-0.3		-0.3	0.1	-0.4
295		3077	ACCESS PANEL	METAL	WHITE	CENTER OF ROOM	1.9	1.4	1.9	0.8	1.1
3029		3029	ACCESS PANEL	METAL	LT PEACH GREEN	NORTH WALL	0.8	0.9	1.0	0.8	0.2
423		M006	BANNER	WOOD	VARNISH	CENTER OF ROOM	-0.3	-0.4	-0.4	0.1	-0.5
486		M010	BASEBOARD	WOOD	BLUE GREEN	WEST WALL	0.5	0.2	0.2	0.1	0.2
136		2075	BASEBOARD	WOOD	BROWN	EAST WALL	-0.3	-0.4	-0.7	-0.5	-0.6
519		1002	BASEBOARD	CEMENT	BROWN	NORTH WALL	0.4	0.0	0.4	0.2	0.2
843		B04	BASEBOARD	WOOD	BROWN	SOUTH WALL	0.7	0.0	0.5	0.4	0.1
521		BM04	BASEBOARD	PLASTER	GREY	SOUTH WALL	-0.2	-0.2	-0.2	0.1	-0.3
522		BM22	BASEBOARD	WOOD	GREY	SOUTH WALL	0.0	0.2	0.2	0.0	0.1
374		6025	BASEBOARD	PLASTER	GREY	NORTH END	0.3	0.2	0.1	0.1	0.0
165		2035	BASEBOARD	WOOD	GREY	NW CORNER	0.0	0.3	0.1	0.1	0.0
108		B27	BASEBOARD	WOOD	GREY	CENTER	0.3	0.0	0.1	0.1	0.0
582		1055	BASEBOARD	PLASTER	GREY	SOUTH WALL	-0.4	0.8	0.0	0.1	0.0
817		5002	BASEBOARD	PLASTER	GREY	WEST WALL	-0.4	0.9	0.2	0.2	0.1
563		5002	BASEBOARD	PLASTER	GREY	EAST END	0.0	0.9	0.2	0.2	0.1
353		6010	BASEBOARD	PLASTER	GREY	WEST WALL	0.4	0.5	0.4	0.3	0.2
389		489	BASEBOARD	PLASTER	GREY	NORTH WALL	0.4		0.4	0.1	0.3
490		BM10	BASEBOARD	WOOD	GREY	SOUTH WALL	0.5	10.0	0.5	0.1	0.4
623		1124	BASEBOARD	METAL	GREY	EAST WALL	-0.5	0.5	0.5	0.1	6.4
88		2031	BASEBOARD	WOOD	MUSHROOM	NORTH WALL	10.0	-0.2	10.0	0.8	9.2
87		B39	BASEBOARD	WOOD	RED	WEST WALL	-0.4	-0.1	-0.2	0.1	-0.3
155		2083	BASEBOARD	WOOD	RED FLUOR	SOUTH WALL	0.2	0.1	0.0	0.1	-0.1
176		805	BASEBOARD	BRICK	TAN	NORTH WALL	0.2	0.2	0.2	0.5	-0.3
158		2099	BASEBOARD	WOOD	VARNISH	WEST WALL	-0.9	-0.9	-0.9	0.1	-1.0
154		2083	BASEBOARD	WOOD	VARNISH	NW CORNER	-0.7	-0.7	-0.7	0.1	-0.6
78		2027	BASEBOARD	WOOD	VARNISH	NORTH WALL	-0.3	-0.3	-0.3	0.1	-0.4
2075		2075	BASEBOARD	WOOD	VARNISH	NORTH WALL	-0.3	-0.3	-0.3	0.1	-0.4
288		3077	BASEBOARD	WOOD	VARNISH	EAST WALL	-0.3	-0.3	-0.3	0.1	-0.4
551		1053	BASEBOARD	WOOD	VARNISH	NORTH WALL	-0.3	-0.3	-0.3	0.1	-0.4
197		3037	BASEBOARD	WOOD	VARNISH	WEST WALL	-0.3	-0.1	-0.2	0.1	-0.3
30		2010	BASEBOARD	WOOD	VARNISH	SW CORNER	-0.2	-0.2	-0.2	0.1	-0.3
181		2088	BASEBOARD	WOOD	VARNISH	WEST WALL	-0.2	-0.2	-0.2	0.1	-0.3
131		2084	BASEBOARD	WOOD	VARNISH	EAST WALL	-0.1	-0.1	-0.1	0.1	-0.2
457		M036	BASEBOARD	WOOD	VARNISH	SOUTH WALL	-0.1	-0.1	-0.1	0.1	-0.2
13		2009	BASEBOARD	WOOD	VARNISH	EAST CENTER	0.2	-0.1	0.0	0.1	-0.1
37		2012	BASEBOARD	WOOD	VARNISH	CENTER SOUTH WALL	0.0	-0.1	0.0	0.0	0.1

PROJECT#: _____
 DATE: FEBRUARY 1-15, 1981
 INSPECTED BY: BARNES, JACKSON, HARRIS

KANSAS CITY, MISSOURI

EMERGENCY RESPONSE

Although the XRF utilizes a very low level radioactive source, which for protective purposes is encased in a tungsten alloy holder and covered by a shutter located at the face of the probe, people in daily contact with the instrument should take safety precautions to limit radiation exposure.

Many states require owners of XRF analyzers to perform a bi-annual leak test as a safety precaution. A leak test measures extremely low Radiation levels to determine if an instrument's source leaks radiation. A leak kit includes a cotton swab, alcohol, plastic envelope and report forms. Performing the testing involves swabbing the front of the probe with alcohol and returning the swab in the envelope along with its paperwork. Shortly thereafter the supplier will return the results of the test. Dosimetry badge suppliers usually provide leak kits.

A licensed owner must receive license reciprocity before operating the XRF analyzer in another Agreement state. In most instances, the owner must provide written notice and a phone call to that state's radiation control agency before entering and operating the XRF analyzer in that state.

Do not lean on the analyzer while taking a reading. Place your hand at the back of the probe housing to steady it, rather than at the front of the probe. Although the Radiation exposure is very small, none is better than some. Remember TDS.

In the event of an accident (dropping the probe, etc.) check the shutter, using a mirror, to determine if it is shielding the source. If the shutter is open, place the probe face down as if taking a reading on the ground or floor; this directs the radiation into the ground. Immediately notify Melissa McKee, (the Radiation Safety Officer, RSO), IRF manufacturers, and Kansas Department of Health. They will advise you how to handle, package, and ship the analyzer back to the factory. In the event the probe is smashed, do not attempt to pick it up. Immediately notify your RSO, XRF manufacturer, Kansas Department of Health, and the local health department. Keep people away from the analyzer until the health department arrives to survey the damage and contain the radioactive source.

All personnel are advised to familiarize themselves with the radiation protection regulations of the State of Kansas, a copy of which is in the RSO's office.

PHONE NUMBERS.....	Melissa McKee
	Work - (816) 524-8811
	Home - (816) 363-7980
XRF Manufacturer.....	(PGT) - 609-924-7310

Kansas Department of Health - RADIOLOGICAL MATERIALS

(913) 296-1562

(913) 665-7153 (after hours)

(913) 296-3176 (24 Hr. No.)

EMERGENCY RESPONSE

(913) 296-1561

(913) 761-2363 (after hours)

WHAT YOU SHOULD KNOW ABOUT NUCLEAR RADIATION

Nuclear radiation is the emission and propagation of electromagnetic energy waves. When matter is raised to a high enough temperature that the thermal motion of the particle provides enough kinetic energy for fusion reactions, the process is called thermonuclear.

We have been bombarded with radiation from natural sources - namely from the sun - since the beginning of creation. But until publicity connected with the Three Mile Island nuclear generating power station incident in April 1979, most of us were not aware of any radiation problems.

Three Mile Island Incident

Many multi-million dollar lawsuits were filed immediately after the Three Mile Island incident because of potential personal radiation damage. As a result of the Three Mile incident, most of the construction on 91 U.S. nuclear power plants in various stages of completion was stopped and all activity on 31 new plants on order was halted.

With the adverse publicity, lawsuits, political confusion and indecision that followed the Three Mile Island incident, very few in authority seemed to understand the facts about radiation. It is obvious that the majority of the public does not understand radiation either.

Milli REMs

Radiation is measured in ROENTGENS, the international unit of Gamma Radiation equal to the radiation charge on one cubic centimeter of dry air. Scientists now use a more subjective measurement, the REM, meaning Roentgen Equivalent Man for measuring the effects of radiation on the human body. Since the normal measurable radiation is infinitesimal and the REM is a fairly large unit, the term Milli REM - or one thousandth (.001) - of a REM is commonly used.

Safe Dosage

The Nuclear Regulatory Commission (NRC), in Parts 19 & 20 of their rules and regulations, has established maximum safe permissible REM dosages for individuals working in nuclear power

plants, in x-ray laboratories or with any type of radioactive materials, at three REM (3,000 Milli REM) per quarter year, and an accumulated total of five REM (5,000 Milli REM) annually.

Thousands of individuals working many years in the field of radiology (x-rays) have absorbed from 3,000 to 5,000 Milli REM annually, with no apparent deleterious effects.

The Environmental Protection Agency (EPA) has set the permissible natural radiation exposure at 170 Milli REM per year for the general public. An approximate overall radiation absorption in the U.S.A., including natural and man-made sources, is less than 200 Milli REM annually.

Keep these figures in mind... 5,000 Milli REM for individuals working directly with radiation equipment and 10 Milli REM annually for the general public. These figures are the safe limits for radiation absorption. But to how much radiation are we actually exposed?

Natural Radiation

From natural radiation, for example, people in Houston can absorb around .50 Milli REM annually, but in mile-high Denver the populace averages 130 Milli REM annually because of their closer proximity to the sun.

In addition to the 50 Milli REM absorbed from the sun, we can pick up another 5 Milli REM from the air, 1 from television, 15 from the ground, 25 from food and 30 from buildings....a total annual dosage in Houston of approximately 125 Milli REM. But in mile-high Denver, people can absorb more than the 170 Milli REM set by the EPA. We can also pick up from 20 to 50 Milli REM from one chest x-ray, 5 more from a coast to coast jet flight and a few from a microwave oven.

Nuclear Power Plant Radiation

How much radiation would a person living within a 50 mile radius of an operating nuclear power plant absorb? The answer is less than half a Milli REM per year. Yes, less than half a Milli REM annually. Even during the Three Mile Island incident, the highest measurable dose on the island was only 1,100 Milli REM and 50 miles away the radiation level was down to 83 Milli REM.

Cancer

With all the potential radiation we can absorb in our daily living we don't come close to the 5,000 Milli REM permitted by NRC. But what happens if any one of us should accidentally absorb the 170 Milli REM, or even the 5,000 Milli REM in a year? Will we automatically get cancer? No! There is some evidence that an accumulation of over 100,000 Milli REM may have some relationship to cancer, but in order to accumulate 100,000 Milli REM a body would have to be exposed to and absorb 5,000 Milli REM annually for over 20 years.

Infinitesimal

Yes, the Three Mile Island incident caused a big furor in the minds of some people, but from the available facts - backed up by the three-month Houston study - it is evident that the dangers from man-made or natural radiation are infinitesimal.

Reliable studies indicate that less than 5 percent of all cancers can be traced to radiation and that more than 100,000 lives are saved annually with the use of radiation.

Health Safety

All radiation, if received in sufficient quantities, can damage living tissue, yet radioactive materials can be of great help in certain applications. We can reconcile the apparent dangers of using radioactive materials with their benefits if we keep in mind the following points:

- * Within certain limits, the body can repair radiation damage so that there is no apparent effect.
- * There exists a sizable body of knowledge on the effect of radiation on the body.
- * When it is known how to maintain exposure within reasonably safe limits, it is reasonable for people to expose themselves to radiation in order to accomplish necessary work.

That is nice philosophy, you say, but I have heard all sorts of things about radiation, and I don't want to take any chances. I don't want any exposure to radiation at all.

The fact is, we cannot avoid exposure to radiation. We are all exposed to radiation from outer space, cosmic radiation, which increases in intensity as we go up in altitude. For instance, people in Denver, Colorado, the mile-high city, receive twice the radiation from cosmic rays than people who live at sea level. Let's get away from cosmic radiation and go down deep in a mine where no cosmic radiation can penetrate. We still haven't solved the problem, because then we are exposed to radiation from radioactive sources within the mine and from radioactive elements within the make-up of our own bodies, such as radioactive potassium and radioactive materials that we ingest, particularly from the water we drink. Water, in some parts of the country, particularly from some mineral springs, has appreciable radioactivity. So, from the beginning of time, man has been exposed to inescapable natural radiation.

In addition to the natural background of radiation, the population as a whole receives a certain amount of radiation from medical diagnostic and therapeutic procedures. It is obvious from our basic premise that radiation can damage living tissue, that some of this medical and dental radiation may have some harmful effects. However, we balance this harmful effect against the good we expect to accomplish from the medical procedure. If we find a specific medical procedure in which the hazard is not outweighed by the good received, the logical course is to modify this specific procedure, not to do away with all radiation exposure.

An example of foolish and unnecessary exposure to radiation is the use of x-ray machines for fitting children's shoes. These are a hazard, not only to the child, but to the shoe clerk. No useful purpose is served which could not be served by other means, and in many jurisdictions these devices have been outlawed.

Probably the chief error in much of our current thinking about radiation hazards is the failure to relate radiation hazards to the other hazards of human existence. All human activity involves risks. Some of them are physical, such as the hazard of being hit on the head with a heavy object dropped from above. Some hazards are more mental than physical, such as those of the advertising executive or play producer, who is under the constant strain of delivering completely satisfactory work or suffering the penalty of being ruthlessly eliminated. Consciously or not, when we select our field of work, we make an appraisal of the hazards involved, along with the other factors, such as pay, general working conditions, prospects for advancement, security of employment, etc. Each occupation has its own peculiar hazards, inherent in the nature of the work. In controlling the hazard we attempt to reduce the probability of accident to a minimum but cannot guarantee absolute freedom from risk.

Radiation hazards are no different from other workplace hazards in that they can be minimized and controlled to reasonably assure the safety of the people working near them.

Types of Radiation Hazards

The effects of excessive radiation exposure on the body are manifested in several ways: radiation sickness, radiation injury, and radiation poisoning.

Radiation sickness is caused by a massive overdose of penetrating external radiation. Symptoms include nausea, vomiting, diarrhea, malaise, infection, and hemorrhage.

Radiation injury consists of localized injurious effects, such as burns, skin lesions, and loss of hair. Generally these injuries are caused by overdoses of less penetrating external radiation and most often to the hands because contact is usually with the hands. Genetic damage is also a form of radiation injury.

Radioactive poisoning is illness resulting when dangerous amounts of certain types of radioactive materials enter the body, causing such diseases as anemia and cancer.

When we look at the foregoing, we realize that the radiation problem is made up of two separate problems: radiation originating from a source outside the body (external hazards), and by exposure resulting from radioactive materials which have been taken into the body (internal hazards). Precautions against one type of hazard will not be particularly helpful in protecting against the other type of hazard.

As a matter of fact, certain radioactive materials are no hazard at all outside the body. However, the same materials inside the body, in sufficient quantity, could cause radioactive poisoning. Therefore, it is fundamental to our understanding to realize the radiation problem is not one

problem, but two problems: the problem of external radiation, and the problem of internal radioactive poisoning. The precautions that we take depend upon which hazard is present.

External Radiation Hazards

X-Rays and Gamma Rays

X-rays and gamma rays constitute the most common type of external radiation hazard. When of sufficient energy, both are capable of deep penetration into the body. As a result, no radiosensitive organ is beyond the range of their damaging powers. The most common source of X-rays is, of course, the X-ray machine. Some X-rays are also generated as a by-product of certain atomic and nuclear reactions. Gamma ray sources include nuclear reactors, particle accelerators, and radio isotopes.

Beta Particles

Beta particles may or may not constitute an external hazard, depending upon their energy. Beta particles with enough energy to penetrate to the basal layer of the epidermis are considered external hazards, while those which are stopped by the outer layer of skin are not. Radioactive isotopes and high energy particles accelerators may be sources of beta radiation.

Alpha Particles

Because of their limited range, alpha particles do not constitute an external radiation hazard. Since the outer layer of tissue consists of cells already dead, external alpha radiation can do little damage in a biological sense.

Internally, however, alpha particles may severely damage the soft, unprotected tissue.

Single Exposure

A large dose of radiation over a short period of time is a greater biological insult than an equivalent dose spread over a longer period of time. It is impossible to determine what a fatal dose of radiation will be for a specific individual because we all vary in our resistance to attacks upon the body, whether it is by radiation, electricity, poison, injury, disease, etc. It is certain, however, that no human being can survive 1,000 roentgens of total body radiation delivery in a short space of time, that is, within 24 hours or less.

Both time and total radiation exposure are important. The effect of 1,000 roentgens delivered to a small portion of the body, just a third degree burn of the palm of the hand, has a different medical significance from a third degree burn of large area of the body.

Similarly, the ability of the body to withstand any insult is, of course, increased if the same amount of insult given to the body is spread out over a longer period of time. Whiskey can be poison, but many people, apparently without any demonstrable injury, can drink an ounce of

whiskey each evening before dinner over an extended period of time. If, however, a person attempts to consume a three months quota of whiskey in one sitting, he will probably die of alcohol poisoning because the body has not been given sufficient time to recover from the poison.

The dose it takes to kill one specific individual is not a good measure of the fatal dose to others because of individual differences. The term that is used in this field is the so-called median lethal dose, or LD/50. The LD/50 for penetrating external radiation is about 450 roentgens delivered to the total body in a short space of time (24 hours or less). Approximately 50% of those persons exposed at this level will die.

Continuous Exposures

Thus far, we have been thinking about a single incident of radiation exposure, one from which the man dies, gets sick and recovers, or receives no apparent effect at all; the sort of problem that is very similar to the ordinary injury situation. A man gets up on a rickety ladder, the ladder breaks and he falls. He can die, he can be injured and recover, possibly with some permanent disability, or he may be fortunate and suffer no ill effects at all. In any case, it is a discrete incident, with discrete medical effects.

There is another possibility with radiation: the problem of repeated small exposure to radiation over an extended period of time. What are the effects of this type of radiation exposure on the individual? What radiation safety levels must we have so that there will be no apparent effect and so that the hazard will be consistent with other industrial hazards? The following paragraphs address these questions.

There are two considerations which we must take into account. The first is the effect of the radiation on the individual himself. That is to say, what damage is done to the individual by repeated small dosages of radiation over a period which might conceivably extend from the time that he enters industrial employment until he retires, perhaps as much as 50 years later? The other consideration, with respect to society is the so called genetic effect, the problem of damage done to the genetic life stream of the population by the exposure of large numbers of individuals to ionizing radiation.

Complicating these questions are the facts that all of us receive background radiation from cosmic rays and naturally occurring radioactivity, that we are all exposed to a certain average level of radiation from medical and dental X-ray procedures, and that we all get a small increase in our background exposure from fallout from atomic weapons tests, regardless of who conducts them. The standards presently used for the regulation of radiation exposure in individuals take all of these factors into account.

It appears that high doses of radiation received over a relatively short time can have some effect on the life span of an individual. The National Academy of Sciences, National Research Council Report cites studies of a group of radiologists, some of whom received as much as 1,000 roentgens of X-ray exposure, which show on the average a life span 5 years shorter than that of

other physicians. There is as yet no conclusive evidence that shows dosages spread over a period of years have any life shortening effect. On the other hand, there is no evidence to indicate that there is a level of radiation exposure below which we can say there is no life-shortening effect at all.

Threshold and Non-Threshold Response

A fundamental question is whether there is a level of radiation exposure below which no harm is caused. This question also arises for other toxic chemicals and agents. With respect to radiation, one can illustrate the problem by plotting the dose against the effect. Very often one gets an S-shaped curve where the effect is zero until some certain dose is reached. This curve is shown as = dashed line. The point where the curve leaves the abscissa and begins to show increasing effect on the ordinate, is called the "threshold." Theoretically any dose lower than threshold would not be expected to cause any ill effect.

Another type of relationship between exposure and effect is possible. This is called a "non-threshold response" and is based on the presumption that any dose, however small, will have a measurable effect, varying in proportion to the size of the dose.

There is general agreement that genetic effects show a non-threshold behavior, that is, any dose of radiation will produce some genetic effect, although it may be considered minor and can be delayed for generations. There is controversy as to whether somatic effects are threshold or non-threshold.

It would seem prudent to err on the side of safety and, until there is further evidence to the contrary, to assume that any amount of radiation will produce some measure of harm, both somatically and genetically. It is for this reason, therefore, that UNC follows the concept of non-threshold response in the safety practices applied to the use of X-ray fluorescence devices.

Permissible Rate of Exposure

With all the foregoing taken into account, the supplement to the Bureau of Standards Handbook 59 sets the permissible rate of radiation exposure to industrial employees at an average of 5 mREM per year for each year after the age of 1. Note that the effect is to keep down not only the total amount of radiation exposure permitted to an individual in his lifetime, but to keep down the rate at which it accumulates, so that his exposure does not exceed 60 rem at the age of 30 and 110 rem at the age of 40.

Normally, incorrect handling or operating procedures are the cause of exposures in excess of 10 mREM per week. The normal exposure is less than 5 mREM per week. On a yearly basis the dosage has been less than 500 mREM which approaches the present background level.

Internal Radiation

The internal radiation exposure problem is much more complicated than the external radiation exposure problem. There are four possible ways to get radioactive materials into the body:

- * Breathing
- * Swallowing
- * Breaks in the skin
- * Absorption through the skin

How long does radiation material stay in the body? A high percentage of anything we inhale is immediately exhaled. Swallowed materials which are not soluble in the body's digestive system are discharged rapidly through the feces. If a material is soluble and is breathed into the body, it will go the blood stream. The blood stream then carries it around the body to the various organs, in effect offering the material to the organs.

The body is a chemical machine and each organ looks at the substance chemically. If an organ rejects the substance chemically, the blood stream carries it away. If no organ will accept the material, the blood takes it to the kidneys and the kidneys dispose of the material through the urinary system. If, on the other hand, an organ has a use for the material or if the organ thinks the substance looks like a material it can use, it accepts the substance. For example, the bones need calcium, and radium is chemically similar to calcium. Therefore, when the blood takes radium to those areas where the bone is building new bone tissue, the bone accepts the radium. Thus, the radioactive material is deposited in the bone.

Some chemical substances, such as sodium and potassium, are widely used throughout the body. Therefore, a radioactive form of one of these elements will be dispersed throughout the entire body. Other elements tend to concentrate in specific organs; e.g., iodine in the thyroid gland. The point to remember is that body organs react to a substance on the basis of its chemical nature only, without regard to whether or not the material is radioactive.

The radioactive half-life of the material is very important when considering its effects on the body. If a material has a radioactive half-life measured in fractions of a minute, it will be dissipated very rapidly. On the other hand, if the material has an extremely long half-life, possibly measured in the thousands of years, then the rate at which it is decaying is very slow. We are only interested in that radiation effect that takes place while we are still alive. Radiation being given off in our skeletons after we die is of no interest to us. For this reason, in general, materials with radioactive half-lives from 5 to 40 years are the most significant from the point of view of half-life.

The biological half-life of material is that period of time required for half of the material to be excreted from the body. Some materials are excreted quite rapidly from the body and will not be in the body long enough to do much harm. When we combine the radiological half-life with the biological half-life, we have the effective half-life of the material in the body.

Standards similar to the permissible levels of radiation exposure from external radiation hazards have been established for the permissible levels of various radioactive isotopes in the body. Working backward from the radioactive body levels, permissible air concentrations of the materials were established, because it is primarily by means of breathing and swallowing that the radioactive materials can get into the body on a continuous basis. These levels are stated in terms of microcuries per milliliter of air.

The hazard of absorption through the skin is handled by the use of suitable protective clothing or gloves. The introduction of radioactive material through wounds is avoided by standard safety techniques to prevent injury.

The radioactive material used in the UNC XRF devices is known as sealed sources. That is, the radioactive isotope is confined in a stainless steel capsule. As long as the containers do not allow the radioactive material to escape, there is no internal radiation hazard.

Additional Radiation Issues

Operation Theory

X-Ray fluorescence (XRF) lead-based analyzers employ a radioisotope to detect lead in paint. Understandably, people are concerned about radiation safety issues involving these type of instruments. How safe are XRF analyzers with regard to radiation exposure? If used on a daily basis, to what levels of radiation are lead inspectors exposed? What precautions should inspectors take to ensure their safety? What are the regulatory requirements for owning an XRF with a radioactive source element?

XRF's detect lead in paint utilizing the principle of X-Ray fluorescence. With this method of detection, a painted surface exposed to the radiation from an XRF analyzer will absorb and then fluoresce radiation back to the detector at specific levels. The XRF analyzer's Cobalt-57 source, when exposed by pulling the trigger, causes the paint to fluoresce. This energy is then detected in the probe and converted into electrical signals, separating out the element lead from other information. The analyzer then converts these "lead signals" into specific lead content in milligrams per square centimeters: mg/cm² as specified by the Department of Housing and Urban Development in Washington, D.C. The results are promptly displayed on an LCD display.

The Source

Cobalt-57 has a 276 day half-life; i.e. the source intensity decreases by one half every 276 days. If the XRF employs a 20 mCi (millicurie) Cobalt-57 source at manufacturing time, the source decays to 5 mCi's in 276 days and 2.5 mCi's in another 275 days, etc. This natural decay occurs whether the analyzer is used or not. To compensate for the weakening source, the XRF increases the time necessary to take a lead reading. hue analyzer can accurately detect lead using a 2.5 mCi source (approximately 28 months after purchase), however, the analysis time is about three-four times that of the first day of service.

Radioactive Exposure

Exposure to radioactivity is measured in milliREM. This measurement unit is based on the Time one is exposed, the Distance from the radioactive material, and the Shielding between you and the material. This is sometimes called the TDS. Age may also be factored in since the older one is the more radioactive exposure one can tolerate. The following comparisons may help understand the exposure levels an inspector may encounter:

1. A day at the beach will get you about 10 to 20 Micro REM's per hour.
2. In Colorado 30 to 50 Micro REM's per hour are common levels. (Less atmosphere to shield the solar furnace).
3. The maximum safe legal occupational industrial limit is 2,500 Micro REM per hour (which is expressed 2.5 Milli REM per hour).

Now, consider that the occupational industrial exposure limit is 2.5 Milli REM per hour. The Microlead 1 inspector then is exposed to only 4% of this safe maximum legal limit. Or, perhaps another way to look at this level: the inspector using a Microlead 1 will receive an exposure of at least 96% below the safe legal limit set by radiation control agencies!

Leak Testing

All agreement states (those not governed by the Nuclear Regulatory Commission) require bi-annual leak tests as a safety precaution. A leak test measures possible radiation contamination present on an XRF probe due to the Cobalt-57 source (leaking radiation). Performing a leak test is simple. One merely moistens a cotton swab in alcohol and wipes all exposed surfaces of the face of an XRF probe. The swab is provided in a leak test kit, with alcohol, a return plastic envelope, reporting forms which lists instrument serial number, source type and strength and address of the client. After the wipe test is performed the swab is placed in the envelope and sent to the testing laboratory for radioactive contamination counting. a report is returned immediately with the results. The laboratory usually also provides film badge services.

Licensing XRF Analyzers

Institutions responsible for documenting and controlling the procurement of radioactive devices from state to state. Therefore, radiation control regulations for Microlead 1 ownership also vary from state to state. One must remember that there are two types of states which are involved in the control of radiation: 1) Agreement State (those governing themselves under the guidelines of the NRC) and 2) NRC states (those governed directly by the Nuclear Regulatory Commission).

It should be noted that the NRC does not license or regulate Cobalt-57 for the levels encountered in the direct reading analyzers. Therefore, no licensing is required for these XRFs in NRC states. Agreement States, however, must approve the ownership of the instruments.

The following are some requirements which must be satisfied before ownership is allowed in Agreement States:

1. The analyzer must be stored in an unpopulated, secured, locked area.
2. A leak test must be performed every six months.
3. An appointed Radiation Safety Officer (RSO) must accept responsibility for the instrument and safety of those who use it. The RSO must have radiation safety instruction.
4. All personnel using the instrument must be properly trained in its use. Often Agreement States require the individuals to be specifically named on the license.

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Prepared by Pace Analytical Services, Inc.
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LABORATORY QUALITY ASSURANCE PLAN

Pace Approval

Ned Hudson 1/23/96
Vice President of Quality and Technical Director Date

President Date

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1.1 FOREWORD

Pace Analytical Services, Inc. is a privately held, full service environmental testing firm operating a system of 7 laboratories and multiple service centers nationwide. The Pace Minnesota laboratory was established in Minneapolis, Minnesota, on August 3, 1978. Since its inception, Pace has provided analytical services for clients ranging from federal and municipal government to industrial firms and private consulting groups. As an independently owned environmental laboratory company, with Pace, there is never the question of conflict of interest. Since the foundation of its first laboratory, Pace has always retained quality as its primary objective.

Pace Analytical Services, Inc. offers extensive services, including: bioassay for aquatic toxicity, air toxics, explosives, field services and mobile laboratory capabilities. The Pace system offers extensive capacity, and the ability to transfer work within the integrated system of laboratories assures that turn-around times are met. And, perhaps most importantly, geographic expansion has brought to Pace many valued and dedicated employees, with diverse interests and areas of expertise. There are nearly 400 people who contribute daily to the success of Pace.

Over the years, Pace has developed and continues to develop a strict system of QA/QC protocols, originally modeled after the USEPA Contract Laboratory Program (CLP) requirements. In addition, Pace has developed an advanced data management system, which is highly efficient and allows for flexible data reporting. Together, the two systems insure data reliability and timeliness.

The advances have not been limited to the company itself. Pace and Pace employees have been instrumental in the development of the environmental testing industry. Pace employees are among the founders and board members of the industry's two major associations: the American Council of Independent Laboratories (ACIL), and the International Association of Environmental Testing Laboratories (IAETL). Pace employees have delivered papers and published articles on laboratory management, Quality, and, most recently, on the newly developed model laboratory contract.

Today, Pace is not only keeping stride with the evolving industry, but is actively engaged in that evolution. Pace is operating a high productivity environmental testing laboratory in northern California near San Francisco. The laboratory was designed with process efficiency and quality as its major objectives. The results in enhancements to analytical, quality, and data management systems will be replicated in other Pace laboratories.

The strength of our company comes from how we are organized. We understand how important it is to develop long-term, on-going communication with our clients. With the client at the center, we have an integrated local support team which revolves around the client. The national system provides the local team with additional capacity, specialty services, and additional experts in all areas of the business, in order to ensure that requirements are met.

Our goal is to continuously combine our expertise in the laboratory with customized solutions to meet the specific needs of our clients. By providing the right chemistry and the right solution, Pace has become known as a leader in the industry with satisfied, long-term clientele.

1.2 CORPORATE PHILOSOPHY

The criteria for selecting an analytical laboratory have changed significantly in recent years. Increased environmental liabilities have altered the attitudes of users and providers of laboratory services. Quality is now the primary criterion.

Our philosophy at Pace, as it always has been, is to provide clients with the standards of service they require and deserve. It is a philosophy dedicated to providing:

Uncompromising Quality

Service Responsive to Clients' Needs

A Single Source of Comprehensive Services

Since the company's inception, Pace professionals have worked diligently to meet these goals. Our continued commitment to these standards remains the top priority at Pace.

1.3 THE MISSION OF PACE ANALYTICAL SERVICES, INC.

To be your Preferred Choice for Environmental Analytical Services in the Laboratory and in the Field

For our clients:

- by consistently meeting our commitments
- by delivering responsive service, on time, with high value
- by assuring data quality and technical excellence

For our employees:

- by offering equal opportunity for professional development
- by providing stimulating, participative, and safe work environments
- by valuing personal worth; encouraging excellence through recognition and reward

For our shareholders:

- by generating a return on investment which meets our obligations and promotes company objectives

For our suppliers:

- by offering long term relationships to those who support Pace's quality and business objectives

For our communities:

by being environmentally responsible and a good corporate citizen

Pace strives to be the preferred choice for all its stakeholders, by providing quality services with the highest level of professional and ethical standards.

1.4 CODE OF ETHICS

In carrying out its Corporate Mission, Pace requires its employees to abide by the highest professional, ethical standards. Employees will conduct their tasks according to the highest professional, technical and ethical standards applicable to their area of expertise. As such, Pace requires a commitment from all staff to abide by the principles set forth by the Company. This applies to all procedures, documented and undocumented, executed by employees. The following information summarizes the essential standards of ethical behavior required of Pace employees.

Simply stated, Pace's fundamental ethical principles are as follows:

- Each Pace employee is responsible for the propriety and consequences of his or her actions.
- Each Pace employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where Pace does business or seeks to do business.
- Employee conduct on behalf of the Company with clients, suppliers, the public and one another must reflect the highest standards of honesty, integrity and fairness.

Strict adherence by each Pace employee to this Code and to the Standards of Conduct is essential to the continued vitality of Pace. Therefore, compliance with and effective enforcement of the Code and Standards are key responsibilities of Pace management and will be addressed as elements of each employee's regular performance evaluation.

Failure to comply with the Code or Standards will result in disciplinary action up to and including termination and referral for civil or criminal prosecution where appropriate. An employee will be notified of an infraction and given an opportunity to explain as prescribed under current disciplinary procedures.

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2.3 LQAP DISTRIBUTION LIST

Individual copy distribution of this document may originate at any Pace facility and from the Corporate Office. When a copy of the Quality Assurance Plan is released, a designation is made on the cover as to whether the document is a controlled copy. Recipients of controlled copies will automatically be issued an updated version whenever revisions are made to the existing document. Each Pace location which distributes copies of this plan shall maintain a record of the name of the individual receiving the document, their affiliation, the number of the copy issued and the control status (i.e., controlled vs. uncontrolled).

3.0 INTRODUCTION

3.1 PROGRAM OBJECTIVES

The Pace Laboratory Quality Assurance Plan (LQAP) presents in specific terms the policies, organization, functions, and specific quality assurance (QA) and quality control (QC) requirements designed to achieve the data quality goals required for clients of Pace Analytical Services, Inc.. The U.S. Environmental Protection Agency's (U.S. EPA) QA policy requires a written and approved Quality Assurance Project Plan (QAPP) for every monitoring and measurement project mandated or supported by the U.S. EPA through regulations, contracts, or other formalized means not currently covered by regulation. Guidelines followed in the preparation of this plan are set forth in the document entitled "EPA Requirement for Quality Assurance Project Plans for Environmental Data Operations", EPA QA/R-5, Draft Interim Final (August 1994). Other documents that have been referenced for this plan include U.S. EPA Region IX Guidance for Preparing Quality Assurance Project Plans for Superfund Remedial Projects (September 1989); U.S. EPA's Guidance on Remedial Investigations Under CERCLA (June 1985); Guidance on Feasibility Studies Under CERCLA (June 1985); Compendium of Superfund Field Operations Methods (September 1987); Data Quality Objectives for Remedial Response Activities (March 1987); and Guidelines for Assessing and Reporting Data Quality for Environmental Measurements (January 1983).

This detailed plan has been prepared for use by contractors who perform environmental services to ensure that the laboratory produces data that are scientifically valid and defensible. The establishment and documentation of these procedures will also ensure that the data are collected, reviewed, and analyzed in a consistent manner.

3.2 STATEMENT OF POLICY

Pace Analytical Services, Inc. is committed to providing the highest quality product to our clients. The validity and reliability of the data generated are ensured by the adherence to rigorous quality assurance/quality control (QA/QC) protocols and a Total Quality Management (TQM) system. Pace emphasizes the application of sound QA/QC principles beginning with the initial planning of the project, through all the field and laboratory activities, and ultimately to the generation of the final report. The principles of concise data quality objectives, representativeness, completeness, comparability, precision and accuracy are applied.

The major elements of the overall Laboratory Quality Assurance Program at Pace are summarized below:

- The use of appropriate methodologies by technically competent, well-trained personnel with state-of-the-art instrumentation and equipment.

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- Adherence to well-defined standard operating procedures with emphasis on good laboratory and measurement practices.
 - Analysis and assessment of quality control samples including (but not limited to) matrix spike samples, matrix spike duplicate samples, duplicate samples, blanks and independent laboratory control standards.
 - Successful participation in external quality evaluation programs.
 - Accreditation by state, federal, and other applicable agencies for the work performed.
 - Internal and external auditing to ensure compliance to protocols and provide assessment of the analytical methods.

Pace is committed to providing the resources, including facilities, equipment and personnel, to ensure the adherence to these rigorous quality assurance/quality control protocols. Pace's quality assurance policy is based on the definition of quality as conformance to requirements; and further, on the premise that the requirements are governed by Company policies, government regulations and standard operating procedures. This commitment recognizes the need for data to be representative of the environmental conditions under consideration, and for data to be generated within a system of functions that is designed to meet applicable regulatory compliance criteria. To this end, Pace has developed a Quality Assurance (QA) Plan and maintains an ongoing QA Program. Our Quality Assurance Program contains provisions for establishing, maintaining and executing protocols which lead to results of known, appropriate and acceptable quality; documentation of these activities is an integral part of the QA program. No other concern will be permitted to interfere with the quality of data Pace provides to clients.

This manual describes the set of policies and principles which guide day-to-day operations. Specific protocols are included by reference and are contained in a series of volumes cited in Section 9.0 of this document.

This document describes ongoing laboratory operations for routine analyses performed at Pace. As such, the material contained within is subject to change. Changes may be based on specific project requirements or procedural system modifications geared towards operational process and quality improvements. At a minimum, this document is reviewed and updated on a yearly basis.

3.3 PURPOSE AND SCOPE

This manual details the quality assurance program in effect at all Pace Analytical Laboratories. It is meant to be a teaching tool and source of information for laboratory personnel. The Manual is divided into logical sections, each dealing with a different phase

of laboratory operation, yet all sections overlap and function together to form a complete quality assurance program. The Manual is based on Good Laboratory Practices, common sense, and industry-accepted standard analytical practices.

The Manual must be read and understood by all laboratory personnel as part of their training program. The Manual should also be referred to regularly as a source of information. A system of continuous updating is built into the Manual to allow it to change as laboratory conditions change or as new regulations are promulgated. This manual is a controlled document, which means that its identity, development, distribution, and status must be known and traceable at all times. All Pace laboratory personnel have access to a controlled copy.

Whenever a technician or analyst is in doubt as to proper procedures in a specific circumstance, the Manual should be consulted. Omissions or errors should be immediately reported to the Quality Assurance Officer, for corrective action. **IT IS THE RESPONSIBILITY OF EACH LABORATORY WORKER TO ENSURE THAT THE PROVISIONS OF THIS MANUAL ARE FOLLOWED.** Disagreement with specific requirements or knowledge of changes causing deviation from the procedures should be discussed with the immediate supervisor before further work is completed. Laboratory personnel are encouraged to comment on the Manual and make recommendations for more efficient procedures.

The latest revision of each section of the Manual is the applicable rule. Therefore, revisions will be announced to all laboratory personnel. An uncontrolled copy of the Manual is offered to clients and regulatory agencies as the definitive quality assurance program used at Pace.

3.4 QUALITY ASSURANCE DOCUMENTS

3.4.1 QA Manual

This document describes management policies related to operation of the analytical laboratories. It provides overall guidance regarding acceptable practices and discusses each element of the Quality Assurance Program. It functions as the Project QA Manual where no other Quality Assurance Project Plan, Statement of Work or other contractually mandated project plan has been specified. Adherence to the practices described in this manual is required of all employees. This manual may be revised and/or superseded only with the written authority of Vice President of Quality/Technical Director. Copies of this manual are controlled and distribution is administered by the Corporate Quality Office.

3.4.2 Standard Operating Procedures Manuals

All procedures related to sample collection, storage, preparation, analysis, disposal, data validation, data reporting and employee training and safety shall be contained in written Standard Operating Procedures (SOPs). Each SOP shall

contain the elements outlined in the Pace Corporate SOP ALL-P-001-A, Guidance Document for Pace Analytical Services, Inc. for the Preparation of Standard Operating Procedure Documents. All sections shall be structured in a step-wise manner using numbered sections. All record-keeping requirements shall be described at each step in the SOP. Examples of forms used shall be included as tables or figures and referenced within the text. Preparation of SOPs which have company-wide application will be the responsibility of the Corporate QAO. Analytical and evidentiary SOPs which are unique to an operating facility shall be prepared by designated personnel (e.g., analytical- section supervisor; evidentiary-laboratory QAO). SOPs shall be assigned a number from the Inventory List for SOPs maintained by the Corporate Quality Office or the Quality Assurance Department of the individual lab, as applicable. This number shall become part of the document control number when the SOP is accepted for implementation by Pace management. Laboratory SOPs shall be reviewed and approved by the relevant Section Supervisor (and Operations Manager for all SOPs related to analytical procedures) and the QA Officer, and submitted by the QA Department to the Operations Manager and the General Manager for approval prior to implementation.

3.4.3 Project QA Manuals

Project QA Manuals shall be implemented as required. These shall include such documents as Quality Assurance Project Plans (QAPPs). For those projects which require specific QA/QC criteria, a QAPP which has been approved by a regulatory agency, usually the EPA, is provided to Pace by the client. Often the analytical section of a QAPP is written by Pace for the client. In this instance, the QAPP is reviewed and approved by the appropriate Pace Quality Assurance Officer and the Pace Operations Manager.

3.4.4 Document Control, Distribution and Revision

In order that this document achieve the goals outlined in Section 3.2, it is necessary that each Pace laboratory employee be familiar with the current provisions of this document. It is also necessary that this document represent a consensus among Pace management and operational personnel as to the quality level desired and the means to that end.

Prior to its publication as a controlled document, this manual must be approved by the Vice President of Quality/Technical Director. To obtain such approval, the document proceeds through an iterative process of review and revision, involving the affected managers and their designated representatives. The signature page at the beginning of the manual represents acceptance.

Each time a revision is made to this manual, it must also be approved. The Vice President of Quality/Technical Director must approve each revision.

3.5 TERMS AND DEFINITIONS

<u>Accuracy:</u>	The degree of agreement between a measured value and the true or expected value.
<u>Aliquot:</u>	A measured portion of a sample taken for analysis.
<u>Analyte:</u>	The specific entity an analysis seeks to determine.
<u>Batch:</u>	A grouping of no more than twenty samples of similar matrix which are prepared and/or analyzed together with the same method and the same lots of reagents within the same time frame. A sample may be analyzed in a different analytical batch than the one with which it was prepared.
<u>Blank:</u>	A blank is an artificial sample designed to detect and/or monitor the contribution of analyte and non-analyte contamination, instrumental background and sample processing to the measurement system.
<u>Blind Sample:</u>	A sample submitted for analysis whose composition is known to the submitter but unknown to the analyst.
<u>CRDL</u>	Contract required detection limit.
<u>CRQL</u>	Contract required quantitation limit.
<u>Calibration:</u>	The process of establishing the relationship between instrument response and known, traceable quantities of analytes of interest.
<u>Calibration Check:</u>	Verification of the ratio of instrument response to analyte amount, a calibration check, is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards.
<u>Comparability:</u>	Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
<u>Completeness:</u>	Measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions. The equation for completeness is: $\frac{\text{\# of data points obtained}}{\text{\# of data points expected}} \times 100 = \% \text{ completeness}$

<u>Continuing Calibration:</u>	The process of analyzing standards periodically to verify the maintenance of calibration of the analytical system.
<u>Control Chart:</u>	A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.
<u>Control Limit:</u>	A range within which specified measurement results must fall to signify compliance. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that nonconforming data be investigated and flagged.
<u>Detection Limit:</u>	The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.
<u>Dry Weight:</u>	The weight of a sample based on percent solids. The weight after drying in an oven.
<u>Duplicate Analysis:</u>	A second measurement made on the same sample extract or digestate to assist in the evaluation of precision of analysis.
<u>Duplicate Sample:</u>	A second aliquot of the same sample that is treated the same as the original sample in order to determine the precision of the method.
<u>Environmental Sample:</u>	<p>An environmental sample or field sample is a representative sample of any material (aqueous, nonaqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows:</p> <ul style="list-style-type: none">Surface Water and Ground WaterDrinking Water - Delivered (treated or untreated) water designated as potable water.Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents.Sludge - Municipal sludges and industrial sludges.

Soil - Predominately inorganic matter ranging in classification from sands to clays.

Waste - Aqueous and nonaqueous liquid wastes, chemical solids, and industrial liquid and solid wastes.

Equipment

Blank:

Special type of field blank used primarily as a check on equipment decontamination procedures. After decontamination, the sampling equipment is rinsed with DI water and the water collected for analysis.

Field Blank:

A quality control sample that is used to assess the contamination effects on accuracy due to the combined activities of sampling and analysis. Typically, it is composed of analyte free matrix (e.g., deionized water) provided by the laboratory.

Field Sample:

A portion of material received by the laboratory to be analyzed, that is contained in single or multiple containers and identified by a unique field ID number.

Holding Time:

The elapsed time expressed in days from the date of sample collection by the field personnel until the date of its processing/analysis. For the Contract Laboratory Program, holding times start at the Verified Time of Sample Receipt by the laboratory. Holding time requirements are dictated by the method or QAPP.

Homogeneity:

The degree to which a property or substance is evenly distributed throughout a material.

Instrument

Detection Limit:

The minimum concentration of a substance that can be measured and reported on a specific analytical instrument with 99% confidence that the analyte concentration is greater than zero. The instrument detection limit is determined by replicate analyses of a standard solution prepared at the instrument. The instrument detection limit is generally more sensitive than the method detection limit because its determination does not include sample preparation steps.

**Initial
Calibration:**

The process of analyzing standards, prepared at specified concentrations, to define the quantitative response, linearity and dynamic range of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a continuing calibration do not conform to the requirements of the method in use or at a frequency specified in the method.

**Internal
Standards:**

Analytes added to every standard, blank, job control sample, matrix spike, matrix spike duplicate, and sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantitating target analytes. Internal standards are used as the basis for quantitation of the target compounds, and are generally applicable to organic analyses.

**Laboratory Control
Sample:**

A control sample of known composition spiked with a known concentration of analytes of interest. Aqueous and solid laboratory control samples are analyzed using the same preparation, reagents, and analytical methods employed for field samples.

LIMS:

Laboratory Information Management System, the Pace company-wide LIMS, has been identified as Environmental Project Information Controller (EPIC).

Lot:

A quantity of bulk material of similar composition processed or manufactured at the same time.

MRD:

Method Requirements Documents are written guidelines which outline a consistent definition of work performance for basic method compliance. The documents serve to interpret and define the subjective (vague) portions of the EPA's method for company-wide application. Each MRD is intended to establish a company-wide, baseline level of consistency for a single regulatory-derived method.

Matrix:

The predominant material of which the sample to be analyzed is composed.

Matrix Spike:

Aliquot of sample fortified (spiked) with known quantities of specified target compounds or analytes and subjected to the entire sample preparation and analysis procedure in order to assess the appropriateness of the method for the sample matrix by measuring recovery.

**Matrix Spike
Duplicate:**

A second aliquot of the sample that is treated the same as the original matrix spike sample. The relative percent difference between the matrix spike and matrix spike duplicate is calculated and used to assess analytical precision.

Method Blank:

An analytical control consisting of a blank matrix containing all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and contamination, and to demonstrate that this level does not exceed acceptance limits. Acceptable levels of contamination are defined by project specific data quality objectives.

**Method
Detection
Limit:**

The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. Method Detection Limits are determined using replicate spike samples prepared by the lab and taken through all preparation and analysis steps of the method. The method detection limit is calculated using the appropriate Student's t-parameter times the standard deviation of a series of spiked samples.

**Pace Reporting
Limit:**

PRLs were developed in conjunction with analysis codes (A-codes) for the EPIC LIMS. PRLs create uniformity across the company by establishing a standardized reporting limit by method to be utilized by all Pace laboratories. The PRL has been defined as the highest statistically derived MDL value for a particular method found at any of the Pace laboratory operations which are performing the method.

**Performance
Audit or
Evaluation:**

A process to evaluate the proficiency of an analyst or laboratory by evaluation of the results obtained on test materials in either a known, single or double-blind fashion.

Precision:

The measurement of agreement of a set of replicate results among themselves without any prior information as to the true result. Precision is assessed by means of duplicate/replicate sample analysis.

Protocol:

A stated plan that clearly defines the objectives, methods and procedures for accomplishing a task.

PQL:

The practical quantitation limit (PQL) is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.

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- QAPP:** A Quality Assurance Project Plan or QAPP is a project specific document that describes the policies, organization, objectives, functional activities, and specific QA and QC activities designed to achieve the data quality goals of a specific project.
- Quality Assurance:** A system of policies and procedures whose purpose is to ensure, confirm and document that the product or service rendered fulfills the requirements of Pace and its client. Quality Assurance includes quality planning, quality control, quality assessment (auditing), quality reporting and corrective action.
- Quality Control:** A system of checks and corrective measures, integrated with the activities that directly generate the product or service, that serves to monitor and adjust the process to maintain conformance to predetermined requirements.
- Reagent Grade:** Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
- Replicate Samples:** A second, separate sample collected at the same time, from the same place, for the same analysis, as the original sample in order to determine precision between the two samples.
- Reporting Limit:** The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the statistically determined MDL, but may be higher based on any of the above considerations. Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. Reporting limits are often set according to action or cleanup levels for a particular site or project which have been established in accordance with Data Quality Objectives (DQOs) under which the analytical work is to be processed.
- Rounding Rules:** If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded to 11.44. If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded to 11.45. If the figure following those to be retained is 5, and if there are no figures other than zeros

beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded to 11.44, while 11.425 is rounded off to 11.42. If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

**Sample Delivery
Group (SDG):**

A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently. A Sample Delivery Group is generally defined by one of the following, whichever occurs first:

- All samples within a project; or
- Every set of 20 field samples within a project; or
- All samples received within a 14-day calendar period

Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soil samples in one SDG, all water samples in another), at the discretion of the laboratory. Clients may establish different SDG classifications to meet project specific requirements.

Sensitivity:

Capability of methodology or instrumentation to discriminate between samples having differing concentrations or containing differing amounts of an analyte.

Split Sample:

A portion or subsample of a total sample obtained in such a manner that is not believed to differ significantly from other portions of the same sample.

Standard:

A substance or material, the properties of which are known with sufficient accuracy, to permit its use to evaluate the same property in a sample.

Standard Blank:

A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.

Standard Curve:

A standard curve is a curve which plots concentrations of known analyte standard versus the instrument response to the analyte.

**Standard
Operating
Procedure:**

A procedure adopted for repetitive use when performing specific measurement or sampling operation. It may be an industry accepted standard method or one developed by the user.

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- Surrogates:** When employed, these are compounds added to every blank, sample, matrix spike, matrix spike duplicate, lab control sample, and standard prior to any processing or preparation; used to evaluate analytical efficiency by measuring recovery. Surrogate compounds are not expected to be detected in environmental media, but are similar to the analytes of interest. Surrogates are generally utilized for organic analyses.
- Systems Audit:** An on-site inspection or assessment of a laboratory's quality control system.
- Traceability:** The ability to trace the source and accuracy of a material (i.e. standard) to a recognized primary reference source such as the National Institute of Standards and Technology (NIST) or USEPA. Also, the ability to independently reconstruct and review all aspects of the measurement system through available laboratory notebooks and documentation and reach the same results.
- Trip Blank:** This blank is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory pure water; any preservative used in the sample is added; and then the blank is stored, shipped, and analyzed with its group of samples.
- Validation:** The process by which a sample, measurement, method, or piece of data is deemed useful for a specified purpose as based upon the DQOs established for quality control measurements such as accuracy, precision, representativeness, and completeness.
- Warning Limits:** The limits (typically 2 standard deviations either side of the mean) shown on a control chart within which most results are expected to lie (within a 95% probability) while the system remains in a state of statistical control.

4.0 QA ORGANIZATION AND PERSONNEL

Pace Analytical Services, Inc. is a privately held, full service environmental testing firm operating an integrated system of seven laboratories, plus multiple service centers nationwide. Each laboratory within the system is set up as an individual entity with local management, but all share common systems and receive support from the corporate office. The chief function of the corporate office is to assist the system laboratories. The corporate office centralizes company wide accounting, business development, financial management, human resources development, information systems, marketing and quality activities. The organizational structure of the corporation is provided in Figure 4.1.

For efficient laboratory operation, it is important that all laboratory employees understand the operational structure, specific areas of responsibility and lines of authority within the organization.

It is equally important for laboratory personnel to understand that the structures of the Quality Organization may be separate from other laboratory operations but that the quality function is totally integrated into every aspect of laboratory operation. All laboratory personnel are responsible for knowing and following proper methods and standard operating procedures; recording quality control information required by those procedures in the proper location; and suspending analyses when quality control criteria are not met.

The organizational structure of a Pace Analytical Services, Inc. analytical chemistry laboratory is provided in Figure 4.2. The laboratory is managed by the General Manager. The Client Services (Sample and Project Management) and Quality Assurance Groups report directly to the General Manager.

Under the direction of the Laboratory Operations Manager, the technical staff of the laboratory is generally organized into the following functional groups:

- Sample Preparation - Organic
- Sample Preparation - Metals
- Wet Chemistry
- Metals Analysis
- GC Analysis
- GC/MS Volatiles Analysis
- GC/MS Semivolatiles Analysis
- Reporting/Data Validation

In some laboratory operations the Laboratory Operations Manager position may not exist; in such a case, the responsibilities of the position are distributed between the Organic and Inorganic Department Managers. Each group is headed by a Group Leader or Section Supervisor who is responsible for operations on a daily basis. Environmental chemists, analysts, laboratory technicians and laboratory assistants report to the Group Supervisors.

4.1 LABORATORY ORGANIZATION

It is the individual responsibility of each analyst and technician to perform their assigned tasks according to the applicable SOPs, QA Project Plans, Study Protocols, and Work Plans. This responsibility includes performing quality control analyses as specified in the method SOP and entering the QC data in the appropriate method control file system. The analyst shall report out-of-control results to the Group Supervisor/Leader.

Group Supervisors/Leaders shall ensure that analysts and technicians are instructed in the requirements of the Pace Laboratory QA Manual, site-specific QA Project Plans, SOPs, Protocols, and Work Plans for the analytical method or other procedure. Group Supervisors/Leaders shall review sample QC data at frequent intervals designed to ensure that QC analyses are being performed at the required frequency, that data are documented in the method control file system and that established corrective action procedures for out-of-control situations are followed and the results documented. It is the responsibility of the Group Supervisor/Leader to ensure that data have been validated and reported to the Operations Manager. Group Supervisors/Leaders shall report to the appropriate Manager.

The Operations Manager shall take overall responsibility for technical conduct, evaluation and reporting of all analytical tasks associated with each study. The Operations Manager ensures that approved procedures are documented and followed, that all data are recorded and verified and that all deviations from approved procedures are documented. The Operations Manager shall ensure that Group Supervisors/Leaders are instructed in the requirements of the Pace Laboratory QA Manual, study-specific QA Project Plans, SOPs, Protocols, and Work Plans. The Operations Manager provides guidance and assistance in the development of laboratory quality control procedures; approves quality control limits for methods; works with supervisors to bring out-of-control methods back to within established acceptance limits; and assists supervisors in correcting analytical problems revealed in QA audits. The Operations Manager shall report to the General Manager.

The Quality Assurance Department, under the direction of the Quality Assurance Officer, shall be responsible for conducting systems audits and inspections for compliance with this manual, SOPs and QA Project Plans or other project-specific protocols, maintaining the archives, maintaining historical files of all QA documents, reviewing QC charts, documenting findings and corrective actions, and reporting findings to management. The Quality Assurance Officer shall report directly to the General Manager of the Pace facility.

The Pace General Manager shall designate and replace if necessary, the Operations Manager, and is responsible for managing all activities related to laboratory services, including the Quality Assurance Program. The Pace General Manager shall ensure that there is a Quality Assurance Department, that personnel and other resources are adequate, that personnel have been informed of their responsibilities, that deficiencies are reported to the Operations Manager and that corrective actions are taken and documented. Any significant changes to written SOPs shall be authorized in writing by

either the General Manager or the Operations Manager and the Quality Assurance Officer of the Pace location.

4.2 DESCRIPTION OF RESPONSIBILITIES

Individuals involved with implementing procedures outlined in the LQAP have the following quality related duties and responsibilities:

4.2.1 Pace's Vice President of Quality is responsible for assisting in the development, implementation and monitoring of quality programs for the company. Responsibilities include:

1. Review and direct implementation of appropriate analytical Standard Operating Procedures.
2. Formulate and implement analytical product deliverables.
3. Provide technical direction to laboratories regarding existing and new analytical operations.
4. Provide leadership and direction to the laboratory Quality Assurance Officer.
5. Perform laboratory and project specific audits.
6. Assist in development, implementation, and monitoring of appropriate training programs.

4.2.2 The Pace Laboratory General Manager is responsible for overall laboratory operations. Specific responsibilities that relate to quality assurance are:

1. Implement the QA Program within the specific laboratory.
2. Regularly determine the effectiveness of the QA program.
3. Supervise quality control activities.
4. Approve laboratory-specific attachments to the QA manual and project-specific Quality Assurance Project Plans.
5. Recommend changes in the QA Program to the laboratory Quality Assurance Officer.
6. Maintain a current distribution list for QAPPs and generic LQAP.
7. Approval oversight for all reports issued by the laboratories.
8. Serve as the focal point for the reporting and disposition of all nonconformances.
9. Maintain a current laboratory organization chart.

4.2.3 The Pace Laboratory Project Manager is the lead person within the laboratory for direct oversight of all aspects of a specific project. Specifically, some of the project manager's responsibilities are:

1. Establishing direct dialogue with the client pertaining to project requirements, including methodology, TAT, technical information, etc.
2. Arranging bottle orders and shipment of sample kits to client.

Date: 12/22/95

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3. Verifying log-in information relative to project requirements and field sample chain of custodies.
4. Interfacing with laboratory operations staff to update and set job priorities.
5. Updating clients on job status.
6. Providing verbal and facsimile results to clients.
7. Assisting laboratory staff with report preparation.
8. Working with clients, laboratory staff, and other appropriate Pace staff to develop project statements of work or resolve problems of data quality, turnaround, or completeness.

4.2.4 The Pace laboratory Quality Assurance Officer (QAO) reviews all aspects of QA/QC for the laboratory. The duties of the laboratory QAO are to:

1. Assist the project manager in specifying QA/QC procedures to be used during the project.
2. Execute QC procedures and techniques to ensure that the laboratory achieves established standards of quality.
3. Evaluate data quality and maintain records on related QC charts and other pertinent information.
4. Monitor laboratory activities to determine conformance with authorized QA policy, and to implement appropriate steps to ensure adherence to QA programs.
5. Coordinate with the client's representative concerning external audits.
6. Review performance evaluation results.
7. Assist in development and implementation of appropriate training programs.

4.2.5 The Operations Manager oversees day-to-day production and quality activities of both inorganics and organics laboratory section providing wet chemistry, metals prep, metals, pesticide/PCB, volatiles and semivolatiles analyses. The specific duties of the Operations Manager are:

1. Provide supervision of laboratory operations.
2. Implement the laboratory quality assurance plan.
3. Ensure proper scheduling and execution of testing programs.
4. Ensure that quality assurance and quality control criteria of analytical methods and projects are satisfied.
5. Assess data quality and take corrective action when necessary.
6. Notify the project team of specific laboratory nonconformances and changes.
7. Approve and release technical and data management reports.
8. Ensure that analysts and technicians maintain sample custody in the laboratory.
9. Approve project specific laboratory quality assurance plans.
10. Coordinate management of projects through technical supervisors.

4.2.6 Group Supervisors/Leaders affect data quality by fulfilling responsibilities to:

1. Serve as the lead analyst within the specific sections.
2. Lead the training of analysts in laboratory operations and analytical procedures.
3. Organize and schedule analyses with consideration for sample holding times.
4. Implement data verification procedures.
5. Assign duties to analysts as data validators.
6. Prepare data summaries for review by the Laboratory Operations Manager.
7. Evaluate instrument performance and supervise instrument calibration and preventive maintenance programs.
8. Report noncompliance situations in regard to the project to the Laboratory Managers or laboratory Quality Assurance Officer, as appropriate.

4.2.7 Analysts are responsible for tasks identified in the scope of work. They perform the laboratory technical activities within these tasks. The duties of analysts are to:

1. Assist in planning for each phase of their tasks and in defining objectives and activities.
2. Respond to work plan revisions related to their tasks.
3. Advise the project manager of progress, needs, and potential problems of their tasks.
4. Train and qualify alternate analysts in specified laboratory QC and analytical procedures.
5. Verify that laboratory QC and analytical procedures are being followed as specified.
6. Review sample QC data at least daily. This includes examination of raw data such as chromatograms (and checking of calculations for a minimum of 10% of the samples analyzed) as well as an inspection of reduced data, calibration curves, and laboratory notebooks.
7. Inform project managers if the daily review indicates a decline in data quality and implement corrective action.

4.2.8 The Sample Custodian serves as sample coordinator for the entire laboratory. Responsibilities are to:

1. Sign for incoming field samples and verify the data entered on the chain-of-custody forms.
2. Enter the sample information into the computerized Laboratory Information Management System for tracking and reporting.
3. Generate computerized sample analysis and data entry forms (SADEF).

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4. Transfer samples and tracking forms to laboratory project analysts.

4.3 TRAINING AND ORIENTATION

Each new permanent employee receives a four part orientation: a human resources orientation, a safety department orientation, a quality assurance department orientation, and a supervisory orientation. The human resources orientation involves matters of immediate personal concern such as benefits, salary, and company policies. The safety department orientation is an in-depth examination of the Pace Chemical Hygiene Plan and safety program, which are consistent with the requirements of OSHA's Hazard Communication Program (29 CFR 1910.1200). The Quality Assurance orientation provides the new employee with information on the Pace QA program through a brief introduction to the QA manual and SOPs, acceptable record keeping practices, and the individual's responsibility with respect to the quality assurance program. The new employee's Group Supervisor provides the employee with a basic understanding of the role of the laboratory within the structure of Pace, Inc. and the basic elements of that individual's position within the laboratory.

Temporary employees receive the same orientation as permanent staff with the exception of the Human Resources orientation. The training of a new employee concentrates on his/her scientific background and work experience to provide the employee with a level of competence so that the individual will be able to function within the defined responsibilities of his/her position ASAP. Training is a process used to assist laboratory personnel in their professional development. The training techniques utilized include:

- On-the-job training
- Lectures
- Programmed learning
- Conferences and seminars
- Short courses
- Specialized training by instrument manufacturers
- Participation in check-sample or proficiency sample programs.

Group Supervisors shall be responsible for providing documentation of training and proficiency for each employee under their supervision. The Training Documentation File indicates what procedures (SOPs) a technician is capable of performing either independently or only with supervision. The files shall also include examples demonstrating performance of passing QC samples. The Group Supervisor is responsible for keeping a training documentation file for each person under their supervision which is updated and current. The QA department shall maintain a file for each technical employee. These files shall include a current curriculum vitae or resume.

4.4 LABORATORY SAFETY

Sample receiving areas and laboratories shall be equipped with suitable hoods, respirators, protective clothing and eye wear, gloves, barrier creams and or other

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measures to prevent or minimize staff contact with hazardous substances. Safety equipment such as eyewash stations, drench showers, spill adsorbents and neutralizers, fire extinguishers, first aid materials, and breathing oxygen shall be available.

As a matter of policy, Pace shall not accept known initiator explosives, known dioxin-contaminated materials or unusual biohazard materials except where a specific Pace facility has been designed to safely handle high hazard samples. Pace shall accept nitroaromatics and nitroamines providing that the client makes provisions for disposal of samples with a positive explosive identification.

A laboratory staff member shall be designated as Safety Manager by the General Manager. The Safety Manager prepares and maintains safety-related SOPs, conducts safety and occupational health orientation, training and review sessions as required, and maintains up to date familiarity with safety and occupational health issues pertinent to the laboratory.

The Safety Manager prepares and maintains educational programs as required to comply with state and federal "right to know" legislation.

The Safety Manager or his designee shall conduct an orientation session with each new staff member to familiarize him/her with routine and emergency safety procedures and equipment. Eye protection and a lab coat shall be issued to the employee. A respirator will be issued, as required, after respiratory protection training. A tour of the laboratory shall be conducted. During the tour, needs for eye, skin, and respiratory protection shall be discussed as well as the use of safety glasses, face shields, goggles, partial and full-face respirators, ventilated work areas, fume hoods, gloves, barrier creams, and Tyvek coveralls. The location of eye wash stations, drench showers, fire extinguishers, and first aid equipment shall be shown to the employee and their use shall be described or demonstrated. Fire and spill notification, emergency procedures, and evacuation stations shall be taught during this session. The orientation concludes with an introduction to potential chemical hazards and the Material Safety Data Sheets (MSDS). MSDS shall be made available for review.

Employees shall be responsible for their own safety. The Operations Manager and Group Supervisors may require that certain levels of protective equipment be worn when in their judgment it is appropriate. Failure of an employee to wear required protective equipment will result in immediate disciplinary action.

4.5 SECURITY AND CONFIDENTIALITY

Three tiers of security shall be maintained within Pace for the purpose of controlling external influences on samples, analytical processes, and data. These security procedures help ensure the completeness, representativeness, accuracy, and precision of analytical results.

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The first tier of security maintained shall be controlled access to laboratory buildings. Exterior doors to laboratory buildings shall remain either locked or continuously monitored by a Pace staff member. Keyless door-lock combinations (and computer access codes/logins) shall be changed every time an employee terminates employment at Pace. Posted signs shall direct visitors to the reception office and mark all other areas as off limits to unauthorized personnel. All visitors to the facilities must sign the Visitor's Logbook maintained by the receptionist. All visitors shall be accompanied by a staff member during the duration of their stay on the premises. The staff member shall escort the visitor back to the reception area at the end of his/her visit where he/she shall sign out in the Visitor's Logbook. Prior to departure of the last staff member at the close of each day, all windows shall be locked and all doors checked and locked by the last staff member.

The second security level shall be within the facility and may be designated as required by the Operations Manager in consultation with the General Manager. Individual Operations Manager or Group Supervisors may close specific areas under their responsibility to entry by unauthorized persons. A list of authorized persons shall be prepared and signed by the General Manager. "Closed Areas" shall be designated by prominent postings at all points of access.

The final tier of security shall be comprised of specific secure areas for sample, data and client report storage which shall be lockable within the facilities, and to which access shall be limited to specific individuals or their designees. Security of sample storage areas shall be the responsibility of the Sample Manager. Security of samples and data during analysis and data reduction shall be the responsibility of Group Supervisors and Operations Manager. Security of client report archives shall be the responsibility of the Quality Assurance Officer or an appropriate designee. These secure areas will be locked whenever these individuals or their designees are not present in the facility.

Designated laboratory sample storage locations are designed to limit access to authorized personnel only, and provisions for lock and key access shall be provided. No samples are to be removed without authorization, which consists of having a work list requesting analysis on an aliquot. No samples are to be removed without filling out the associated chain-of-custody records.

Standard business practices of confidentiality shall apply to all documents and information regarding client analyses. Specific protocols for handling confidential documents are described in Pace SOPs. Additional protocols for internal identification of samples and data by number only shall be implemented as required under contract-specific Quality Assurance Project Plans.

Figure 4.1

Pace Analytical Services, Inc. Organizational Structure

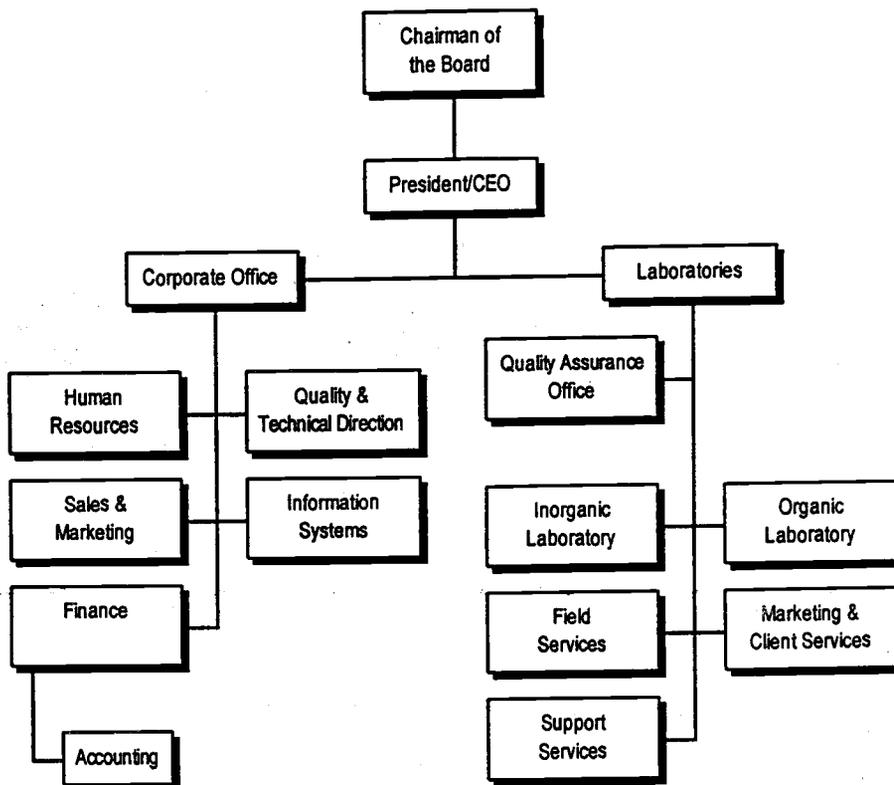
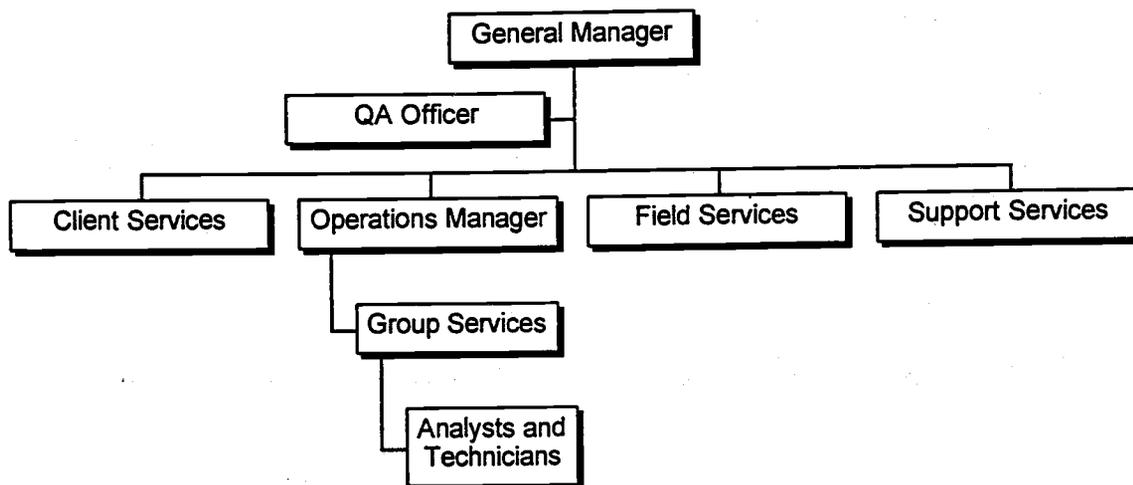


Figure 4.2

**Pace Analytical Services, Inc. Laboratory
Organizational Structure**



5.0 QUALITY ASSURANCE OBJECTIVES

The purpose of this Quality Assurance Plan is to define procedures for the documentation, evaluation, validation, and reporting of data. The objective is to provide a uniform basis for sampling, sample handling, instrument maintenance and calibration, methods control, performance evaluation and analytical data generation and reporting. Specific procedures to be used for sampling, chain of custody, calibration of field instruments (pH, conductivity meters, etc.), laboratory analysis, reporting, internal quality control, audits, preventive maintenance, and corrective actions are described in specific sections of this plan. This section addresses the objectives of precision, accuracy, representativeness, completeness, and comparability (PARCC) which are used to assess whether the data meet the established DQOs (Data Quality Objectives), that are based upon the intended end use of the data.

The quality assurance objective of the laboratories is to provide data of known and documented quality. Data quality is assessed by precision, accuracy, representativeness, completeness, and comparability. The QA protocols used in the laboratories for the majority of analyses performed are taken from the following sources: EPA Contract Laboratory Program's Statement of Work (Organics and Inorganics), 40 CFR 136 methodologies, and SW 846 methodologies which contain detailed descriptions of the quality control measures routinely employed by Pace Analytical Services, Inc..

As stated, the objective of the Quality Assurance Program for the laboratory is to provide data of known quality. To accomplish this, Pace will:

- Maintain an effective, on-going QA/QC program that measures and verifies laboratory performance.
- Provide a quality organization independent of the pressures of project performance with the responsibility and authority for auditing and recommending corrective action.
- Provide a quality organization with clear paths of communication with management.
- Provide sufficient flexibility to allow controlled changes in routine methodology to meet client specific data requirements contained in project-specific quality plans.
- Recognize as soon as possible and provide correction for any factors which adversely affect data quality.
- Monitor operational performance of the laboratory on a routine basis and provide corrective action as needed.
- Maintain complete records of sample submittal, raw data, laboratory performance, and completed analyses to support reported data.

5.1 LEVEL OF QA EFFORT

The reliability of data generated in the laboratory will be evaluated at the 99% confidence level (mean +/- 3 standard deviations) for control and at the 95% confidence level (mean +/- 2 standard deviations) for warning. Precision of analyses will be evaluated using sample duplicates and matrix spike duplicates. Analytical accuracy will be monitored using recovery of analytes from surrogate spikes, matrix spikes, EPA reference check standards (when available) and/or lab control samples, and Performance Evaluation (PE) samples.

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5.2 PRECISION AND ACCURACY

Precision measures the reproducibility of repetitive measurements. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Analytical precision is a measurement of the variability associated with duplicate (two) or replicate (more than two) analyses of the same sample in the laboratory and is determined by analysis of laboratory duplicates. Total precision is a measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate or replicate field samples and incorporates variability introduced by both the laboratory and field operations. Precision data must be interpreted by taking into consideration these possible sources of variability. Duplicate (two) samples or spiked samples are analyzed to assess field and analytical precision as required under certain programs (e.g., Air Force tasks), and the results are assessed using the relative percent difference (RPD) between duplicate measurements. Precision objectives are presented for each analytical method in the corresponding Pace Standard Operating Procedure (SOP).

Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systematic error. It therefore reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is typically measured by determining the percent recovery of known target analytes that are spiked into a field sample (a surrogate or matrix spike) or reagent water (laboratory control sample [LCS] or QC check sample). Surrogate compound recovery is reported and is used to assess method performance for each sample analyzed for volatile and semivolatile organic compounds. The stated accuracy objectives apply to spiking levels at least five times the method detection limits (MDLs) or background concentration.

Both accuracy and precision are calculated for analytical batches, and the associated sample results must be interpreted by considering these specific measures. Calculation of precision and accuracy to measurement sample results is discussed in the QC section of each SOP.

The QA objectives for precision and accuracy are to achieve the QC acceptance criteria specified in the proposed analytical procedures. For the organic and inorganic procedures, the precision and accuracy guideline requirements are specified in the individual methods.

Field blanks and duplicates are collected and analyzed to assess field sampling activities. The results check procedural contamination and/or ambient conditions at the site.

Due to the extensive number of organic parameters and potential matrices, the development of precision and accuracy objectives and control limits for every matrix is difficult. This is typically done with (1) matrix spike and matrix spike duplicate compounds which are added to selected samples before extraction and analysis, and/or

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(2) surrogate spike compounds which are added to every sample, before extraction and analysis. Although the surrogate and matrix spike analyses do not provide statistically valid statements about precision and accuracy for every compound in a sample, they do give the data reviewer enough information to make judgments about precision and accuracy on a sample-by-sample basis.

Inorganic precision and accuracy data are determined by using duplicate or matrix spike duplicate samples (precision), matrix spike and laboratory control samples (accuracy). The following procedure is used:

For a duplicate (or matrix spike duplicate) sample analysis, at least one duplicate (or MSD) sample is analyzed per sample matrix type (e.g., water, soil) and concentration (e.g., low, medium) per batch of samples or for each 20 samples received, whichever is more frequent, or as specified by state/project requirements. Samples identified as field blanks can NOT be used for duplicate (or MSD) samples analyses. If two analytical methods are used to obtain the reported values for the same element for a batch of samples (i.e., ICP, GFAA), duplicate samples will be run by each method. The relative percent difference (RPD) for each component is calculated for later use during data assessment.

The QC limits for accuracy and precision are developed based upon laboratory derived data. When applicable, interlaboratory control limits established by the EPA CLP are used to judge acceptability of data generated by the laboratories. Where EPA acceptability criteria does not exist for a given method being utilized for the first time, the laboratories will establish control limits derived from a minimum of four data points. Until verified by a statistically significant data population, the control limits will be considered as advisory limits only and will not automatically initiate a rerun or reanalysis criteria if they are not met.

Representative QC objectives for selected organic parameters are listed in Tables 5.1 to 5.6. Similarly representative QC objectives for selected metal and inorganic parameters are listed in Tables 5.7 to 5.8. Generally, QC acceptance limits are laboratory specific, having been statistically derived from an individual laboratory's data. QC objectives for a specific laboratory will be included in a project specific QAPP or for general information as a facility specific addendum to this document.

5.3 COMPLETENESS

Completeness is a measure of all information necessary for a valid scientific study. For completeness, it is expected that the methodology proposed for chemical characterization of the samples collected will provide data meeting QC acceptance criteria following standard laboratory data review and validation for at least 95% of all samples collected. Completeness may also be defined as a comparison of the number of tests successfully completed (with acceptable QC) to the number of tests requested. Discrepancy reports are completed to provide explanation when QC criteria are not met.

Every attempt will be made to generate completely valid data. However, it is recognized that some samples will exhibit highly contaminated matrices necessitating multiple

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analyses and/or extensive dilutions. As a result of these atypical applications, recoveries and MDLs or RIs, as applicable, may be deemed questionable based on internal QC results by the external data validation process. The objective will be to have 95% completeness on samples unaffected by matrix interferences. For uncontaminated background samples and first time samples not showing interferences, completeness should be 100% with a mandatory requirement for reanalysis of these critical samples if objective is not met.

5.3.1 Random Error

EPA has established (preamble to 40 CFR Part 136, Vol. 49, No. 209, October 26, 1984) that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.

The number of compounds present in numerous EPA methods (e.g., GC/MS methods 8240B and 8270B, and metals included in ICP method 6010A) increases the probability that one or more analytes will not meet acceptance criteria to significantly more than the 5% per analyte frequency. The number of target analytes included in these tests can be used to show that a minimum of four to seven target analytes will exceed the control limits established for these methods due to the statistical probability for random error. The establishment of QC criteria that are not consistent with the measurement of the quality objectives for which they are intended should be discouraged.

5.4 REPRESENTATIVENESS

Representativeness is a qualitative element that is related to the ability to collect a sample that reflects the characteristics of that part of the environment that is to be assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project. It is specifically addressed in the work plan.

Representativeness is a measure of how closely the measured results reflect the actual concentration or distribution of the chemical compounds in the sample. Sample handling protocols (e.g., collection, storage, preservation and transportation) have been developed to preserve the representativeness of the samples. Proper documentation will establish that protocols have been followed and sample identification and integrity assured. Every attempt will be made to ensure that the aliquots taken for analysis are homogeneous and representative of the samples received.

5.5 COMPARABILITY

Comparability is also considered during preparation of a site specific work plan. The objective of comparability is to ensure that results of similar activities conducted by different parties are comparable. This often involves the use of two independent laboratories on a project or site, whereby the second laboratory is used to confirm a pre-

established percentage of sample analyses. Pace uses EPA-approved or other methods and procedures to ensure comparability with data from previous or following studies. Pace participates in external and interlaboratory performance evaluation (PE) studies as additional means of establishing comparability in the laboratory.

5.6 TRACEABILITY

Traceability is the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis. Refer to the sections on sample custody and records management for more specifics on Pace procedures.

5.7 QUALITY ASSURANCE PROJECT PLAN EXCEPTIONS

Due to the unknown nature of environmental samples prior to analysis, Pace has minimal control over analytical and quality control complications which arise from unique sample matrix conditions. These conditions may include such items as: highly concentrated samples containing target compounds of interest and/or non-target components; extremes in sample pH, viscosity, and solubility; and high organic content (both natural and synthetic). Each of these conditions presents a variety of challenges to the laboratory.

Most often these extremes in sample matrix composition necessitate the laboratory to employ dilution techniques in order to change the sample state into one which can be analyzed by the desired protocol. Unfortunately, dilution techniques raise reporting limits (RLs) and often adversely impact the surrogate standard and matrix spiking acceptance criteria.

The laboratory has the responsibility to clearly identify cases where matrix interferences preclude the generation of "compliant" data. This is done by demonstrating through reproducibility (i.e., reanalysis of the affected sample) that the quality control measurement failure resulted from unique sample matrix conditions beyond the control of laboratory, and not as a result of laboratory error. For example, in situations where the surrogate standard recoveries fall outside of control limits, samples are re-extracted and/or re-analyzed. Similar "non-compliant" results in the reanalysis indicate that it is something inherent to the sample which prevented the laboratory from reporting results deemed method compliant under data validation criteria.

Analytical projects containing particularly "dirty" samples (i.e., highly contaminated) will often fail to meet pre-established QA completeness goals (set forth in the QAPP) when prior site history does not reveal the potential for excessive values. Again, while the laboratory performs all analytical testing by the prescribed protocols, the results obtained may not meet validation criteria as a result of elevated RLs or the frequency at which surrogate and matrix spikes failed to meet acceptance limits. In cases where the laboratory is unable to meet QC criteria because of sample matrix complications beyond their control, results which are flagged "qualified" or "rejected" by data validation guidelines are often still "useable" by the end user of the data.

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Pace is committed to adhering to method requirements and program quality control applications as established by our client and will work rigorously to provide data which is of the highest quality possible. However, the uncertainties associated with environmental samples do not allow Pace to assume responsibility for conditions beyond our reasonable control which directly impact the "validity" versus the usability of the associated analytical data generated.

5.8 PERSONNEL QUALITY OBJECTIVES

Pace is committed to the philosophy that quality operations result from quality planning, design, and work performance by skilled operational personnel. Pace's policy is to perform its varied types of technical work in accordance with standard quality assurance practices such as Good Laboratory Practices (GLP) and the EPA Contract Laboratory Program (CLP), as well as other appropriate regulatory agency guidelines and requirements. Each laboratory within Pace has a Quality Assurance Officer responsible for maintenance of standard operating procedures, laboratory audits, performance evaluations, federal and state certifications and quality assurance documentation.

Each laboratory worker is responsible for checking standard operating procedures when necessary; following these procedures during routine analyses; recording quality control information required by those procedures in the proper location, and taking appropriate corrective action including suspending analyses when quality control criteria are not met.

Table 5.1

**Representative Spike Recovery Acceptance Criteria for
Volatile Organic Analysis by Methods 8010B and 8020A**

<u>Analyte</u>	MS %R (SW-846) <u>Water/Soil</u>	LCS %R (Statistical)*	
		<u>Water</u>	<u>Soil</u>
Method 8010A			
1,1-Dichloroethene	28-167	63-145	47-137
Chloroform	49-133	70-137	66-140
Carbon tetrachloride	43-143	72-138	63-143
1,2-Dichloroethane	51-147	74-138	60-157
Trichloroethene	35-146	75-147	63-152
Tetrachloroethene	26-162	79-134	72-138
Chlorobenzene	38-150	76-126	65-136
1,4-Dichlorobenzene	42-143	70-123	64-127
Method 8020			
Benzene	39-150	74-135	43-156
Chlorobenzene	55-135	74-130	80-126
1,4-Dichlorobenzene	42-143	70-125	75-120
		%R (Statistical)*	
<u>Surrogate</u>	<u>Water</u>	<u>Soil</u>	
Bromochloromethane	54-115	60-109	
1,4-Bromofluorobenzene	70-125	68-112	

* Statistically derived acceptance limits will vary by individual laboratory operation.

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Table 5.2

**Representative Spike Recovery Acceptance Criteria for
Purgeable Petroleum Hydrocarbon Analysis
(California LUFT Method)**

<u>Analyte</u>	<u>MS %R</u>	<u>LCS %R</u>	
	<u>(SW-846)</u>	<u>(Statistical)*</u>	
	<u>Water/Soil</u>	<u>Water</u>	<u>Soil</u>
Benzene	39-150	80-110	49-103
Toluene	46-148	80-110	49-103
Ethylbenzene	32-160	83-113	52-106
Xylene	32-160	83-113	56-104

<u>Surrogate</u>	<u>%R</u>	
	<u>(Statistical)*</u>	
	<u>Water</u>	<u>Soil</u>
Bromofluorobenzene	70-113	51-120

Table 5.3

**Representative Spike Recovery Acceptance Criteria for
Extractable Petroleum Hydrocarbon Analysis
(California LUFT Method)**

<u>Analyte</u>	<u>MS %R</u>	<u>LCS %R</u>	
	<u>(Advisory)</u>	<u>(Statistical)*</u>	
	<u>Water/Soil</u>	<u>Water</u>	<u>Soil</u>
Diesel	50-150	62-122	57-123

<u>Surrogate</u>	<u>%R</u>	
	<u>(Statistical)*</u>	
	<u>Water</u>	<u>Soil</u>
2-Fluorobiphenyl	53-131	38-128
o-Terphenyl	41-149	37-169

* Statistically derived acceptance limits will vary by individual laboratory operation.

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Table 5.4

**Representative Spike Recovery Acceptance Criteria for
Pesticides/PCB Analysis by Method 8080A and CLP SOW**

<u>Analyte</u>	<u>MS %R</u> <u>(SW-846)</u>	<u>MS %R</u> <u>(CLP)</u>		<u>LCS %R</u> <u>(Statistical)*</u>	
	<u>Water/Soil</u>	<u>Water</u>	<u>Soil</u>	<u>Water</u>	<u>Soil</u>
Aldrin	42-122	40-120	34-132	32-128	32-104
gamma-BHC	32-127	56-123	46-127	33-135	31-103
DDT	25-160	38-127	23-134	39-135	23-125
Dieldrin	36-146	52-126	31-134	37-139	36-108
Endrin	30-147	56-121	42-139	42-138	37-115
Heptachlor	34-111	40-131	35-130	34-130	34-106

<u>Surrogate</u>	<u>%Recovery</u> <u>(SW-846 Statistical)*</u>		<u>%Recovery</u> <u>(CLP Advisory)</u>	
	<u>Water</u>	<u>Soil</u>	<u>Water</u>	<u>Soil</u>
Tetrachloro-m-xylene	31-121	32-108	60-150	60-150
Decachlorobiphenyl	29-153	56-125	60-150	60-150

Table 5.5

**Representative Spike Recovery Acceptance Criteria for
Volatile Organic Analysis by Method 8240B and CLP SOW**

<u>Analyte</u>	<u>MS %R</u> <u>(SW-846)</u>	<u>MS %R</u> <u>(CLP)</u>		<u>LCS %R</u> <u>(Statistical)*</u>	
	<u>Water/Soil</u>	<u>Water</u>	<u>Soil</u>	<u>Water</u>	<u>Soil</u>
1,1-Dichloroethene	59-155	61-145	59-172	63-123	54-126
Trichloroethene	71-157	71-120	62-137	77-119	77-113
Chlorobenzene	37-160	75-130	60-133	81-117	85-115
Toluene	47-150	76-125	59-139	79-115	84-114
Benzene	37-151	76-127	66-142	83-119	83-119

<u>Surrogate</u>	<u>%Recovery</u> <u>(SW-846)</u>		<u>%Recovery</u> <u>(CLP)</u>	
	<u>Water</u>	<u>Soil</u>	<u>Water</u>	<u>Soil</u>
Toluene-d ₈	88-110	81-117	88-110	84-138
4-Bromofluorobenzene	86-115	74-121	86-115	59-113
1,2-Dichloroethane	76-114	70-121	76-114	70-121

* Statistically derived acceptance limits will vary by individual laboratory operation.

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Table 5.6

**Representative Spike Recovery Acceptance Criteria for
Semivolatile Organic Analysis by Method 8270B and CLP SOW**

Analyte	MS %R	MS %R		LCS %R	
	(SW-846)	(CLP)		(Statistical)*	
	Water/Soil	Water	Soil	Water	Soil
1,2,4-Trichlorobenzene	44-142	39-98	38-107	53-95	29-103
Acenaphthene	47-145	46-118	31-137	65-101	45-98
2,4-Dinitrotoluene	39-139	24-96	28-89	63-96	46-92
Pyrene	52-115	26-127	35-142	56-111	49-100
N-Nitroso-di-n-propylamine	D-230	41-116	41-126	67-103	40-101
1,4-Dichlorobenzene	20-124	36-97	28-104	48-84	22-95
Pentachlorophenol	14-176	9-103	17-109	40-139	43-123
Phenol	5-112	12-110	26-90	51-105	34-102
2-Chlorophenol	23-134	27-123	25-102	54-106	36-100
4-Chloro-3-methylphenol	22-147	23-97	26-103	64-106	47-99
4-Nitrophenol	D-132	10-80	11-114	40-130	49-108

Surrogate	%Recovery		%Recovery	
	(SW-846)		(CLP)	
	Water	Soil	Water	Soil
2-Fluorophenol	21-100	25-121	21-110	25-121
Phenol-d ₅	10-94	24-113	10-110	24-113
Nitrobenzene-d ₅	35-114	23-120	35-114	23-120
2-Fluorobiphenyl	43-116	30-115	43-116	30-115
2,4,6-Tribromophenol	10-123	19-122	10-123	19-122
Terphenyl-d ₁₄	33-141	18-137	33-141	18-137

* Statistically derived acceptance limits will vary by individual laboratory operation.

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Table 5.7

**Representative Spike Recovery Acceptance Criteria for
Metals Analysis by SW-846 and CLP SOW**

<u>Parameter</u>	<u>Method</u>	<u>Analyte</u>	<u>MS %R</u>	<u>LCS %R (Statistical)*</u>	
				<u>Water</u>	<u>Soil</u>
Metals-ICPS	6010A/CLP	Aluminum	75-125	85-114	51-149
		Antimony	75-125	66-135	17-183
		Arsenic	75-125	89-116	48-152
		Barium	75-125	87-109	68-132
		Beryllium	75-125	75-111	63-137
		Boron	75-125	75-116	---
		Cadmium	75-125	82-123	59-141
		Calcium	75-125	88-113	67-133
		Chromium	75-125	88-116	60-140
		Cobalt	75-125	90-113	63-137
		Copper	75-125	90-112	61-139
		Iron	75-125	86-125	62-138
		Lead	75-125	88-116	55-145
		Magnesium	75-125	89-111	62-138
		Manganese	75-125	91-112	68-132
		Molybdenum	75-125	88-113	61-139
		Nickel	75-125	90-119	59-141
		Potassium	75-125	80-115	64-136
		Selenium	75-125	85-115	50-150
		Silver	75-125	79-120	43-157
		Sodium	75-125	86-112	52-148
		Strontium	75-125	80-120	---
		Thallium	75-125	91-120	48-152
Tin	75-125	80-120	---		
Titanium	75-125	80-120	---		
Vanadium	75-125	90-113	66-134		
Zinc	75-125	93-121	55-145		
Metals-GFAA	7041/CLP	Antimony	75-125	80-120	17-183
	7060A/CLP	Arsenic	75-125	74-126	48-152
	7421/CLP	Lead	75-125	74-127	55-145
	7740/CLP	Selenium	75-125	68-119	50-150
	7841/CLP	Thallium	75-125	79-130	48-152
	7470A/CLP	Mercury	75-125	75-118	52-148
Other Metals	7196A	Chromium(VI)	75-125	87-116	86-115
	CA DHS	Organic lead	75-125	60-145	71-128

* Statistically derived acceptance limits will vary by individual laboratory operation.

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Example**Table 5.8****Representative Spike Recovery Acceptance Criteria for
General Chemistry Analyses**

Analyte	MS %R	LCS %R (Statistical)*	
		Water	Soil
Cyanide	75-125	76-125	41-159
Total alkalinity (titration)	75-125	85-117	---
Total alkalinity (Automated)	75-125	78-110	---
Bromide (IC)	75-125	86-108	---
Chloride (IC)	75-125	81-112	---
Chloride (Automated)	75-125	79-127	---
Fluoride (ISE)	75-125	84-122	---
MBAS (colorimetric)	75-125	79-114	---
Nitrate (IC)	75-125	81-110	---
Nitrate (Automated)	75-125	83-119	---
Nitrite (IC)	75-125	79-116	---
Nitrite (Automated)	75-125	92-110	---
Nitrate/Nitrite (Automated)	75-125	85-117	---
Oil & grease	75-125	63-121	---
Total phenolics (Automated)	75-125	67-127	---
o-Phosphate (IC)	75-125	77-115	---
o-Phosphate (Automated)	75-125	75-125	---
Total phosphate (Automated)	75-125	85-114	---
Sulfate (IC)	75-125	86-113	---
TKN	75-125	75-125	---
TRPH	75-125	69-125	---

* Statistically derived acceptance limits will vary by individual laboratory operation.

6.0 SAMPLING PROCEDURES

6.1 INTRODUCTION

Obtaining representative samples and maintaining their integrity are critical parts of any monitoring program. Analytical methods have been standardized but the results of analyses are only as good as the sampling and the sample preservation methods. Defining the magnitude and the nature of an environmental problem requires collecting representative samples for laboratory analysis and data evaluation. The careful collection of samples is key to obtaining an accurate assessment of the site's environmental impact, and to developing the appropriate remedial solution. Defining in detail the numerous available sampling procedures and their associated quality elements applicable to environmental testing is beyond the scope of this document. Quality elements required to meet the DQOs for a given sampling event must be contained in a project specific sampling plan or within an overall site work plan. The plans should present the best approved techniques currently available for sampling and sample preservation.

In sampling, the objective is to remove a small portion of an environment that is representative of the entire body. Once the sample is taken, the constituents of the sample must stay in the same condition as when collected. The length of time that these constituents will remain stable is related to their character and the preservation method used. Since preservation methods relate to the parameters to be analyzed, these techniques are classified by parameter.

6.2 SAMPLING SERVICES

Various Pace locations provide a variety of sampling services. A well-defined communication mechanism is critical to obtaining samples which are representative of site conditions. Figure 6.1 lists minimum elements which must be established, communicated, and followed during each phase of the sampling and analysis project. Listed below are the types of sampling events for which Pace can provide services.

6.2.1 Ground Water Monitoring

Collection and analysis of ground water samples from sanitary landfills, Superfund sites, abandoned hazardous waste dumps and spill sites involves the use of an extensive array of state-of-the-art sampling equipment with the ability to pre-pump and sample wells of all sizes, to depths of more than 200 feet.

6.2.2 Waste Water Monitoring

Collection of samples for routine wastewater monitoring; special compliance monitoring for metals, cyanide and total toxic organics (TTO); NPDES permit application monitoring; and more, as required by local, state or federal agencies.

6.2.3 Hazardous Waste Sampling

Pace can conduct an inventory, collect and analyze samples, and repackage and properly label hazardous waste for shipment.

6.2.4 Flow Monitoring

Monitoring flow in most types of discharges through installation of wiers or flumes and also determine flow using fluormetric dye tracing methods.

6.2.5 Soil & Soil Gas Sampling

Technicians statistically develop soil sampling programs to identify pollution problems. Typically this involves collecting soil samples for volatile and semi-volatile organics, inorganics, hazardous waste constituents and most other chemical parameters of concern. Also collecting and analyzing soil gas samples.

6.2.6 PCB Services

Collection of samples from transformers, electrical switches and capacitors to be tested for the presence of PCBs. Handling of PCB-related spills includes the collection and analysis of wipe, swab or soil samples.

6.2.7 Ambient Air Monitoring and Stack Emission Testing

Pace has experience in providing sampling as specified in the Toxic Organic (TO method series) protocols for ambient air monitoring, along with NIOSH and AIHA specified applications. Full capability stack sampling and testing (e.g., VOST, impinger, etc.) is available for process optimization and emission monitoring.

6.3 FIELD SUPPORT

Pace provides shipping containers, custody documents, custody seals, sample bottles, labels, chemical preservatives for water samples, "blue ice" packs to maintain thermal preservation, and trip and field blanks to support field sampling events. Tables 6.1, 6.2 and 6.3 list sample container types, preservatives and holding times. Certain Pace locations can provide pick up and delivery services to their clients.

Upon receipt of the field samples at the laboratory, Pace ensures that sample bottles are maintained according to preservation requirements and that sample storage conditions do not contribute to the presence of test analytes in the samples.

Pace Shipping Containers

Pace typically uses commercial coolers for the transport of environmental samples from the field to the laboratory. Chain-of-custody seals and forms, employed for each cooler packed at Pace ensure complete documentation and provide evidence of unbroken custody of the cooler contents. Coolers meet or exceed all protocol requirements (i.e., DOT, USEPA, ASTM) for shipping. Coolers are prepared at the laboratory to provide the client with all of the sample containers needed for the analyses required by a project.

6.4 PRESERVATION

Pace provides the required chemical preservatives for water samples and "blue ice" packs, for thermal preservation when the samples are shipped back to the lab. High quality, reagent grade chemical preservatives are used. The ice packs are supplied pre-frozen or at ambient temperatures based upon the client's needs. It is the responsibility of those collecting the samples to properly use these materials and ensure that proper preservation techniques are performed and preservative requirements are met.

Upon receipt of samples at the laboratory, the temperature of each cooler is measured and recorded on the chain of custody documents. Similarly, the pH of bottles to which chemical preservative was added is measured (with the exception of sample collected for volatile organic compounds), and the check recorded. A disposable pipette is used to remove an aliquot of the sample for the pH check. When deviations from the required chemical or thermal preservation are noted, the project manager is notified, and clients may become involved in determining a course of action to follow.

Water samples for GC and GC/MS volatile aromatics determinations are monitored for pH just prior to analysis, at which time the pH of each individual sample bottle used is checked. The portion of sample used for the analytical determination is removed from the vial prior to checking the sample's pH. Sample pH measurements are recorded on laboratory chronicles as they are taken.

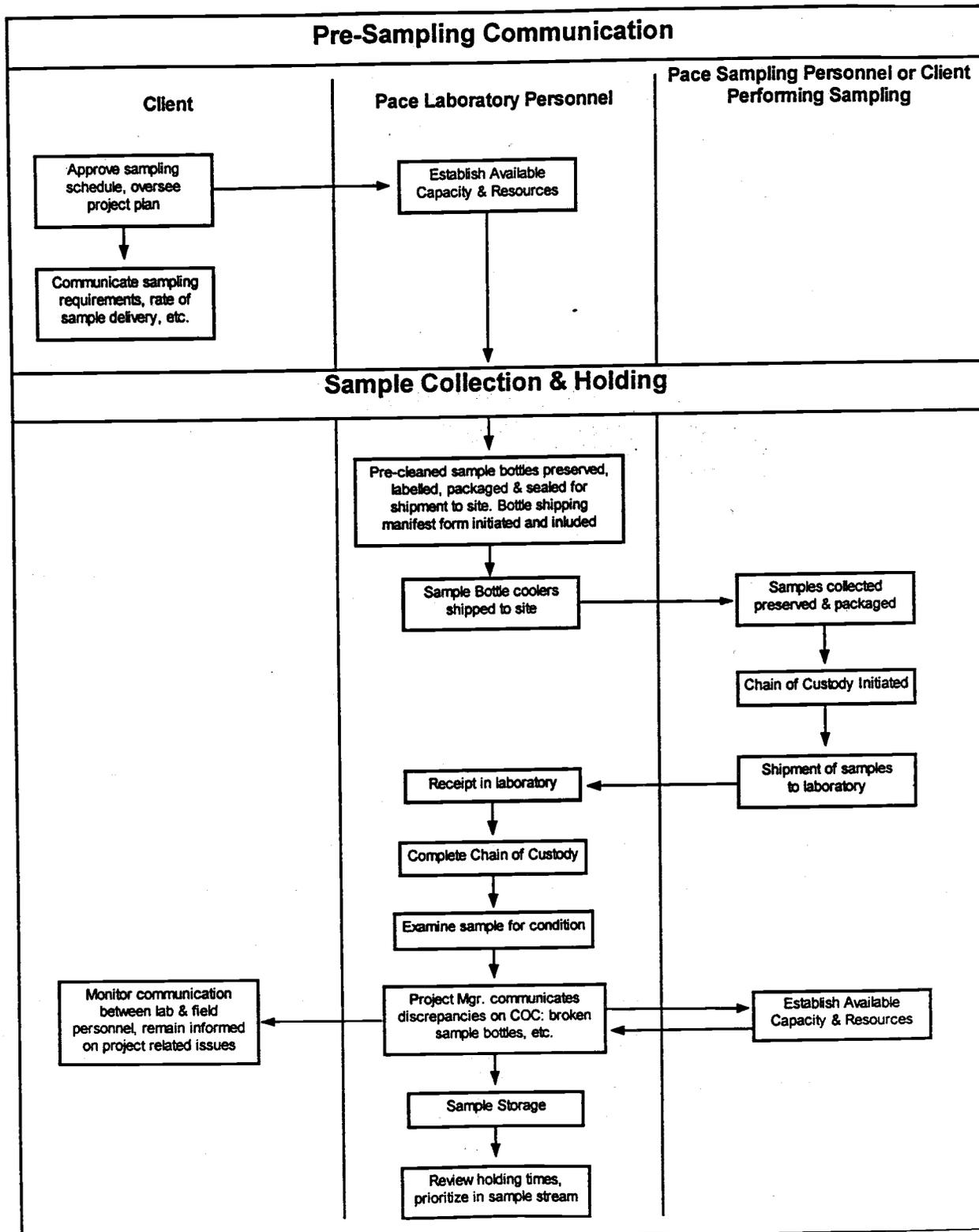
6.5 SAMPLE CONTAINERS

Pace provides precleaned sample containers in the shipping containers for sample collection. Used sample bottles are never used by the laboratory. Vendor prepared (certified contaminant-free) containers can be provided as projects necessitate.

6.6 SAMPLE RECEIPT SCHEDULE

Samples are normally delivered to the Pace facility during normal business hours within one day following field sampling unless different arrangements are made in advance with an authorized Pace representative. Shipping containers received at the laboratory on business holidays, weekends or after normal work hours will be placed in the walk-in refrigerator and opened on the next regular business day unless prior arrangements are made in advance for that day's receipt and log-in.

Figure 6.1



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TABLE 6.1 List of Containers, Preservatives and Holding Times for Inorganic and Organic Analyses of Aqueous Samples:

NAME	CONTAINER ¹	PRESERVATION ²	MAXIMUM HOLDING TIME ³
<i>Inorganic Tests:</i>			
Acidity	P,G	Cool, 4°C	14 days
Alkalinity	P,G	Cool, 4°C	14 days
Ammonia	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Biochemical oxygen demand	P,G	Cool, 4°C	48 hours
Bromide	P,G	None Required	28 days
Biochemical oxygen demand, carbonaceous	P,G	Cool, 4°C	28 days
Chemical oxygen demand	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Chloride	P,G	None Required	28 days
Chlorine, total residual	P,G	None Required	Analyze immediately
Color	P,G	Cool, 4°C	48 hours
Cyanide, total amenable to chlorination	P,G	Cool, 4°C, NaOH to pH>12 0.6g ascorbic acid ⁴	14 days
Fluoride	P	None Required	28 days
Hardness	P,G	HNO ₃ , to pH<2, H ₂ SO ₄ to pH<2	6 months
Hydrogen ion (pH)	P,G	None Required	Analyze immediately
Kjeldahl and organic nitrogen	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
<i>Metals:</i>			
Chromium VI	P,G	Cool, 4°C	24 hours
Mercury (SW846)	P,G	HNO ₃ to pH<2	38 days in glass 13 days in plastic
Mercury (CLP, 200 series)	P,G	HNO ₃ to pH<2	28 days
Metals, except chromiumVI and mercury	P,G	HNO ₃ to pH<2	6 months
Nitrate	P,G	Cool, 4°C	48 hours
Nitrate-nitrite	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Nitrite	P,G	Cool, 4°C	48 hours
Oil and grease	G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Organic carbon	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Orthophosphate	P,G	Filter immediately, Cool, 4°C	48 hours
Phenols	G only	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Phosphorus (elemental)	G	Cool, 4°C	48 hours
Phosphorus, total	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Residue, total	P,G	Cool, 4°C	7 days
Residue, filterable	P, G	Cool, 4°C	7 days
Residue, nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, volatile	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days

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TABLE 6.1 (cont.) List of Containers, Preservatives and Holding Times for Inorganic and Organic Analyses of Aqueous Samples:

NAME	CONTAINER ¹	PRESERVATION ²	MAXIMUM HOLDING TIME ³
Inorganics Continued:			
Specific conductance	P,G	Cool, 4°C	28 days
Sulfate	P,G	Cool, 4°C	28 days
Sulfide	P,G	Cool, 4°C, add zinc acetate & sodium hydroxide to pH>9	7 days
Sulfite	P,G	None Required	Analyze immediately
Surfactants	P,G	Cool, 4°C	48 hours
Turbidity	P,G	Cool, 4°C	48 hours
Organic Tests:			
Oil and Grease	G	Cool, 4°C, HCl or H ₂ SO ₄ to pH<2	28 days
Organic carbon, Total (TOC)	P,G	Cool, 4°C, HCl or H ₂ SO ₄ to pH<2	28 days
Purgeable Halocarbons	G,Teflon-lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁴	14 days
Purgeable Aromatic Hydrocarbons	G,Teflon-lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁴ , HCl ^{5,6}	14 days
Acrolein and acrylonitrile	G,Teflon-lined septum	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁴ , Adjust pH to 4-5	14 days
Phenols	G,Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁴	7 days until extraction, 40 days after extraction
Benzidines	G,Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁴	7 days until extraction, 40 days after extraction
Phthalate esters	G,Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitrosamines	G,Teflon-lined cap	Cool, 4°C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁴	7 days until extraction, 40 days after extraction
PCBs	G,Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitroaromatics and cyclic ketones	G,Teflon-lined cap	Cool, 4°C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁴	7 days until extraction, 40 days after extraction
Polynuclear aromatic hydrocarbons	G,Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁴	7 days until extraction, 40 days after extraction
Haloethers	G,Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁴	7 days until extraction, 40 days after extraction
Chlorinated Hydrocarbons	G,Teflon-lined cap	Cool, 4°C, HCl or H ₂ SO ₄	7 days until extraction, 40 days after extraction
Dioxins and Furans	G,Teflon-lined cap	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁴	7 days until extraction, 40 days after extraction
Total organic halides (TOX)	G,Teflon-lined cap	Cool, 4°C, HCl or H ₂ SO ₄ to pH <2	28 days
Pesticides	G,Teflon-lined cap	Cool, 4°C pH 5-9	7 days until extraction, 40 days after extraction

Table Footnotes:

- ¹ Polyethylene (P) or glass (G)
- ² Sample preservation should be performed immediately upon sample collection.
- ³ Holding times are based upon from time of sample collection.
- ⁴ Should only be used in the presence of residual chlorine.
- ⁵ Free chlorine must be removed prior to addition of HCl by the appropriate addition of $\text{NO}_2\text{S}_2\text{O}_3$
- ⁶ Sample receiving no pH adjustment must be analyzed within seven days of sampling.

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Table 6.2 Required Containers, Preservation Techniques, and Holding Times for Aqueous, Non-Aqueous, Soil or Solid Matrices (as specified in SW-846):

NAME	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME ¹
Semivolatile Organics/Organochlorine Pesticides/PCBs and Herbicides			
Concentrated waste samples	8 oz. wide mouth glass w/Teflon liner	None	14 days until extraction, 40 days after extraction
Liquid samples, no residual Chlorine present	1 gal. or 2 1/2 gal. amber glass w/Teflon liner	Cool, 4°C	Samples must be extracted within 7 days & extracts analyzed within 40 days
Residual Chloride, present	1 gal. or 2 1/2 gal. amber glass w/Teflon liner	Add 3mL 10% sodium thiosulfate	Samples must be extracted within 7 days & extracts analyzed within 40 days
Soil/sediments and sludges	8 oz. wide mouth glass w/Teflonliner	Cool, 4°C	14 days until extraction, extracts analyzed within 40 days.
Volatile Organics			
Concentrated waste samples	8 oz. wide mouth glass w/Teflonliner	None	14 days
Liquid samples, no residual Chlorine present	3x40 mL vials w/Teflon lined septum caps	Cool, 4°C ²	14 days
Residual Chlorine, present	3x40 mL vials w/Teflon lined septum caps	Collect sample in a 4 oz. soil VOA container which has been pre-preserved w/4 drops of 10% sodium thiosulfate. Gently mix sample & transfer to a 40mL VOA vial ² . Cool to 4°C	14 days
Acrolein & Acrylonitrile	3x40 mL vials w/Teflon lined septum caps	Adjust to pH 4-5, Cool to 4°C	14 days
Soil/sediments and sludges	4 oz. (120mL), wide mouth glass w/Teflon liner or wide mouth glass container sealed w/a septum	Cool to 4°C	14 days

¹ Holding times are based upon from time of sample collection.

² Adjust pH<2 w/H₂SO₄, HCl or solid NaHSO₄

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Table 6.3 Required Containers, Preservation, and Technical Hold Times for Air Methods:

TEST	MEDIA	PRESERVATION	MAXIMUM HOLDING TIME ¹
TO1	Tenax tubes	Freezer - 20°C	14 days
TO2	Carbo Sieve	Cool to 4°C	14 days
VOST	Tenaz/Tenaz- charcoal	Cool to 4°C	14 days
TO4	Puf 3" long, 60mm diameter	Freezer - 10°C or below	Extracted 7 days after collection
TO10	Puf 10cm long, 20mm diameter	Cool to 4°C	Extracted 7 days after collection
TO11	Absorbent cartridge	Cool to 4°C	30 days
TO13	Puf XAD/XAD		Extracted 7 days after collection

¹ Holding times are based upon from time of sample collection.

7.0 SAMPLE CUSTODY

7.1 SAMPLE RECEIPT

Sample shipments are received at the sample receiving area. Sample custodians verify the number of shipping containers received against the numbers listed on the shipping manifest/chain-of-custody. Any damage to the shipping containers or other discrepancy observed is noted on the chain-of-custody before signing it or on the sample receiving non-conformance report. A copy is filed for future reference.

When practical, the external chain-of-custody must be signed by the carrier for relinquishment of samples and signed by sample custodian personnel for sample receipt. The actual chain-of-custody may be supplied by Pace (Figure 7.1), or may be the client's own form. The chain-of-custody remains in the project file at all times.

7.2 CHAIN OF CUSTODY

Chain-of-Custody encompasses three major elements: field sampling, laboratory analysis and final data file. A Chain-of-Custody (COC) document may be the means in some types of legal proceedings by which evidence of custody of samples from time of receipt to completion of analysis is proven in the courts. Pace has implemented standard operating procedures to ensure that sample custody objectives of traceability and responsibility are achieved for every project. This section covers quality related activities from the receipt of samples at the laboratory through the issuance of final analytical data and the storage of data in its final data file.

The National Enforcement Investigations Center (NEIC) of EPA defines evidence of custody in the following manner:

1. It is in your actual possession, or
2. It is in your view, after being in your physical possession, or
3. It was in your possession and then you locked or sealed it up to prevent tampering,
or
4. It is in a secure area.

Samples may be physical evidence and should be handled according to certain procedural safeguards. Field personnel or Client representatives complete a Chain-of-Custody Form for all samples. Samples are received by the laboratory accompanied by these forms.

The sampler should provide the following information:

- Client project name
- Project location
- Field sample number/identification
- Date and time sampled
- Sample type

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- Preservative
- Analysis requested
- Sampler signature
- Signature of person relinquishing samples
- Date and time relinquished
- Sampler remarks
- Custody Seal Number (if applicable)

The record is filled out completely and legibly. Errors are corrected by drawing a single line through and initialing and dating the error. The correct information is then recorded with indelible ink. All transfers of samples except to and from commercial couriers must be recorded on the Chain-of-Custody via the "relinquished" and "received by" sections. All information except signatures may be printed.

7.3 SAMPLE VERIFICATION

7.3.1 Upon arrival of a sample shipment, sample control personnel perform sample inspection. Pace's Sample I.D. and Condition Sheet or equivalent (Figure 7.2) serves as a check-off list of procedures to follow and as documentation of the following:

1. Presence/absence of custody seals or tapes of the shipping containers and the condition of the seals (i.e., intact, broken).
2. Presence/absence of chain-of-custody; (if present, is it complete?)
3. Presence/absence of sample tags; (if present, are they removable?)
4. Agreement/non-agreement between the sample tags, chain-of-custody, and any client documentation.
5. Condition of the samples when received, including:
 - Sample temperature
 - Intact, broken/leaking
 - Headspace in VOA vials
 - Sample holding time
 - Sample pH when required

If discrepancies are found, the Pace project manager is contacted immediately (verbally and by using a Discrepancy Report Form or equivalent (Figure 7.3)). If the project manager is not available, the QAO is contacted for further directions. A copy of the Discrepancy Report Form is attached to the project data package.

7.4 SAMPLE LOG-IN

7.4.1 General Policies

- a. Upon completing sample receipt/custody procedures, all sample and analysis data must be complete and documented on the chain of custody or accompanying forms for input into the Laboratory Information Management System (LIMS).

Sample and analysis data must include:

1. Client name and contact
 2. Client number
 3. Pace project number
 4. Pace project manager
 5. Sample descriptions
 6. Due date
 7. List of analyses requested
- b. Sample and requested analyses data are input into the LIMS.
- c. All samples received are logged into the LIMS on the day of receipt.
- d. A Sample and Analysis Data Entry Form (SADEF) or equivalent (Figure 7.5) is generated immediately by the LIMS.

Distribution of SADEF:

To the Pace Project Manager with a photocopy of the chain-of-custody form. (Include a copy of the Discrepancy Report if applicable).

To the QC project file with the original chain of custody.

Photocopy to the Organic or Inorganic Department Manager as it applies for RUSH samples.

To the client.

- e. SADEF is to be reviewed against the chain of custody.
- f. Sample containers are labeled with the corresponding sample number and the stamped date of receipt.
- g. Samples are ready for storage.

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7.5 WHEN SAMPLES ARE RECEIVED WITH NO PAPERWORK

7.5.1. If delivered by a client: Client is asked if previous arrangements were made for analysis (and with whom). The client completes a chain of custody and/or request for analysis, relinquishes samples to sample custodian personnel, and is given a copy of the COC.

7.5.2. If received by courier or shipping, the following ordered steps are taken:

1. Routine Client File is checked
2. Anticipate Sample Alert File is checked
3. Sampling Kit Request File is checked
4. Pace key client contact is consulted
5. QC department manager is consulted to determine the designated Pace project manager
6. Information is requested from the Pace project manager

7.5.3. If analysis information cannot be determined on the day of sample receipt, sample data entry personnel proceed to assign sample numbers and put samples on hold. Follow-up with project manager occurs until the analyses are determined and samples can be properly logged in.

7.6 RESPONSIBILITIES FOR SAMPLE LOG IN

7.6.1. Sample Custodian

- Has the primary responsibility of ensuring that sample information is input into the LIMS as described in the SOP.
- Has the responsibility to make recommendations to the QC manager for revising the SOP.

7.6.2. Sample Management Officer

- Has the overall responsibility for ensuring that this procedure is implemented for all samples received into the laboratory.
- Has overall responsibility for ensuring that samples are logged in correctly (given that appropriate information has been supplied).

7.7 SAMPLE STORAGE

7.7.1. General Procedures

Samples are stored immediately upon receipt to prevent sample degradation.

7.7.2. Refrigerated Storage Area Maintenance

All refrigerated storage areas are maintained at 4°C (+/- 2°C). The temperature is monitored and recorded each work day (certain programs may require more frequent monitoring; e.g., twice daily). If the temperature fails outside the limits of 2°- 6°C, corrective action is to be taken as follows and appropriately documented.

1. Temperature is monitored at 60 minute intervals with the refrigerator door closed.
2. QAO is notified if the problem persists longer than one hour.
3. Samples are relocated to a proper storage environment if temperature cannot be maintained after corrective actions are implemented.

7.7.3. Routine Sample Storage

1. General Samples

Samples within each project are stored in sample number order. Waters and soils are generally stored on labeled separate shelves and in separate refrigerated units.

7.7.4. Specific Procedures

1. Volatiles

Samples within a project are stored in numerical order in vial containers. The holders are then stored where space permits in one of the designated volatile organic refrigerated storage areas.

2. Semi-Volatiles

Samples within a project are stored in numerical order in a designated, refrigerated storage area.

3. Hazardous Materials

Pure product or potentially heavily contaminated samples are tagged as "hazardous" and stored within a secured area, separate from other samples. This area is used only for hazardous samples and is labeled per Occupational Health and Safety Administration (OHSA) requirements.

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4. Special Projects

- Volatiles

Samples within a project are stored in sample number order in vial containers. The holders are then stored as space permits in the Special Project Volatiles (VOA) refrigerated storage area.

7.7.5. Responsibilities for Sample Storage

1. Sample Management Officer has direct responsibility for ensuring that the Standard Operating Procedure (SOP) is followed, samples are stored properly upon receipt, and refrigerated storage area temperatures are maintained.
2. Sample custodians are responsible for storing all samples upon receipt into the appropriate storage area, maintaining high level security for those samples under custody, and for keeping a current custody sample inventory.
3. Sample management personnel have the responsibility of daily sample storage area maintenance, disposal of old samples, and providing space for incoming samples in routine storage areas.
4. Assigned individuals are responsible for maintaining and documenting: (a) refrigerated storage area temperatures, and (b) corrective actions.

7.8 SAMPLE/DATA ACCESS AND INTERNAL CHAIN-OF-CUSTODY

7.8.1. General Policies and Procedures

Pace has implemented standard operating procedures to assure the integrity of samples and data so that they are not degraded or disclosed to unauthorized personnel. In order to ensure that this policy is maintained, the laboratory facilities are operated under controlled access. Only employees are allowed into the laboratory facilities; visitors must register at the front desk.

Samples are removed from their proper location by designated personnel and returned to the storage area immediately after the required sample quantity has been taken. This procedure minimizes unnecessary time spent searching for samples and helps prevent matrix degradation from prolonged exposure to room temperature. After the final report is sent and clients are allowed adequate time to review the results, the samples are properly discarded or returned to the client.

Upon client request, additional and more rigorous chain-of-custody protocols for samples and data can be implemented. For samples involving a high degree of confidentiality or potential litigation, Pace has developed extensive sample and data handling protocols to ensure the scientific and legal defensibility of the

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report submitted. These protocols include those specified by the USEPA Contract Laboratory Program.

Analysts and technicians follow strict internal chain-of-custody procedures to further ensure the validity of all data. All samples are signed out in a sample custody log book when they are removed for analysis. The sample ID, date, time, analyst, and lab of analysis is recorded in the sample custody log (Figure 7.4) or equivalent. Samples are signed back in noting date, time, and storage location, upon return.

7.8.2. Responsibilities for SOP Compliance

1. The QAO has the overall responsibility for ensuring that the SOP is implemented and followed.
2. Sample custodian personnel have the responsibility for ensuring that the SOP is properly followed, and to notify the QAO of problems.
3. All employees checking out samples are required to follow procedures.

7.9 SUBCONTRACTING ANALYTICAL SERVICES

Every effort is made to perform chemical analyses for Pace clients within a Pace laboratory. There are, however, instances where subcontracting of analytical services is necessary. Should subcontracting be necessary, samples are generally placed at other labs within the Pace integrated system of laboratories if at all possible. Currently, the following analyses are processed by specialty laboratories within Pace:

- Air Analyses
- Bioassay
- Explosives

When subcontracting becomes necessary, a preliminary verbal communication with an appropriate laboratory is undertaken. Work performed under specific protocols may involve special consideration, for instance, work involving NFESC samples may be subcontracted only to NFESC approved laboratories. The contact and preliminary arrangements and terms of agreement are made between the Pace Project Manager and the appropriate subcontract laboratory personnel (i.e., Laboratory Manager, customer services contact, or the appropriate laboratory section manager). The specific terms of the subcontract laboratory agreement should include (when applicable):

- Method (EPA or otherwise) of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required
- Applicable laboratory certification status
- Price per analysis
- Turn around time requirements

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Chain-of-Custody forms must be generated for samples which require subcontracting to other laboratories. The sample management personnel repackage the samples for shipment, create a transfer chain-of-custody form and record the following information:

- Pace Laboratory Number
- Matrix
- Requested analysis
- Special instructions (quick turn around, required detection or reporting limits, unusual information known about the samples or analytical procedure).
- Signature in "Relinquished By"

All subcontracted sample data reports are sent to the Pace Project Manager. The Project Manager sends the report to the appropriate Pace laboratory manager for review.

Any Pace work sent to other labs within the Pace network is handled as subcontracted work. All of the conditions and considerations noted in Section 7.10 and 7.11 apply.

7.10 SAMPLE DISPOSAL

After completion of sample analysis and submission of the analytical report, unused portions of samples are retained by the laboratory for a minimum of 2 weeks. After 2 weeks, samples will be disposed of according to the nature of the samples. The Hazardous Waste Manager receives a copy of the data report and uses that information to select the appropriate waste stream for the samples. The samples are considered hazardous waste, then they will be disposed of by state and federally licensed hazardous waste disposal firms.

Upon disposal of samples, a computer spreadsheet is maintained by the Hazardous Waste Manager listing the sample number, inherent waste stream and date disposed. This data file is updated on a weekly basis and is kept on file by the Hazardous Waste Manager and Sample Management.

7.11 EXCESS SAMPLE DISPOSITION

Samples not consumed during the analyses are returned to the client or disposed of by Pace. It is the project manager's responsibility to ensure that proper disposal has taken place. If the sample is determined to be non-hazardous by the project manager, it may then be disposed of by Pace via a non-manifested process.

7.1.1. Notification of Sample Return

The project manager and client receive written notification at the time of project initiation in the following manner:

1. The project proposal states the following paragraph in its Conditions and Terms Statement:

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"Pace Analytical Services, Inc.'s Standard Operating Procedure is to return all samples of hazardous materials to the client at project completion, and Pace Analytical Services, Inc. reserves the right to return or dispose of all samples at its discretion unless contractually agreed otherwise."

2. The Sample and Analysis Data Entry Form (or equivalent) states the following:

"Pace Analytical Services, Inc. reserves the right to return all samples at our discretion."

This form is printed by the LIMS at sample check-in.

3. The Sample and Analysis Data Entry Form cover letter contains the following paragraph:

1. "Pace Analytical Services, Inc.'s Standard Operating Procedure is to return all samples of hazardous materials or wastes to the client at project completion. Pace Analytical Services, Inc. reserves the right to return or dispose of all samples at our discretion" (Figure 7.5). This is a pre-printed cover letter that accompanies the Sample and Analysis Data Entry Form.

4. The Sample and Analysis Data Entry Form and cover letter (or equivalents) are generally sent to the client by the project manager depending upon project requirements.

7.11.2 Sample Return and Disposal

If samples are to be returned to the client or held longer than 60 days, a sample disposition form is generated. Otherwise, samples are disposed of a minimum of two weeks after project completion.

- a. The example Sample Disposition Form (Figure 7.6) contains the following information:

1. Client name, address, and contact
2. Pace project number
3. Client project identification number
4. Pace sample identification number
5. Pace project manager name

This form may vary by location

7.11.3 Procedure for Use of the Sample Disposition (SD) Form (or equivalent)

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1. The project manager separates the sample disposition form from the report package, signs the form, and routes it to the sample custodian. If the project requires, the hazardous waste manager may hold the form for a required amount of time before return or disposal.

It is important that this form be used and not discarded. It is part of the internal Chain of Custody and is filed with the project report

The hazardous waste manager or designee will use action codes such as:

1 = Return to client	2 = In house disposal
C = Clean	D = Dirty

As a general rule, soil samples will be returned and water samples will be disposed of in-house. Water samples which are highly contaminated will be returned. If a sample has an extremely high level of contamination, the contaminant will be noted by the project manager on the SD form.

For In-House Sample Disposal

All preserved -Non-hazardous-Neutralize/sink
 Hazardous-Toxic waste

Unpreserved water-Non-hazardous-Sink
 Hazardous-Toxic waste

Soil/Sludge-Non-hazardous-Refuse Disposal
 Hazardous-Toxic waste

All VOA's-Non-hazardous-Neutralize/sink
 Hazardous-Toxic waste

All Extracted Samples

CAM Extracts - Non-hazardous - Neutralize/sink
 Hazardous - Acid metals waste

Other Extracts - Hazardous waste

Liquid/Unknown Miscellaneous - Project manager specify

2. Subsequent to receipt of the Sample Disposition Form by the sample custodian, samples will be removed from storage using the information provided on the form.

If the Sample Disposition Form indicates "disposed," the Sample Custodian will remove samples from storage and place them at a sample disposal station for proper disposal. The process of disposal is performed by the

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sample custodian or appropriate laboratory staff. The Sample Disposition Form is signed and dated by the sample custodian then routed to the project manager for filing with other project information.

If samples are to be returned, the Sample Custodian removes them from storage, initials and dates the Sample Disposition Form. The samples, Sample Disposition Form, and a copy of the client's chain of custody are then delivered to the shipping clerk by the sample custodian for return to the client.

3. Upon receipt of the samples and Sample Disposition Form, the shipping clerk signs and dates the form.

The Sample Disposition Form is copied and the original form with the samples is returned to the client, along with a copy of the client's chain of custody. A copy of the Sample Disposition Form and the original chain of custody is routed to the file clerk for filing with other project information (QC file).

The shipping clerk labels the box with an appropriate hazard label and ships the samples back to the client.

4. Sample Disposition Forms are filed in project files.

7.11.4 Hazardous Material/Waste Sample Disposition Option

The preferred method for disposition of hazardous samples is to return the excess sample to the client. It may not be feasible to return samples in all cases or the client may require Pace to dispose of excess samples. Pace will dispose of excess samples when required and will charge a disposal fee to recover costs for management and disposal.

Procedure for Disposal Option for Excess Hazardous Material/Waste Samples:

1. When analyses are complete, the project manager indicates disposal as the option on the Sample Disposition Form and completes and attaches Hazardous Sample Disposal Option Form (Figure 7.7) or equivalent. An entry must be made in all fields of this form as it will determine the basis for lab packing and disposal.
2. The project manager routes the Disposal Option Form to sample check in.
3. The project manager is responsible for billing the client for disposal.
4. The sample custodian is responsible for maintaining a file of Disposal Option Forms for all samples awaiting disposal. Hazardous material/waste samples are stored in a safe manner and segregated by compatibility groups as indicated by the hazardous waste disposal SOP.

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5. The hazardous waste manager is responsible for reviewing accumulated samples awaiting disposal and initiating the disposal process when warranted. The Field Services, Inorganic, Organic, and Environmental Services Departments cooperate and participate in the disposal process. (For compatibility and compositing, see the Hazardous Waste Disposal SOP.)

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Example
Figure 7.1
Pace Analytical Services, Inc. CHAIN-OF-CUSTODY FORM

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FIGURE 7.2
SAMPLE I.D. AND CONDITION FORM
(Format may vary by location)

Client: _____
 Project No.: _____
 Date Received: _____

SAMPLE CONDITION UPON RECEIPT CHECKLIST

Complete checklist (A) during sample receipt. If any items are marked "NO," complete section (B) of this form. Otherwise, go to record samples.

		<u>YES</u>	<u>NO</u>
(A)	1. Are there custody seals or tapes on the shipping container?	___	___
	2. Are custody seals on the shipping container intact?	___	___
	3. Is there a completed Chain-Of-Custody (C-O-C)?	___	___
	4. Do the numbers of samples received and the sample matrices agree with C-O-C?	___	___
	5. Are there tags attached to each sample?	___	___
	6. Are sample tags, sample containers and C-O-C all in agreement?	___	___
	7. Is the C-O-C complete with requested analyses?	___	___
	8. Are the samples preserved correctly?	___	___
	9. Is there enough sample to do all analyses?	___	___
	10. Do the samples have the proper temperature?	___	___
	11. Are the sample containers intact (e.g., not broken, leaking)?	___	___
	12. Are VOA vials head-space free?	___	___
	13. Are all samples within the holding times for requested analyses?	___	___
	14. Is pH recorded for non-VOA's?	___	___

(B) Explain "NO" item here: _____

Send a copy of this form to Project Manager with Discrepancy Report Form. Copy of both forms remain in the QC file.

Custodian Signature: _____

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FIGURE 7.3

**Pace Analytical Services, Inc.
DISCREPANCY REPORT FORM
(Format may vary by location)**

Urgency Level: 1() Requires immediate attention
2() Requires attention today
3() Requires attention this week

Initiator _____ Client: _____

Date: _____

Project # _____

Sample(s) # _____

Discrepancy (if more space needed, use the back of this form): _____

To QC Manager: _____ Date: _____

Client Notified? YES () NO () Date & Time: _____

Project Manager Notified? YES () NO () Date & Time: _____

QC Response: _____

Project Manager Response: _____

Cause and Resolution (proposed or carried out): Completed by: _____

Manager's Initials:

PM Signature: _____ Date: _____

QC Signature: _____ Date: _____

cc: Project File

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FIGURE 7.4

**Pace Analytical Services, Inc.
 Internal Chain of Custody
 (Format may vary by location)**

Fractions Available

Project _____
 Date Created _____
 Initials _____

RELEASED _____
 SDG CLOSED _____

Potentially Radioactive? r Yes r No

SAMPLES				
A	F	K	P	U
B	G	L	Q	V
C	H	M	R	W
D	I	N	S	X
E	J	O	T	Y

SAMPLES	OUT Initials Date/Time	Which Fraction	Where is it Going?	IN Initials Date/Time	Any Totally Consumed?	Comments

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FIGURE 7.5

Pace Client Letter SAMPLE

May 24, 1995

Dear Valued Client:

A new policy has been implemented in the Sample Receiving Department of Pace Analytical Services, Inc. We hope that this policy will be helpful to you.

Upon acceptance of samples into the laboratory, the Sample Custodian completes a Sample and Analysis Data Entry Form. This form is designed to accommodate a short description of the samples received (sample name and/or sample reference), the type of container, and a list of the analyses requested to be performed on each sample. A copy of this form will be sent to the Client.

Enclosed is a copy of the Sample and Analysis Data Entry Form relevant to the samples we recently received from you. Please compare the information on the form to assure that it is consistent with your request. If there is any inconsistency or if you have any questions on your project, please call the Pace Contact indicated on the Sample and Analysis Data Entry Form. The Pace Contact has primary responsibility for monitoring the progress of your project through the laboratory.

It is also part of Pace Analytical Services, Inc.'s Standard Operating Procedure to return all samples pertaining to the information attached that are hazardous materials or hazardous wastes to the client at project completion. Pace Analytical Services, Inc. reserves the right to return or dispose of all samples at our discretion.

We have implemented this procedure to better serve our clients, and would appreciate any comments you may have.

Sincerely,

FIGURE 7.6

SAMPLE DISPOSITION FORM
(Format may vary by location)

Date removed: _____
Initials: _____

Date shipped: _____
Initials: _____

RE: Client Project ID: _____

 Pace Project No.: _____

Sample ID

_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

Dear _____ :

All requested analyses of the samples for the above referenced project have been completed. Enclosed are the remaining portions of the samples which are being returned to you for final disposition.

If you have any questions, please call me.

Sincerely,

Project Manager

FIGURE 7.7

**HAZARDOUS SAMPLE
DISPOSAL OPTION FORM**
(Format may vary by location)

Pace Project # _____

Project Manager _____

Pull Sample Date _____

<u>Sample #</u>	<u>Matrix</u>	<u>Location</u>	<u>Disposal Method</u>	<u>Charge</u>

Remarks: 1 = Return to Client C = Clean
 2 = In House Disposal D = Dirty

Removed from Refrigerator (initial/date) _____

Returned to Client (initial/date) _____

Disposed of Samples (initial/date) _____

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8.0 CALIBRATION PROCEDURES AND FREQUENCY

All instruments and equipment used in the laboratory must follow a well defined calibration routine. Calibration may be accomplished by laboratory personnel using certified standard reference materials traceable to the National Institute of Standards and Technology (NIST) or EPA certified materials or by external standardizing bodies or commercial standard manufacturers. The discussion presented here is general in nature because the requirements for calibration are instrument (or equipment) and method specific. Details of calibrations can be found in Pace Standard Operating Procedures, analytical methods, and instrument operations manuals. In addition to the summary calibration information pertaining to general analysis categories contained in the following subsections, Tables 8.1 and 8.2 list detailed calibration information for representative methods and applications most frequently performed at Pace.

8.1 STANDARDS AND TRACEABILITY

Analytical standards are prepared from pure compounds or are purchased as neat chemicals or diluted standard solutions from reputable vendors. They are used to prepare serial dilutions from which calibration and spiking standards are prepared. Each laboratory section is responsible for the preparation, storage and disposal of its standards. The preparation information is recorded into section specific Standards Notebooks in order to document traceability of prepared standards to their source material(s).

Each standard is given an internal identification number. The preparation of all stock standards shall be documented in a Standards Notebook which is used to record the date of preparation, analyst's initials, the source of the reference material, standard components, amounts used, final volume, final concentration(s), solvent used, expiration date of prepared standard, and the serial reference number of that stock solution. All standards shall be labeled with the standard serial reference number and expiration date (small glass ampules), and if space permits, with the name, concentration, date of preparation and initials of preparer. All diluted working standards not consumed during an analytical session shall be labeled fully, including the serial reference number of any stock standard used in its preparation.

If no expiration date has been assigned by the manufacturer, then an expiration date of one year from the date of preparation (or the date first opened in the case of sealed ampules) is reported unless degradation prior to this date is observed. To help determine if a standard has degraded, one must note inconsistencies. For instance, very poor recoveries from newly prepared quality control spikes or abnormally low instrument response to a specific standard are indications of possible standard degradation. However, for some standards, degradation is more easily noted. For instance, DDT breaks down to form DDD and DDE. Here one can visually note, on a chromatogram, the degradation of DDT by the increased concentrations of DDD and DDE. If degradation is observed before the default expiration date, it should be noted in the Standard Notebook for that standard and the standard removed from service.

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Before any set of standards can be utilized in a calibration curve they must be verified either externally by the standard supplier or internally by a secondary source process:

- Analysis of qualified QC Check Sample (e.g., A₂LA approved), or
- Analysis of an independently prepared check standard prepared either from a different manufactured lot for the same vendor supplying the calibration standard or from a second supplier.

8.2 GENERAL CALIBRATION PROCEDURES

Calibration standards for each parameter are chosen to bracket the expected concentrations of those parameters in the sample and to operate within the linear response range of the instrument. Samples that fall outside the calibration range are diluted until bracketed by the calibration standards. A low level standard is routinely analyzed to verify the reporting limit. Calibration standards are prepared typically at a minimum of three concentration levels, usually chosen at two to five times, five to ten times, and up to twenty times the estimated method detection limit plus a calibration blank, with the exception of most organic analyses which do not require a calibration blank. Either an internal standard or external standard quantification technique can be utilized. The reporting limit is verified by analysis of a standard at the reporting limit.

Calibration standards are prepared from materials of the highest available purity. To establish instrument calibration, working standards are prepared from more concentrated working stock solutions. All organic standards are refrigerated or frozen. Inorganic standards are refrigerated as necessary. Standard preparation information is recorded within each laboratory section in designated Standards Notebook.

Instrumental responses to calibration standards for each parameter are subjected to an appropriate statistical test of fitness (least squares linear regression, quadratic equation, or relative standard deviation of response factors) or as required by the method or QAPP. The calibration must reflect an acceptable correlation of data points or linearity to be acceptable. Point-to-point curve fitting shall not be used for establishing initial calibration correlation acceptance. In cases where the calibration data are outside these criteria, the analyst must rerun the calibration standards (meeting the same criteria) and/or prepare a new curve, changing instrumental conditions as necessary.

For analyses which are performed frequently and for which substantial calibration data is available, a complete recalibration is not required each time an analysis is performed providing that the following criterion is met: one calibration standard is analyzed at the beginning of the analysis which may vary from the expected response (based on the most recent initial calibration curve) by $\leq 25\%$ difference or as specified by the method, SOP or QAPP. If this criterion is not met, a complete recalibration is necessary.

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During the course of analysis, calibration standards are routinely analyzed to ensure that the instrumental response has not changed. The continuing calibration criteria stipulated in each method or SOP are used by the analyst to determine whether the instrument must be recalibrated or the instrument conditions further optimized.

The accuracy of prepared standards is periodically checked by comparison with a standard from an independent source.

Certain equipment such as balances, pH meters, and turbidity meters are normally calibrated with NIST traceable standard reference material.

8.2.1 Analytical Balances

Every 12 months, calibration of the entire analytical range shall be checked by a qualified service technician. The calibration of each balance is checked each day the balance is used with weights traceable to NIST. Calibration weights are ASTM Class 1 (replaces Class S designation) and are recertified every two years. If balances are calibrated by an external agency, verification of their weights shall be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the QA department.

8.2.2 Thermometers

Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are recertified every year with equipment directly traceable to the NIST.

Working thermometers are compared with the reference thermometers every 12 months; digital working thermometers are verified for accuracy on a quarterly frequency. Each thermometer is tagged and individually numbered. In addition, working thermometers are visually inspected by laboratory personnel prior to use.

Calibration temperatures and acceptance criteria are based upon the working range of the thermometer and the accuracy required for its use. Laboratory thermometer inventory and calibration data are maintained in the QA department or designated area.

8.2.3 pH/Electrometers

The meter is calibrated before use each day, and once after each four hours of continuous use, using fresh buffer solutions.

8.2.4 Spectrophotometers

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During use, spectrophotometer performance is checked at established frequencies in analysis sequences against initial calibration verification (ICV) and continuing calibration verification (CCV) standards. The instrument operating capability is also evaluated annually (more frequently if required by certification agencies) by qualified laboratory personnel or by an outside instrument maintenance service.

8.3 GC/MS CALIBRATION PROCEDURES

The minimum operations necessary to satisfy analytical requirements associated with the determination of organic compounds in water and soil/sediment samples are listed below. The following operations should be performed routinely in the laboratory:

- Documentation of GC/MS mass calibration and abundance pattern
- Documentation of GC/MS response factor stability
- Internal standard response and retention time

Prior to initiating data collection, it is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP) for base/neutral and acid (BNA) compounds or p-bromofluorobenzene (BFB) for volatile compounds. Each GC/MS system used for analysis of volatile or semivolatile organic compounds must be tuned to meet method or program specific ion abundance criteria before analysis of standards, blanks, or samples can proceed.

Prior to the analysis of samples and after tuning criteria have been met, the GC/MS system must be initially calibrated with a minimum of five concentrations of each compound being analyzed to determine the linearity of response. USEPA criteria specify both the concentration levels for initial calibration and the specific internal standard to be used on a compound-by-compound basis for quantitation. The response factor (RF) for each compound at each concentration level is calculated using the following Equation 8.1:

$$RF = \frac{A_x}{A_{is}} * \frac{C_{is}}{C_x} \quad (8.1)$$

Where:

A_x	= area of the characteristic ion for the compound to be measured
A_{is}	= area of the characteristic ion for the specific internal standards
C_{is}	= concentration of the internal standard (mg/ml)
C_x	= concentration of the compound to be measured (ng/ul)

Using the RF from the initial calibration, the percent relative standard deviation (%RSD) for compounds identified as Calibration Check Compounds (CCCs) is calculated using Equation 8.2:

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$$\%RSD = \frac{s}{\bar{x}} \times 100 \quad (8.2)$$

Where: RSD = relative standard deviation
 s = standard deviation of initial five response factors (per compound).
 \bar{x} = mean of initial five response factors (per compound).

The % RSD for each individual CCC must be less than 30% or as specified by the method. This criterion must be met for the initial calibration to be valid.

A calibration check standard containing all compounds of interest as well as all required surrogates, is performed each day of analysis. The RF data from the standard is compared each day against the average RF from the initial calibration for a specific instrument. If the response to a calibration check standard differs from the initial calibration by more than $\pm 20\%$ or as specified by the method, then investigation and corrective action must be performed, including a complete recalibration if necessary.

8.4 NON GC/MS CHROMATOGRAPHY CALIBRATION PROCEDURES

Initially, a three or five point calibration curve, consisting of all compounds of interest (plus a calibration blank for certain analyses such as VOCs), is established to define the usable range of the instrument. Calibration may be accomplished as best-fit line, quadratic equation, or average RF. The curve is determined to be linear if the correlation coefficient is ≥ 0.995 . Linearity may also be determined using response factors. Response factors are calculated for each compound at each concentration level. These RFs will be averaged to generate the mean RF for each compound over the range of the standard curve. The curve is determined to be linear if the RSD of the response factors is $< 20\%$. The mean response factor will be used to calculate the sample concentration of the compound of interest. When sample responses exceed the range of the standard curve, the sample will be diluted to fall within the range of the standard curve and be reanalyzed. The results of the daily GC standardization will be tabulated and filed with the corresponding sample analyses. Daily full calibration is not necessary if a calibration check standard validates the initial calibration curve. If the response to a calibration check standard differs from the initial calibration by more than $\pm 15\%$ for any analyte being quantitated or as specified by the method, then investigation and corrective action will be performed, including complete recalibration, if necessary.

Continuing Calibration is checked as described in Pace SOPs or methods.

8.5 Calibration of Inductively Coupled Argon Plasma Spectrometer (ICP) and Atomic Absorption Spectrophotometer (AAS)

The ICP and AAS are standardized for the metal of interest by the analysis of a set of calibration standards prepared by diluting a stock solution of known concentration. Working standards are prepared by dilution of the stock standard. For the AAS, the concentration of the calibration standards is chosen so as to cover the working range of

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the instrument. For ICP, a standard is analyzed as a sample to determine the upper limit of the calibration. Subsequently, all sample measurements are performed within this working range. After the working standards have been prepared, they are analyzed on the ICP or AAS and the instrument response is calibrated to provide a direct readout in concentration.

The calibration is accomplished by entering the metal concentration equivalent to the readout in absorbance units (or emission intensity) during analysis of the working standards.

After the initial calibration, the analysis of the working standards is repeated during sample analysis to standardize instrument response during analysis and to confirm the calibration settings. A typical analysis sequence is presented below.

- Working standards are prepared by dilution of a stock standard solution of the metal of interest.
- A calibration curve within the working range of the instrument is established by analysis of three to five working standards.
- An independent standard is analyzed to confirm the calibration settings. If the calibration settings are not confirmed, the instrument is recalibrated.
- The samples are analyzed for the metal of interest.
- During sample analysis, a check standard is analyzed to monitor instrument stability. If the analysis indicates that instrument calibration has changed by more than $\pm 10\%$ for ICP or more than $\pm 20\%$ for AAS, the instrument is recalibrated and the analysis is repeated.
- Following completion of the sample analyses, the check standard is reanalyzed to confirm calibration settings. If calibration settings are confirmed, the analysis is completed. However, if the calibration settings are not confirmed, the problem is corrected, and the analyses are repeated.

Written records of all calibrations shall be filed with the raw data.

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TABLE 8.1 Summary of Calibration Requirements

Instrument	Calibration Standards Used, Initial and Daily Minimum	Acceptance Limits	Corrective Actions
GC/MS (8270B)	Tune: DFTPP Initial: 5 level (20,50,80,120,160, ppb) Daily: 1 level (50) (every 12 hours)	Meets criteria RSD <30% for RFs of CCCs RF \geq 0.050 (SPCC)	Re-tune instrument Repeat DFTPP analysis Repeat Calibration Evaluate system Repeat Calibration Evaluate system Take corrective action Repeat Calib. Check; see Lab Supervisor
GC/MS (8240B)	Tune: BFB Initial: 5 level (10,20,50,100,200 ppb) Daily: 1 level (50) (every 12 hours)	Meets Method Criteria RSD <30% for RFs of CCCs RF \geq 0.300 (0.250 for bromoform) (SPCC) % Difference <20% of the average five-point RF (CCC)	Re-tune instrument Repeat BFB Analysis Repeat Calibration Evaluate System Repeat Calibration Evaluate System Take Corrective Action Repeat Calib. Check; See Lab Supervisor
Gas Chromatograph (8080A)	Initial: 5 level (conc. based upon instr. response) Mid level DDT/Endrin standard	Std curve or calibration factor (CF) if % RSD \leq 20 DDT/Endrin breakdown < 20%	Make new standards or establish new calibration curve. Rerun standard once Perform col. maint.

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TABLE 8.1 Summary of Calibration Requirements (continued)

Instrument	Calibration Standards Used, Initial and Daily Minimum	Acceptance Limits	Corrective Actions
Gas Chromatograph (herbicides)	Daily: 1 level of check standard (midrange) Std check every 10 samples	CF \pm 15% of initial calibration CF \pm 15% of daily calibration (< \pm 20% for confirmation). Retention times within retention time windows. (For methods using retention time windows.)	Repeat initial calibration Reanalyze samples that were analyzed after standard that failed criteria and before next standard that passes criteria
Gas Chromatograph (herbicides)	Initial: 5 levels (conc. based upon instr. response) Daily: 1 level of check standard (midrange) Std check every 10 samples	Standard curve or calibration factor (CF) if % RSD < 20 CF \pm 15% of initial calibration CF \pm 15% of daily calibration (< \pm 20% for confirmation). Retention times within retention time windows. (For methods using retention time windows.)	Make new standards or establish new calibration curve Rerun samples that were analyzed between standards failing criteria
Inductively Coupled Plasma Emission Spectrometer	Initial: high standard + blank Daily: instrument check standard and calibration std. & blank every 10 samples	ICV: < 90-110% CCV: < 80-120%	Recalibrate. Repeat twice; if outside control limit, then recalibrate making new stds if necessary

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TABLE 8.1 Summary of Calibration Requirements (continued)

Instrument	Calibration Standards Used, Initial and Daily Minimum	Acceptance Limits	Corrective Actions
Atomic Absorption Spectrophotometer	Initial: 5 levels + blank Daily: 1 check standard (midrange) & blank per 10 samples	Linear regression correlation coefficient ≥ 0.995 ; ICV: 90-110% CCV: 80-120%	Make new standards or establish new calibration curve
pH Meter	Daily: 2 levels	+0.05 pH unit Bracket sample range	Clean or replace electrode; recalibrate
UV-Visible Spectrophotometer	Initial: 5 levels + blank Daily: check standard	Linear regression correlation coefficient > 0.995 ; ICV: 90-110%	Recalibrate services

TABLE 8.2 Summary of Routine Calibration Requirements

Instrument	Calibration Standards Used, Initial and Daily Minimum	Acceptance Limits	Corrective Actions
Analytical Balance	Daily: Sensitivity (with ASTM Class "1" weight)	± 0.001 gm (varies by method)	Adjust sensitivity, re-level
Thermometers	Annually: Calibrate in constant temperature baths at two temperatures against precision thermometers certified against an NIST thermometer	± 0.1 to ± 0.5 C (depending upon method)	Tag and remove from service, replace

9.0 ANALYTICAL PROCEDURES

Pace laboratories are capable of analyzing the full range of environmental samples from all media, including surface and groundwater, soil, sediment, tissue, and waste. Refer to Table 9.2 for a representative listing of specific Pace analytical capabilities. Methodologies are employed with guidance from agencies such as EPA, ASTM, USGS, NIOSH and, in certain instances, state regulatory agencies. In some situations, Pace develops and validates methodologies which are more applicable to a specific problem or objective.

Analytical procedures are detailed descriptions of any and all processing, preparation and analysis of samples in the laboratory. In some instances, data format, presentation and delivery are also described. All analytical procedures shall be conducted in strict adherence with written Standard Operating Procedures manuals which have been reviewed and approved by the Laboratory Operations Manager, the Pace QA Officer and the Pace General Manager. Documents from which SOPs are developed include the references listed in Table 9.1. Additional SOPs may be adapted from other sources or generated in-house as project needs require.

9.1 ANALYTICAL METHODS

Numerous sources of information are available to offer guidance in analytical methods. Selection of the appropriate method is dependent upon data usage and the regulatory requirements during the analysis. Table 9.1 describes the analytical references routinely used by Pace Laboratories. Pace may modify existing methods based on the following considerations: 1) in order to meet project specific objectives; 2) in order to incorporate modifications or improvements in analytical technology; 3) in order to comply with changing regulations and requirements; 4) in order to address unusual matrices not covered in available methods.

Pace will make every effort to disclose to its clients any instances in which modified methods are being used in the analysis of samples.

The following subsections contain method synopses for representative methods most frequently performed at Pace laboratories. For clarity purposes, certain method summaries also contain calibration criteria, several of which have been previously detailed in Section 8.

9.2 SAMPLE PREPARATION METHODS

9.2.1 Digestion of Aqueous Samples for Metals - Method 3005A

This method is an acid digestion procedure used for the preparation of water samples for metals analysis. The digested samples can be analyzed for dissolved and total recoverable metals by flame (FAA) or furnace (GFAA) atomic absorption spectrophotometry or by inductively coupled argon plasma emission spectroscopy (ICPS). Method 3005A may be used to prepare samples for analysis of the following metals:

Aluminum	Cobalt	Potassium
Antimony	Copper	Selenium
Arsenic	Iron	Silver
Barium	Lead	Sodium
Beryllium	Magnesium	Thallium
Cadmium	Manganese	Vanadium
Calcium	Molybdenum	Zinc
Chromium	Nickel	

For the analysis of total recoverable metals, the entire sample is acidified at the time of collection with nitric acid (HNO₃). Sample preparation involves heating the sample with acid and concentrating to a specified volume. The sample is not allowed to boil because some of the elements are in a volatile state and may be easily lost. The digestate is then filtered (if necessary) and diluted to the desired concentration for analysis.

For the analysis of dissolved metals, the samples are filtered through a 0.45-um filter immediately upon collection and prior to acidification with nitric acid. In the lab, the sample is heated with acid and the volume is reduced. The digestate is filtered again (if necessary) and diluted to volume.

9.2.2 Digestion of Aqueous Samples for Metals - Method 3010A and the CLP SOW

These methods describe the preparation of aqueous samples for total metals determination by flame atomic absorption spectrophotometry (FAA) and by inductively coupled argon plasma emission spectroscopy (ICPS). By method 3010A, samples are vigorously digested with nitric acid. By CLP protocol, samples are digested with a mixture of nitric acid and hydrochloric acid.

9.2.3 Digestion of Aqueous Samples for Metals - Method 3020A and the CLP SOW

These methods describe the preparation of aqueous samples for total metals determination by graphite furnace atomic absorption spectroscopy (GFAA). By method 3020A, samples are vigorously digested with nitric acid. By CLP protocol, samples are digested with a mixture of nitric acid and hydrogen peroxide.

9.2.4 Digestion of Solid Samples for Metals - Method 3050A and the CLP SOW

These methods are applicable to the preparation of sediment, sludge, and soil samples for metals determination by FAA or GFAA or by ICPS. One gram of solid sample is digested with nitric acid and hydrogen peroxide. The digestate is then refluxed with nitric or hydrochloric acid, depending on the analysis performed. When using hydrochloric acid as the final refluxing acid, the digestates may not be boiled because antimony is in a volatile state and may be easily lost. A separate sample aliquot is dried to determine the percent moisture in the sample.

9.2.5 Separatory Extraction - Method 3510B

Method 3510B is designed to quantitatively extract nonvolatile and semivolatile organic compounds from liquid samples using separatory funnel techniques. The sample and extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup and detection methods are described in the organic analytical method that will be used to analyze the extract. Samples are pH-adjusted and serially extracted by vigorous shaking for 1-2 minutes with the appropriate solvent for the analytical method. Samples are extracted three times, the combined extracts are dried with anhydrous sodium sulfate and concentrated in a Kuderna-Danish apparatus.

9.2.6 Continuous Liquid/Liquid Extraction - Method 3520B

Method 3520B is designed to quantitatively extract nonvolatile and semivolatile organic compounds from liquid samples using continuous liquid-liquid extractors. The sample and extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup and detection methods are described in the organic analytical method that will be used to analyze the extract. Samples are pH-adjusted and extracted with the appropriate solvent for the analytical method. Samples are extracted for 18 to 24 hours, the extracts are dried with anhydrous sodium sulfate, and then concentrated in a Kuderna-Danish apparatus.

9.2.7 Soxhlet Extraction - Method 3540B

Method 3540B is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent. Extraction is accomplished by mixing the solid sample with anhydrous sodium sulfate, placing it in an extraction thimble or between two plugs of glass wool, and extracting it with an appropriate solvent in the Soxhlet extractor for 18 to 24 hours. The extract is dried and concentrated and then treated using a cleanup method or analyzed directly by the appropriate measurement technique.

9.2.8 Sonication Extraction - Method 3550A

Method 3550A is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. The Sonication process ensures intimate contact of the sample matrix with the extraction solvent. A weighed sample of the solid waste is mixed with sodium sulfate, then dispersed into the solvent using sonication. The extract is dried with anhydrous sodium sulfate and concentrated with a Kuderna-Danish apparatus. The resulting solution may then be cleaned up or analyzed directly using the appropriate technique.

9.2.9 Waste Dilution - Method 3580A

Method 3580A is a technique for solvent dilution of non-aqueous waste samples prior to sample cleanup and/or analysis. It is designed for wastes that may contain organic constituents at concentrations greater than 20,000 ug/kg and that are soluble in the dilution solvent.

9.2.10 Purge-and-Trap Sample Introduction - Method 5030A

Method 5030A is used to determine the concentration of volatile organic compounds in a variety of liquid and solid waste matrices using a purge and trap gas chromatographic procedure. The success of this method depends on the level of interferences in the sample. Results may vary due to the large variability and complexity of various matrices.

Inert gas is bubbled through a 5-mL or 25-mL aqueous sample aliquot at ambient temperature to transfer the volatile components to the vapor phase. The vapor is swept to a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is flash heated and backflushed with inert gas to desorb and transfer the volatile components onto the head of a GC column. The column is heated to elute the volatile components, which are detected by the appropriate detector for the analytical method used.

Solid samples may be analyzed using one of two techniques. For Low level soil analysis, 5 g of solid sample is dispersed into 5 mL of Contaminant-free laboratory water and the sample is purged as described above. This technique is referred to as the direct purge method. For medium level soil analysis, an aliquot of solid sample is dispersed in methanol to dissolve the volatile constituents and a portion of the methanol extract is combined with contaminant-free laboratory water and purged as described above.

9.2.11 Extraction Procedure Toxicity Test (EP-Tox) - Method 1310A

This method is used to determine whether a waste exhibits the characteristics of extraction procedure (EP) toxicity. If a representative sample of the waste contains >0.5% solids, the solid phase of the sample is ground to pass a 9.5 mm sieve and extracted with deionized (DI) water that is pH adjusted with acetic acid. Wastes containing <0.5% solid material are extracted and analyzed as a single phase.

9.2.12 Toxicity Characteristic Leaching Procedure (TCLP) - Method 1311

This method is used to determine whether a waste exhibits toxicity leaching characteristics. The procedure includes a leaching extraction for semivolatile compounds and metals and a zero-headspace extraction for volatile compounds.

9.2.13 California Assessment Manual Waste Extraction Test (CAM WET)

This waste extraction test, described in the California Administrative Code, Title 22, Article 11, Section 66700, can be used to determine the amount of extractable substance in a waste or other material.

9.3 CALIBRATION AND ANALYSIS PROCEDURES FOR ORGANICS

9.3.1 Halogenated Volatile Organics - Method 8010B

Halogenated volatile organics in water and soil samples are analyzed using method 8010B, which is a gas chromatography (GC) method using purge and trap sample introduction (method 5030A). An inert gas is bubbled through a water matrix to transfer the volatile halocarbons from the liquid to the vapor phase. Volatile halocarbons are collected on a sorbent trap, then flash thermally desorbed and transferred to a GC column. Target analytes are detected with an electrolytic conductivity detector (ELCD). Soil samples may be heat purged directly in reagent water or are extracted with methanol; if extracted in methanol an aliquot of sample extract is added to blank reagent water for purge and trap GC analysis.

Positive results are confirmed by GC analysis using a second GC column of dissimilar phase. When second column analysis is performed, peak retention times (RTs) on both columns must match expected RTs within the calculated RT windows. Also, calculated quantitations from each column should be in agreement with one another (generally they should match within a factor of two) for the presence of an analyte to be considered confirmed.

Calibration - Calibration standards are prepared and analyzed at five concentration levels. A linear calibration curve not forced through the origin is developed for each analyte of interest. This function is used for the calibration curve if the correlation coefficient (r) for that analyte is ≥ 0.995 , otherwise, a curve function is used that meets this criterion. Each working day, the calibration is verified with the analysis of a continuing calibration standard at the beginning and end of the run sequence and after every 10 analyses. The calibration factor for each analyte to be quantitated must not exceed a 15% difference when compared to the initial standard of the analysis sequence. When this criterion is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is necessary before recalibrating and proceeding with sample analysis. All samples that were injected after the standard exceeding the criterion must be reinjected to avoid errors in quantitation, if the initial analysis indicated the presence of the specific target analytes that exceeded the criterion.

9.3.2 Aromatic Volatile Organics - Method 8020A

Aromatic volatile organics in water and soil samples are analyzed using method 8020A, which is a gas chromatography (GC) method using purge and trap sample introduction (method 5030A). An inert gas is bubbled through a water matrix to transfer volatile aromatic hydrocarbons from the liquid to the vapor phase. Volatile aromatics are collected on a sorbent trap, then flash thermally desorbed and transferred to a GC column. Target analytes are detected using a photoionization detector (PID). Soil samples may be heat purged directly in reagent water or are extracted with methanol; if extracted with methanol an aliquot of sample extract is added to blank reagent water for purge and trap GC analysis.

Positive results are confirmed by GC analysis using a second GC column of dissimilar phase. When second column analysis is performed, peak RTs on both columns must match expected RTs within the calculated RT windows. Also, calculated quantitations from each column should be in agreement with one another (generally they should match within a factor of two) for the presence of an analyte to be considered confirmed.

Calibration - Calibration standards are prepared and analyzed at five concentration levels. A linear calibration curve not forced through the origin is developed for each analyte of interest. This function is used for the calibration curve if $r \geq 0.995$ for that analyte; otherwise, a curve function is used that meets this criterion. Each working day, the calibration is verified with the analysis of a continuing calibration standard at the beginning and end of the run sequence and after every 10 analyses. For each analyte of interest, the %D of the response in the continuing calibration standard must agree with the expected response by $\leq 15\%$ in order for the run sequence to continue.

9.3.3 Organochlorine Pesticides and PCBs - Method 8080A and the CLP SOW

Organochlorine pesticides and PCBs are analyzed by gas chromatography following either method 8080A or the CLP Organic SOW. Each of these analyses involves solvent extraction of the sample followed by analysis by gas chromatography with electron capture detection (GC-ECD).

Positive results are confirmed using a second GC column of dissimilar phase. For an analyte to be considered confirmed, the peak RTs on both columns must match the expected RTs. Also, the calculated quantitations between the two columns should be in agreement with one another (generally they should match within a factor of two) for the presence of the analyte to be considered confirmed by method 8080A. For analysis by CLP protocol, the results are flagged with a "P" if the two quantitations differ by more than 25%. In addition, the breakdown of 4,4'-DDT and endrin is monitored. If the breakdown of either of these compounds is found to exceed 20%, the analytical sequence must be discontinued. For analysis by CLP protocol, the combined breakdown must also not exceed 30%.

Calibration for Method 8080A - Calibration standards are prepared and analyzed at five concentration levels. A linear calibration curve not forced through the origin is developed for each analyte of interest. This function is used for the calibration curve if $r \geq 0.995$ for that analyte; otherwise, a curve function is used that meets this criterion. Each working day, the calibration is verified with the analysis of a continuing calibration standard at the beginning and end of the run sequence and after every 10 analyses. For each analyte of interest, the %D of the response in the continuing calibration standard must agree with the expected response by $\leq 15\%$ in order for the run sequence to continue.

Calibration for the CLP SOW - Calibration and analysis are performed in strict accordance with the CLP Organic SOW.

9.3.4 Volatile Organics - Method 8240B and the CLP SOW

Samples may be analyzed for volatile organics by gas chromatography/mass spectrometry (GC/MS) following the procedure described in method 8240B or the CLP Organic SOW. Analyte identification and quantitation are accomplished using response factors and retention times generated from a five-point calibration curve, relative to the closest eluting internal standard. The three internal standards used for these methods are:

- Bromochloromethane
- 1,4-Difluorobenzene
- Chlorobenzene-d₅

If requested by the client, non-target analytes are reported as tentatively identified compounds (TICs), when an acceptable match is obtained between the spectrum of the analyte and a spectrum found by library search. Unidentified TICs are labeled "unknown". The TICs are quantitated using response factors of 1 relative to the nearest eluting internal standards.

Instrument Performance Check - The mass spectrometer is tuned daily and after every 12 hours of operation to yield an acceptable spectrum for p-bromofluorobenzene (BFB). Relative ion abundance criteria for BFB are given in Table 9.3.

Calibration for Method 8240B - After passing the instrument performance check criteria and prior to analyzing samples, a 5-point initial calibration is performed. From that calibration, the calibration check compounds (CCCs) must meet the RSD criteria of $\leq 30\%$ and the system performance check compounds (SPCCs) must meet the minimum RRF criteria given in the method. A continuing calibration standard is analyzed after every 12 hours of operation. In that standard, the CCC compounds must meet the %D criteria of $\leq 20\%$ and SPCC compounds must meet the minimum RRF criteria listed in the method.

Calibration for the CLP SOW - After passing the instrument performance check criteria and prior to analyzing samples, a 5-point initial calibration is performed. From that calibration, the compounds listed in Table 2 of Exhibit D, Section IV (VOA) of the CLP SOW must meet the RSD criteria of $\leq 20.5\%$ and the minimum RRF criteria listed in the method. A continuing calibration standard is analyzed after every 12 hours of operation. In that standard, the Table 2 compounds must meet the %D criteria of $\leq 25\%$ and the minimum RRF criteria listed in the method.

9.3.5 Semivolatile Organics - Method 8270B and the CLP SOW

Semivolatile extracts are analyzed by gas chromatography/mass spectrometry following method 8270B or the CLP Organic SOW. All samples are prepared following extraction methods described in the applicable protocol. Identification and quantitation is performed using response factors and retention times generated from a five-point calibration curve, relative to the closest eluting of six internal standards. The six internal standards are:

- 1,4-Dichlorobenzene-d₄

- Naphthalene-d₈
- Acenaphthene-d₁₀
- Phenanthrene-d₁₀
- Chrysene-d₁₂
- Perylene-d₁₂

If requested by the client, non-target analytes are reported as tentatively identified compounds (TICs), when an acceptable match is obtained between the spectrum of the analyte and a spectrum found by library search. Unidentified TICs are labeled "unknown". The TICs are quantitated using response factors of 1 relative to the nearest eluting internal standards.

Instrument Performance Check - The mass spectrometer is tuned daily and after every 12 hours of operation to give an acceptable spectrum for DFTPP. DFTPP ion abundance criteria are given in Table 9-4.

Calibration for Method 8270B - After passing the instrument performance check criteria and prior to analyzing samples, a 5-point initial calibration is performed. From that calibration, the CCC compounds must meet the RSD criteria of $\leq 30\%$ and the SPCC compounds must meet the minimum RRF criteria given in the method. A continuing calibration standard is analyzed after every 12 hours of operation. In that standard, the CCC compounds must meet the %D criteria of $\leq 20\%$ and SPCC compounds must meet the minimum RRF criteria listed in the method.

Calibration for the CLP SOW - After passing the instrument performance check criteria and prior to analyzing samples, a 5-point initial calibration is performed. From that calibration, the compounds listed in Table 5 of Exhibit D, Section IV (SV) of the CLP SOW must meet the RSD criteria of $\leq 20.5\%$ and the minimum RRF criteria listed in the method. A continuing calibration standard is analyzed after every 12 hours of operation. In that standard, the Table 5 compounds must meet the %D criteria of $\leq 25\%$ and the minimum RRF criteria listed in the method.

9.3.6 Purgeable Petroleum Hydrocarbons

Gasoline and volatile aromatic compounds, including benzene, toluene, ethylbenzene, and the xylenes (BTEX), are analyzed by a modified method 8015A using the direct purge technique described above for method 5030A. Analysis is performed on a GC equipped with a photoionization detector (PID) and a flame ionization detector (FID) connected in series. If BTEX compounds are found without the associated presence of gasoline, confirmation analysis is performed with a second GC column of dissimilar phase and retention characteristics in accordance with the requirements of method 8020A.

Calibration - Calibration standards are prepared and analyzed at five concentration levels. A linear calibration curve not forced through the origin is developed for each analyte of interest. This function is used for the calibration curve if $r \geq 0.995$ for that analyte; otherwise, a curve function is used that meets this criterion. Each working day, the calibration is verified with the analysis of a continuing calibration

standard at the beginning and end of the run sequence and after every 10 analyses. For each analyte of interest, the %D of the response in the continuing calibration standard must agree with the expected response by $\leq 15\%$ in order for the run sequence to continue.

9.3.7 Extractable Petroleum Hydrocarbons

Aqueous samples analyzed for diesel, kerosene, jet fuel, and motor oil are prepared using method 3510B (separatory funnel liquid/liquid extraction) or method 3520B (continuous liquid/liquid extraction). Solid samples are prepared using method 3540B (Soxhlet extraction), method 3550 (sonication extraction), or wrist action shaker extraction (California LUFT method). One liter of water or 30 g of soil/sludge are extracted and concentrated to a volume of 1 mL. Analysis is performed by a modified method 8015A on a GC equipped with a capillary or megabore column and an FID detector.

Calibration - Calibration standards are prepared and analyzed at five concentration levels. A linear calibration curve not forced through the origin is developed for each analyte of interest. This function is used for the calibration curve if $r \geq 0.995$ for that analyte; otherwise, a curve function is used that meets this criterion. Each working day, the calibration is verified with the analysis of a continuing calibration standard at the beginning and end of the run sequence and after every 10 analyses. For each analyte of interest, the %D of the response in the continuing calibration standard must agree with the expected response by $\leq 15\%$ in order for the run sequence to continue.

9.4 REPRESENTATIVE CALIBRATION AND ANALYSIS PROCEDURES FOR INORGANICS

9.4.1 Metals by ICPS - Method 6010A and the CLP SOW

These methods describe the simultaneous or sequential determination of metal elements using ICPS. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is passed through a plasma torch. Element-specific atomic-line emission spectra are produced which are dispersed by a grating spectrometer and monitored for intensity by photomultiplier tubes.

Calibration - The calibration procedures for ICPS are detailed in method 6010A and the CLP Inorganics SOW. Prior to the analysis of samples, an initial multipoint calibration is analyzed for all elements of interest. The initial calibration is checked with an initial calibration verification standard (ICV). For each element, the ICV responses must agree with the initial calibration within $\pm 10\%$ for the calibration to be verified. Following the ICV and after the analysis of every 10 samples, a continuing calibration verification standard (CCV) is analyzed. The response for each element in the CCV must agree within $\pm 20\%$ of the expected value for the analysis to continue.

9.4.2 Metals by GFAA - Methods 7060A, 7421, 7740, 7841, and the CLP SOW - Graphite furnace atomic absorption (GFAA) techniques may be used for the

determination of arsenic, lead, selenium, thallium, and other metals depending upon the sensitivity required. Following sample digestion, an aliquot of sample is placed in a graphite tube in the furnace, evaporated to dryness, charred, and atomized. The sample is placed in the light path of an atomic absorption spectrophotometer. The absorption of light by the atomized metal is measured with a photomultiplier tube.

Calibration - Calibration procedures for the GFAA analyses are detailed in the respective methods in SW-846 and the CLP SOW. For the element of interest, a multipoint initial calibration is performed. The calibration correlation coefficient must be ≥ 0.995 to be acceptable. The initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree with the expected response within $\pm 10\%$ in order for the calibration to be verified. A CCV check standard is analyzed following the analysis of the ICV and after the analysis of every 10 samples. The response of the CCV must agree with the expected value within $\pm 20\%$ in order for the analysis to continue.

- 9.4.3 Mercury by CVAA - Methods 7470, 7471A, and the CLP SOW - Cold-vapor atomic absorption (CVAA) techniques are used for the determination of mercury. Sample preparation is specified in the method. Following dissolution, mercury in the sample is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer.

Calibration - The calibration procedure is detailed in SW-846 and the CLP SOW. Prior to the analysis of samples, a multipoint initial calibration is performed. The calibration correlation coefficient must be ≥ 0.995 to be acceptable. The initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree with the expected response within $\pm 20\%$ in order for the calibration to be verified. A CCV check standard is analyzed following the analysis of the ICV and after the analysis of every 10 samples. The response of the CCV must agree with the expected value within $\pm 20\%$ in order for the analysis to continue.

- 9.4.4 Total and Amenable Cyanide - Method 9010A/ 9012 and the CLP SOW
These methods are used to determine the concentration of inorganic cyanide in aqueous or solid samples. These methods are used to determine values for both total cyanide and cyanide amenable to chlorination. Cyanide, as hydrocyanic acid (HCN), is released by refluxing the sample with strong acid and distilling the HCN into an absorber-scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by UV spectrophotometry.

Calibration - Prior to sample analysis, a multipoint initial calibration is performed. The calibration correlation coefficient must be ≥ 0.995 to be acceptable. The initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree

with the expected response within $\pm 10\%$ for the calibration to be verified. A CCV check standard is analyzed following the analysis of the ICV and after the analysis of every 10 samples. The response of the CCV must agree with the expected value within $\pm 10\%$ for analysis to continue.

9.4.5 Anions - Method 300.0

Method 300.0 may be used to analyze anions, including chloride, nitrite, nitrate, o-phosphate, bromide, and sulfate, in aqueous samples by ion chromatography (IC). A volume of sample is injected into the ion chromatograph. The anions of interest are separated and measured using a chromatography system consisting of a guard column, separator column, suppressor device and conductivity detector. Samples must be refrigerated at 4°C and analyzed within 48 hours of sample collection if nitrate, nitrite, and/or o-phosphate are analyzed, or within 28 days of sample collection if chloride, bromide and/or sulfate are analyzed.

Calibration - Prior to sample analysis, a multipoint initial calibration is analyzed. The calibration correlation coefficient must be ≥ 0.995 to be acceptable. The initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree with the expected response within $\pm 10\%$ for the calibration to be verified. A CCV check standard is analyzed following the analysis of the ICV and after the analysis of every 10 samples. The response of the CCV must agree with the expected value within $\pm 15\%$ for analysis to continue.

9.4.6 pH - Methods 150.1, 9040, & 9045A

Methods 150.1 and 9040 are used to measure the pH of aqueous and multiphase samples where the aqueous phase constitutes at least 20% of the total sample volume. The pH of the sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode. Method 9045A is used to determine the pH in soil samples.

Calibration - The pH meter is calibrated with three standard buffer solutions. The reading must be within $+0.05$ to ± 0.1 pH units (depending upon the instrument) of the true value of each buffer solution.

9.4.7 Non-Filterable Residue - Method 160.1

This method is applicable to drinking, surface and saline waters, and domestic and industrial wastes. A well mixed sample is filtered through a glass fiber filter. The residue that passes through the filter is dried and measured gravimetrically.

Calibration - The analytical balance must be checked each day of use with ASTM Class 1 weights. Balance readings must read within ± 0.001 g of the true weight.

9.4.8 Filterable Residue - Method 160.2

This method is applicable to drinking, surface and saline waters, and domestic and industrial wastes. A well mixed sample is filtered through a glass fiber filter. The residue on the filter is dried and measured gravimetrically.

Calibration - The analytical balance must be checked each day of use with Class S weights. Balance readings must read within ± 0.001 g of the true weight.

9.4.9 Nitrate-Nitrite - Method 353.2

Method 353.2 is used to determine the concentrations of nitrate and nitrite in aqueous samples. Nitrite concentration is determined by diazotization with sulfanilamide and complexation with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically. Combined nitrate-nitrite concentration is determined by first carrying out a copper-cadmium reduction step. A filtered sample is passed through a column containing granulated copper and cadmium to reduce nitrate to nitrite. Nitrate concentration is determined from the difference of the nitrate-reduced nitrite value and the nitrite value. Samples must be preserved with sulfuric acid to pH ≤ 2 and refrigerated at 4°C. If analysis for NO₂ or NO₃ only is desired, no preservative should be used.

Calibration - Prior to sample analysis, a multipoint initial calibration is analyzed. The calibration correlation coefficient must be ≥ 0.995 to be acceptable. The initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree with the expected response within $\pm 15\%$ for the calibration to be verified. A CCV check standard is analyzed following the analysis of the ICV and after the analysis of every 10 samples. The response of the CCV must agree with the expected value within $\pm 15\%$ for analysis to continue.

9.4.10 Total Organic Carbon (TOC) - Methods 9060 and 415.1

Methods 9060 and 415.1 are used to determine the concentration or total organic carbon in samples. TOC is analyzed by combustion of organic material in the sample to carbon dioxide, followed by infrared (IR) detection of the carbon dioxide.

Calibration - The instrument is calibrated by analyzing four replicates of a single concentration standard. The initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree with the expected response within $\pm 15\%$ for the calibration to be verified. A CCV check standard is analyzed following the analysis of the ICV and after the analysis of every 15 samples. The response of the CCV must agree with the expected value within $\pm 20\%$ for analysis to continue.

9.4.11 Oil and Grease - Methods 9070/9071A and 413.1

Methods 9070 and 413.1 are used to determine the concentration of oil and grease in waters and wastes. The aqueous sample is acidified with HCl to pH < 2 and extracted with Freon-TF (1,1,2-trichloro-1,2,2-trifluoroethane) in a separatory funnel. Sample extracts are evaporated to dryness and measured gravimetrically on an analytical balance. Method 9071A is used to prepare solid samples for gravimetric analysis of oil and grease. By this method, solid samples are Soxhlet

extracted with Freon-TF and the extracts are evaporated to dryness and measured gravimetrically on an analytical balance.

Calibration - A balance calibration check is performed at the beginning and end of each analytical sequence with 1 g and 100 g ASTM Class 1 weights. Measurements must agree to within ± 0.001 g of the true weight.

9.4.12 Oil and Grease - Method 413.2

This method is used to determine the concentration of oil and grease in waters and wastes. Samples are acidified with HCl to pH <2 and extracted with Freon-TF in a separatory funnel. Sample extracts are analyzed by infrared (IR) spectrophotometry.

Calibration - Prior to sample analysis, a multipoint initial calibration is performed. The calibration correlation coefficient (r) must be ≥ 0.995 for the calibration to be acceptable. A continuing calibration standard is analyzed after the analysis of every 10 samples. The continuing calibration must agree with the initial calibration within $\pm 20\%$.

9.4.13 Total Recoverable Petroleum Hydrocarbons (TRPH) - Method 418.1

This method is used to determine the concentration of total petroleum hydrocarbons in waters and wastes. The sample is acidified with HCl to pH <2 and extracted with Freon-TF in a separatory funnel. Extracts are shaken with silica gel to remove interferences, then the extracts are analyzed by infrared (IR) spectrophotometry.

Calibration - Prior to sample analysis, a multipoint initial calibration is performed. The calibration correlation coefficient (r) must be ≥ 0.995 for the calibration to be acceptable. A continuing calibration standard is analyzed after the analysis of every 10 samples. The continuing calibration must agree with the initial calibration within $\pm 20\%$.

9.5 METHOD VALIDATION

When non-promulgated methods (i.e. methods other than EPA, NIOSH, ASTM, AOAC, etc.) are required for specific projects or analytes of interest, or when the laboratory develops a method, the laboratory establishes the validity of the method prior to applying it to client samples. Method validity is established by meeting certain criteria for precision and accuracy as established by the data quality objectives specified by the end user of the data.

9.6 METHOD DETECTION LIMITS

Method detection limit studies are performed for each method in use at least annually and after any procedural or configurational change.

Method detection limits are determined at Pace for analyses done on samples originating under Safe Drinking Water Act (SDWA) and Clean Water Act (CWA) provisions by using replicate spiked analyte-free water samples. A minimum of seven replicates of a sample spiked for the purpose are processed through the entire analytical method. The concentration of the detection limit sample should be between 2 and 5 times the anticipated detection limit.

The laboratory calculates the detection limit as the Student's $t(n-1)$ value (e.g., t value = 3.143 for seven replicate determination) times the standard deviation of 7 replicate spiked sample measurements. The reader is referred to 40 CFR Part 136, Appendix B for further discussion.

For samples which are analyzed by methodology approved under the Resource, Conservation and Recovery Act (RCRA), the MDL is determined by multiplying the appropriate one-sided 99% t -statistic by the standard deviation obtained from a minimum of three analyses of a matrix spike containing the analyte of interest at a concentration three to five times the estimated MDL, where the t -statistic is obtained from standard references. Estimate the MDL by obtaining the concentration value that corresponds to: a) an instrument signal/noise ratio within the range of 2.5 to 5.0, or b) the region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve). The reader is referred to SW-846, Third Edition, Chapter One, Volume 1A for further discussion.

IT IS IMPERATIVE TO NOTE THAT METHOD DETECTION LIMITS ARE HIGHLY MATRIX DEPENDENT. LIMITS DETERMINED BY Pace MAY NOT BE ACHIEVABLE IN ALL MATRICES.

9.7 COMPLIANCE

9.7.1 Definition - Compliance is the proper execution of recognized, documented procedures which are either approved or required. Adherence to these procedures is required in order to provide data products acceptable to a regulatory body of competent jurisdiction in a specific regulatory context. Compliance is separate from, but not inconsistent with, technical scientific quality. Pace accepts compliance as part of the Pace corporate definition of quality: "Quality is the *fulfillment of expectations and needs* in all activities, demonstrated by the satisfaction of those we serve." Pace understands that the expectations of our clients commonly include the assumption that data and reports will satisfy a regulatory purpose and will be found acceptable *and compliant* with regulatory requirements for the performance of tests and generation of data.

9.7.2 Understanding the Regulatory Framework - Compliance is not likely to be achieved in the absence of an understanding of the regulatory framework. Pace will attempt to ascertain, prior to beginning a project, what regulatory jurisdiction (USEPA, NJDEPE, etc.) pertains to a project; within the regulatory jurisdiction, what body of regulation is meant to be satisfied (RCRA, SDWA, 21E, etc.); and finally, within this context, what protocols are required/expected (CLP, AFCEE, NFESC, ASP, etc.).

Pace will work with its clients to come to a mutual understanding of all requirements.

- 9.7.3 Commitment - Experience has shown that the complexity of environmental regulations and their overlapping jurisdiction can result in conflicting DQOs to be established for a project or site by local, state and federal regulatory agencies. As a result of these types of complicating factors, clients and intermediaries working on their behalf may, but often do not, fully understand their compliance needs. Clients may sometimes fail to communicate their compliance requirements to Pace. Nevertheless, Pace Analytical Services, Inc., in defining quality as in 9.7.1 above, has accepted much responsibility for compliance.

Pace makes the following commitments to its clients:

- Pace will proactively attempt to identify and understand the regulatory context of clients' needs.
- Pace will strive to be expert in understanding and executing the regulatory requirements for compliance.
- Pace will identify and disclose to clients instances of non-compliance in a forthright fashion.

- 9.7.4 Resolving Compliance Contradictions and Hierarchies - It is a common occurrence that multiple regulatory jurisdictions overlap in a specific case. This causes uncertainty or even contradictions to arise in a work plan. Pace will make every effort to detect such inconsistencies, and will communicate them to clients so that an informed decision can be made by the client regarding execution of the project. Similarly, methods and protocols will often be prescribed in a scope of work or QAPP which either will not achieve stated or implied DQOs or which are in conflict with the regulatory requirements. Pace will attempt to detect these inconsistencies, and upon detection, disclose same to our client. Pace voluntarily accepts a responsibility to provide advice to clients, however, **the primary responsibility for this issue remains with the client.**

- 9.7.5 Disclosure of Noncompliance - As stated previously, it is Pace policy to disclose in a forthright manner any detected noncompliance that may effect the usability of data produced by Pace. It is not within our expertise to predict the manner in which a specific regulator or regulatory body will interpret the rules governing analysis; therefore, Pace is unable to guarantee compliance. It is Pace policy that our responsibility begins with a bona fide and competent attempt to evaluate potential compliance issues and ends with disclosure of any findings that may be useful to our client in their making the final judgment.

TABLE 9.1**ANALYTICAL PROTOCOLS**

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, October 26, 1984.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846. 2nd edition, 1982 (revised 1984), 3rd edition and 1st Update, Update II and IIA, 1994, Office of Solid Waste and Emergency Response, U.S. EPA.
- "Methods for Chemical Analysis of Water and Wastes", EPA 600/4-79-020, 1979 Revised 1983, U.S. EPA.
- U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis, SOW 2/88, OLM01.8, 8/91, OLM02.0, and OLM03.0.
- U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis, SOW No. 788, ILM01.0, 3/90 through ILM03.0.
- "Standard Methods for the Examination of Water and Wastewater", 15th, 16th, 17th and 18th editions, 1980, 1985, 1989, 1992. APHA-AWWA-WPCF.
- "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society for Testing and Materials, 1987.
- "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society for Testing and Materials, 1987.
- "NIOSH Manual of Analytical Methods", Third Edition, 1984, U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health.
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory - Cincinnati (September 1986).
- New York State Department of Environmental Conservation. Analytical Services Protocol, September, 1989 (revised December 1991).

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Table 9.2
LIST OF ANALYTICAL METHODS

1. Organic Analyses

Parameter	Method	DW Method	WW Method	SW-846	Specialty
Halogenated Volatile Organics	GC		601	8010A	
Non-Halogenated Volatile Organics	GC			8015A	
Purgeable Aromatics and Unsaturated Organics	GC	503/5022	602	8020A/8021A	
Acrolein and Acrylonitrile	GC		603	8030A	
Organochlorine Pesticides and Polychlorinated Biphenyls	GC	508	608/608.1 608.2	8080A	Mod 8080
Polynuclear Aromatic Hydrocarbons	GC			8270B	
Chlorinated Hydrocarbons	GC	1613	612	8120A	
Base/Neutrals & Acids	GC/MS	525	625	8270B	
Organophosphorus Pesticides	GC		614/622	8140	
Chlorinated Herbicides	GC	515.1	615/608.1 608.2	8150B	
Volatile Organic Compounds	GC/MS	524.2	624	8240B/ 8260A	
Fuel Hydrocarbons and BTEX	GC or IR		602/418.1	8015A	
Alachlor, Atrazine	GC		619/645	8080A/8140	
Chlordane, Heptachlor, Heptachlor Epoxide, Lindane, Methoxychlor	GC		608/617	8080A	
Carbofuran	HPLC	531.1			
Endothall	GC	548			
Total Petroleum Hydrocarbons	IR		418.1		

Table 9.2 (cont.)

2. Inorganic Analyses

Parameter A. Non Metals	Method	Standard Methods 18th Edition	EPA Methods 1983	ASTM	SW-846
Acidity	Potentiometric Titration	2310	305.1	D1067	
Alkalinity	Potentiometric Titration	2320	310.1	D1067	
Biochemical Oxygen	5-Day, 20°C		405.1		
Boron	ICP		200.7		6010
Bromide	Ion Chromatography		300.0		
Chemical Oxygen Demand	Dichromate Reflux (High)	5220	410.1	D1252	
	Dichromate Reflux (Low)	5220	410.2		
Chloride	Mercuric Nitrate	4500-Cl ⁻	325.3	D512	9252
Chloride	Ion Chromatography		300.0	D512	
Chlorine, Residual	Titration Colorimetric	4500-Cl ⁻ 4500-Cl ⁻	330.5		
Color	Visual	2120	110.2		
	Comparison		110.3		
Cyanide, Total	Pyridine-Barbituric Acid, Colorimetric	4500-CN ⁻	335.2	D2036	9010A 9012
Amenable	Chlorination- Colorimetric	4500-CN ⁻	335.1	D2036	9010A 9012
Fluoride, Total	Distillation- Electrode	4500-F ⁻	340.2	D1179	
Fluoride, Dissolved	Electrode Ion Chromatography	4500-F ⁻	340.2 300.0	D1179 D1179	
Hardness, Total	EDTA Titration Calculation	2340	130.2	D1126	

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Table 9.2 (cont.)

Parameter A. Non-Metals	Method	Standard Methods 18th Edition	EPA Methods	ASTM	SW-846
Hardness, Calcium	EDTA Titration	3111	242.1	D511	
Hardness, Calcium Calculation			200.7		
Iodine(ide)	Ion Chromatography		300.0		
Nitrogen, Ammonia	Distillation Titration Potentiometric	4500-NH ₃	350.2 350.3		
Kjeldahl	Digestion/Distillation	4500-N organic	351.3	D3590	9200
Nitrate	Automated Cadmium Brucine Sulfate	4500-NO ₃	353.2 352.1	D3867 D091	
Nitrite	Ion Chromatography Automated Cadmium	4500-NO ₃	300.0 353.2	D3867	
Nitrite	Ion Chromatography Spectrophotometric	4500-NO ₂	300.0 354.1		
Organic	Kjeldahl-NH ₃ Kjeldahl-Potentiometric	4500-N organic	351.3 351.4	D3590	
Oil and Grease	Soxhlet Partition-Gravimetric IR	5520B 5520B	413.1 413.2		9070 9071
pH (Hydrogen Ion)	Electrode	4500-H+	150.1	D1293	9040A
Phenol	Distillation-Extraction Colorimetric		420.1	D1783	9065
Phosphorus Total	Persulfate Digestion Ascorbic Acid Reduc.	4500-P	365.2	D515	
Ortho	Ascorbic Acid Reduc.	4500-P	365.2	D515	
Silica, Dissolved	Molybdosilicate ICP	4500-S1	370.1 200.7	D859	

Table 9.2 (cont.)

Parameter A. Non-Metals	Method	Standard Methods 18th Edition	EPA Methods 1983	ASTM	SW-846
Solids					
Total	Gravimetric	2540	160.3		
Total Volatile	Gravimetric	2540	160.4		
Suspended	Gravimetric	2540	160.2		
Suspended Volatile	Gravimetric	2540	160.4		
Total Dissolved	Gravimetric	2540	160.1		
Specific Conductance	Meter	2510	120.1	D1125	9040A
Sulfate	Ion Chromatography Turbidimetric	4500-SO ₄ ²⁻	300.0	D516	9035
			375.2		
			375.4		
Sulfide	Colorimetric Titration	4500-S ²⁻ 4500-S ²⁻	376.2		9030A
Sulfite	Titration	4500-SO ₃ ²⁻	377.1	D1339	
Surfactants (MBAS)	Methylene Blue	5540	425.1	D2330	
Turbidity	Meter	2130	180.1	D1889	

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Table 9.2 (cont.)

Parameter B. Metals	Method	Standard Methods 18th Edition	EPA Methods 1983	SW-846
Aluminum	AA-Direct Aspiration	3111	202.1	7020
	AA-Furnace	3113	202.2	
	ICP-AES		200.7	6010A
Antimony	AA-Direct Aspiration	3113	204.1	7040
	AA-Furnace	3113	204.2	7041
	ICP-AES		200.7	6010A
Arsenic	AA-Furnace	3313	206.2	7060A
	ICP-AES		200.7	6010A
Barium	AA-Direct Aspiration	3110	208.1	7080A
	AA-Furnace	3113	208.2	7081
	ICP-AES		200.7	6010A
Beryllium	AA-Direct Aspiration	3110	210.1	7090
	AA-Furnace	3113	210.2	7091
	ICP-AES		200.7	6010A
Cadmium	AA-Direct Aspiration	3110	213.1	7130
	AA-Furnace	3113	213.2	7131A
	ICP-AES		200.7	6010A
Calcium	AA-Direct Aspiration	3110	215.1	7140
	AA-Furnace	3500-Ca	215.2	
	ICP-AES		200.7	6010A
Chromium, Total (Hexavalent)	AA-Direct Aspiration	3110	218.1	7190
	AA-Furnace	3113	218.2	7191
	ICP-AES		200.7	6010A
	Colorimetric MIBK Extraction	3500-Cr		7196A 7197
Cobalt	AA-Direct Aspiration	3110	219.1	7200
	AA-Furnace	3113	219.2	7201
	ICP-AES		200.7	6010A
Copper	AA-Direct Aspiration	3110	220.1	7210
	AA-Furnace	3113	220.2	7211
	ICP-AES		200.7	6010A
Iron	AA-Direct	3110	236.1	7380
	AA-Furnace	3113	236.2	7381
	ICP-AES		200.7	6010A

Table 9.2 (cont.)

Parameter B. Metals	Method	Standard Methods 18th Edition	EPA Methods 1983	SW-846
Lead	AA-Direct Aspiration	3110	239.1	7420
	AA-Furnace	3113	239.2	7421
	ICP-AES		200.7	
Lithium	AA-Direct Aspiration	3500-Li		6010A
Magnesium	AA-Direct Aspiration	3111	242.1	7450
	ICP-AES		200.7	6010A
Manganese	AA-Direct Aspiration	3111	243.1	7460
	AA-Furnace	3113	243.2	7461
	ICP-AES		200.7	6010A
Mercury	AA-Cold Vapor	3112	245.1	7470A
	Automated Cold Vapor		245.2	7471A
Molybdenum	AA-Direct Aspiration	3111	246.1	7480
	AA-Furnace	3113	246.2	7481
Nickel	AA-Direct Aspiration	3111	249.1	7520
	AA-Furnace	3113	249.2	6010A
	ICP-AES		200.7	
Potassium	AA-Direct Aspiration	3111	258.1	7610
	ICP-AES			6010A
Selenium	AA-Furnace	3113	270.2	7741A
			200.7	7740
	ICP-AES			6010A
Silica	ICP-AES		200.7	6010A
Silver	AA-Direct Aspiration	303A	272.1	7760A
	AA-Furnace	3113	272.2	7761
	ICP-AES		200.7	6010A
Sodium	AA-Direct Aspiration	3111	273.1	7770
	ICP-AES		200.7	6010A
Strontium	AA-Direct Aspiration	3111		7780
	ICP-AES		200.7	6010A

Table 9.2 (cont.)

Parameter B. Metals	Method	Standard Method 18th Edition	EPA Methods 1983	SW-846
Thallium	AA-Direct Aspiration	3111	279.1	7840
	AA-Furnace	3113	279.2	7841
	ICP-AES		200.7	6010A
Tin	AA-Direct Aspiration	3111	282.1	7870
	AA-Furnace	3113	282.2	6010A
	ICP-AES		200.7	
Titanium	AA-Direct Aspiration	3111	283.1	
	AA-Furnace	3113	283.2	6010A
	ICP-AES		200.7	
Vanadium	AA-Direct Aspiration	3111	286.1	7910
	AA-Furnace	3113	286.2	7911
	ICP-AES		200.7	6010A
Zinc	AA-Direct Aspiration	3111	289.1	7950
	AA-Furnace	3113	289.2	7951
	ICP-AES		200.7	6010A

Table 9.2 (cont.)

3. Wastes & Oil Analysis

Parameter	Method	Standard Methods 18th Edition	ASTM	SW-846
% Ash	Gravimetric	2540		
Density	Gravimetric	2710		
Flash Point Closed Cup	TAG		D93-80	1010
Free Liquids	Paint Filter			9095
Leach Test. EP Toxicity	Extraction			1310A
Sulfide, Total	Titration			9030A
Reactive	Titration		261.23	Chapter 7-7.3.4.2
pH	Electrode			9040A
Specific Conductance	Meter			9050/9045B
Specific Gravity	Mass & Displacement	2710		
Cyanide, Total	Distill -Color			9010A
Amenable	Chlorination- Colorimetric			9010A
Reactive	Purge-Color		261.23	Chapter 7-7.3.3.2
TCLP	Leach		40CFR268	1311

Table 9.2 (cont.)

6. **List of Sample Preparation Methods**

1311	TCLP
1312	Synthetic precipitation leaching procedure
3015	Microwave dig. aqueous
3051	Microwave dig. sludges, oil soil
3510	Separatory Funnel Liquid - Extraction
3520	Continuous Liquid - Extraction
3540	Soxhlet Extraction
3541	Automatic soxhlet extraction
3550	Sonication Extraction
3640	Gel Permeation Chromatography
3580	Waste Dilution
3630	Silica gel
3660	Sulfur clean up
5050	Bomb combustion. method for T. Halides
5080	Purge and Trap
3005	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by Flame AA or ICP
3010	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by Flame AA or ICP
3020	Acid Digestion of Aqueous Samples and Extracts for Total Metals by Furnace AA
3050	Acid Digestion of Sediments, Soils, and Sludges

7. **Screening Methods**

3810	Headspace
3820	Hexadecane extraction and screening of purgeable organics

8. **Other**

40 CFR 261	Characteristic of Ignitability
40 CFR 261	Characteristic of Corrosivity
40 CFR 261	Characteristic of Reactivity
40 CFR 261	TCLP
NIOSH 0600	Nuisance Dust, Respirable
NIOSH 0500	Nuisance Dust, Total
NIOSH 7500	Respirable Silica (XRD)

Table 9.2**References**

1. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, U.S. EPA 600/4-79-019, March, 1979.
2. Federal Register, 40 CFR Part 136, October 26, 1984.
3. Test Methods for Evaluating Solid Waste. Physical/Chemical Methods, SW-846, 3rd Edition & Final Updates One and Two , U.S. EPA, revised Sept., 1994.
4. Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.
5. Standard Methods for the Examination of Water and Wastewater, APHA, AWWA, WPCF: 18th Edition, 1992.
6. NIOSH Manual of Analytical Methods, U.S. Department of Health, Education, and Welfare; Second Edition, 1977.
7. Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079-C.
8. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, 1983.
9. The Determination of Inorganic Anions in Water by Ion Chromatography - Method 300.0 Test Method, EPA-600/4-84-017. March, 1984.
10. Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.
11. Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.
12. Requirements for Quality Control of Analytical Data for the Environmental Restoration Program, Martin Marietta, ES/ER/TM-16, December, 1992.
13. Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, 1988.

Table 9.3 BFB Key Ions and Ion Abundance Criteria

Mass	SW-846, Method 8240B	CLP Statement of Work - VOA
50	15-40% of mass 95	8-40% of mass 95
75	30-60% of mass 95	30-66% of mass 95
95	base peak, 100% of rel. abundance	base peak, 100% rel. abundance
96	5-9% of mass 95	5-9% of mass 95
173	less than 2% of mass 174	less than 2% of mass 174
174	greater than 50% of mass 95	50-120% of mass 95
175	5-9% of mass 174	4-9% of mass 174
176	95-101% of mass 174	93-101% of mass 174
177	5-9% of mass 176	5-9% of mass 176

Table 9.4 DFTPP Key Ions and Ion Abundance Criteria

Mass	SW-846, Method 8270B	CLP Statement of Work - SV
51	30-60% of mass 198	30-80% of mass 198
68	<2% of mass 69	<2% of mass 69
69	N/A	Present
70	<2% of mass 69	<2% of mass 69
127	40-60% of mass 198	25-75% of mass 198
197	<1% of mass 198	<1% of mass 198
198	base peak, 100% rel. abundance	base peak, 100% rel. abundance
199	5-9% of mass 198	5-9% of mass 198
275	10-30% of mass 198	10-30% of mass 198
365	<1% of mass 198	<0.75 of mass 198
441	Present but less than mass 443	Present but less than mass 443
442	<40% of mass 198	40-11-% of mass 198
443	17-23% of mass 442	15-24% of mass 442

10.0 DATA REDUCTION, VALIDATION AND REPORTING

Data reduction, validation and reporting describes the processes that result in the delivery of quantitative analytical data to the data user. These processes include calculation of raw data into final concentration units, reviewing results for accuracy and assembly of the technical report contents for delivery to the data user.

All analytical data generated within the Pace laboratories undergo a well-defined, well-documented multi-tier review process before being reported to the client. The following describes procedures employed at Pace for translating raw analytical data into accurate, finished sample reports and data storage. Figure 10.2 shows schematically the sample flow through the laboratory, while Figure 10.3 shows the parallel flow of information concerning the sample analysis and reporting.

10.1 DATA REDUCTION

When primary analytical data, otherwise known as "raw data," are manually generated, the data are recorded either in bound logbooks with prenumbered pages or on preprinted forms. Records of analysis indicate the method used, raw data, calculations, and final results. Entries are made in black ink and are initialed and dated by the individual who makes the entry. It is acceptable to initial and date once for an entire page. Errors are corrected by drawing a single line through the entry; this change is initialed and dated by the individual who makes the change. Raw data may not be obscured in any way. The use of white-out is prohibited on all raw data, including instrumental hardcopy.

All data generated by Pace are reviewed by designated, trained personnel. The analysts who acquire the data are responsible for initial on-line checks for compliance to the analytical requirements. After a sample batch is acquired, the data review procedure includes data interpretation and quantitation, inspection of quality control data against criteria, data reduction, narrative or comments writing, and ensuring that the data package includes all required analytical and quality control results, raw data and laboratory chronicles. The analyst who completes the analysis assembles all relevant raw data and results together with chromatograms, strip chart recordings, instrument settings and other information essential to data interpretation. For data which are reduced by manual calculations, the calculations are documented in a laboratory notebook or on an analyst's worksheet. The results are transferred to a standardized laboratory reporting form which has been approved by the appropriate Group Supervisor and Laboratory Operations Manager. Reporting forms include at a minimum the sample identification number, the date analyzed, the result expressed per unit volume, the method reference and the analyst's initials. From the reporting forms, the results are entered into the LIMS.

10.2 DATA VALIDATION

Data validation is the process of examining data and accepting or rejecting it based on pre-defined criteria. Pace data review personnel use the following criteria to validate laboratory data:

-
- Use of approved analytical procedures.
 - Use of properly operating and calibrated instrumentation.
 - Precision and accuracy comparable to that achieved in similar analytical programs.

Analysts performing the analysis and subsequent data reduction have the primary responsibility for the quality of the data produced. The primary analyst initiates the data validation process by reviewing and accepting the data, provided QC criteria have been met for the samples being reported. Data review checklists may be used to document the data review process.

The completed data package is then sent to the Group Supervisor or designated reviewer. The Group Supervisor provides a technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This involves a quality control audit for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. Group Supervisors also review analyst generated calculations.

For data which are reduced via computer, calculations are checked by the analyst (or designee) assigned to this task at a frequency designed to assure that the data manipulations are valid. This data validation step is documented by the analysts' initials on the hardcopy of the raw data. The results are either manually transferred to a standard reporting form or reported via computer generation of forms.

Once the data have been technically reviewed and approved, authorization for release of the data from the analytical section is indicated by initialing and dating the data review checklist or otherwise initialing and dating the data. The Group Supervisor drafts any narrative comments if required by the Quality Assurance Project Plan, and forwards the report and the data package to the Reporting Department.

Each data package is reviewed by designated reporting personnel to ensure compliance with client orders by reviewing on-line input in the Pace computer tracking system. The laboratory data are assembled in the client's technical reports. Reports are reviewed for completion prior to copying and binding. Figure 10.1 provides a summary listing of staff responsibilities concerning data generation, review, validation and reporting. The Reporting Department assembles the data with other data from the sample set, generates the final report, checks for transcription errors, and provides the final report to either the Laboratory Operations Manager, Project Manager, or an appropriate designee for final signature.

The Operations or Project Manager examines the report for method appropriateness, detection limits and whether or not QC criteria were satisfied. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution. The Operations or Project Manager or an appropriate designee signs the completed report prior to its release to the client.

The Operations Manager may delegate the final review and signing of reports as necessary.

Use of checklists ensure that all data are systematically handled and no steps are omitted. Checklists are reviewed and are retained and accessible should they need to be referenced at a later date. The data and deliverables are checked and signed during processing procedures and then systematically filed by reference identification numbers.

10.3 DATA REPORTING

All data segments pertaining to a particular Pace Laboratory Number are channeled to the Reporting Department for assembly into the final report format and generation of the analytical narrative. All points mentioned during technical and QC review are included in the narrative if it is deemed to impact the quality of the data.

The final report is given to either the Laboratory Operations Manager, Project Manager or an appropriate designee for final review and release. After verifying the report's completeness and accuracy, the Operations or Project Manager signs the cover letter or authorization line within report indicating acceptance of the report.

Technical reports are prepared to include the components or level of deliverables that are requested by clients for samples or projects, or contractually required. The standard Pace commercial report to the client consists of the following sections:

- 1) A cover letter
- 2) A technical narrative (when necessary)
- 3) Sample receipt condition report (information may be included on C.O.C. form)
- 4) Sample ID table
- 5) Sample results
- 6) Chain-of-Custody forms

The narrative briefly describes the condition of the samples upon receipt, sample holding time performance, instrument calibration information, and the quality control results. Any discrepancies discovered and matrix problems encountered are also addressed in this section.

The sample results are tabulated by sample number and parameter. Pace number, client identification, and dates of sample preparation and analysis are presented along with the observed concentrations for each parameter analyzed and corresponding reporting limits.

Pace prepares technical reports that include full data deliverables for validation purposes, and lesser, abbreviated reports. Full deliverables include all raw and processed data applicable to the analyses performed. Pace prepares single sample technical reports or multi-sample report packages. The multi-sample technical reports contain results for a sample delivery group (SDG) or other client or laboratory defined sample set. Pace recommends multi-sample reports when full deliverables packages are required.

The Pace laboratory prepares electronic data deliverables (EDD) as required for contracts and upon client request.

10.4 DATA ARCHIVE

Each data report which supports the analytical process for all samples received by the laboratory is thoroughly reviewed for completeness and accuracy. After the technical review it is routed to the Reporting Department for assembling the final report for submission to the client. The report is approved, signed, and submitted.

Sufficient records are retained to recreate analytical events at the laboratory. Pace will retain analytical data for five years and financial data for three years relating to services performed following the transmittal of the final report to the client. Certain contractual arrangements or regulatory requirements for specific projects may shorten or extend the records retention period stated here. Records are catalogued and maintained in limited access areas. Data archive and storage is managed by designated individuals who control the access to stored information. One copy of the report remains with all the raw data which is stored in the data archives under the control of the QA Department or other designated group.

All information retained at the Pace facility is stored in secured areas. The Data Archivist has oversight responsibility for the data archive ensuring the continued integrity of all documentation generated in support of laboratory analyses. All hard-copy information is stored on-site at the laboratory or off-site at a commercial document storage facility equipped with a professional security system. All electronic data is stored on-site at the laboratory or off-site at a commercial document storage facility equipped with a professional security system and a controlled environment suitable for storage of magnetic media.

The archive room is a secure storage area with limited access to non-authorized personnel. Sign-out procedures are in place where every document removed from the archive room must be signed out by authorized personnel.

A copy of the report or summary of samples classified as hazardous is forwarded to the Hazardous Waste Coordinator or designee for use in characterizing the samples for ultimate disposal.

Pace reserves the right to transfer hard-copy information onto microfilm or write-protected electronic media. Pace reserves the right to store information in hard-copy files, on magnetic media and/or microfilm. The information is retained and accessible for a minimum of seven years unless otherwise specified through a client specific contract.

10.5 RESPONSE TO INQUIRIES

The Pace laboratory which conducted analyses for the client recognizes the importance of its timely response to inquiries regarding the laboratory's work for samples and

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projects. The laboratory will respond to inquiries as rapidly as possible as part of its corrective action plan. The Pace laboratory which originally received samples from the client should be considered the primary contact for all data inquiries when subcontract or other Pace laboratories are used for analyses.

FIGURE 10.1
Analytical Data Review Process, Pace Analytical Services, Inc.

Responsibilities	
Analyst	<ul style="list-style-type: none"> · Sample analysis LIMS* entry and generation · Data review - 1st level (bench) · Control charting - real time · Narrative notes · Discrepancy initiation · Provide copies of log books, as necessary
Supervisor	<ul style="list-style-type: none"> · Oversee daily analytical activities · Review control chart comments daily · LIMS data entry and validating · Draft and review of narrative · Supervise contractual and technical compliance · Discrepancy review · Review quality control daily (calibrations, etc.)
Manager	<ul style="list-style-type: none"> · Sign-off case narrative · Ensure program compliance · Review discrepancies requiring manager resolution · Technical conference calls with client · Ensure technical validity of data
Data Review/Reporting	<ul style="list-style-type: none"> · Generate forms package · Final data review and validation · Prepare package and paginate · Electronic deliverables generation · Maintain data package files
Quality Assurance Office	<ul style="list-style-type: none"> · 10 percent contractual compliance review (data packages) <ul style="list-style-type: none"> - Custody when required; - Calculations; - Methods criteria; - QC criteria; - Forms; and - Control charting.
Project Manager	<ul style="list-style-type: none"> · Review narratives for accuracy · Review packages for completeness and quality · Cover letter · Collate organic and inorganic packages · Client/laboratory liaison · Deliver package to client

Note: *Laboratory Information Management System

FIGURE 10.2
Laboratory Sample Flow Schematic

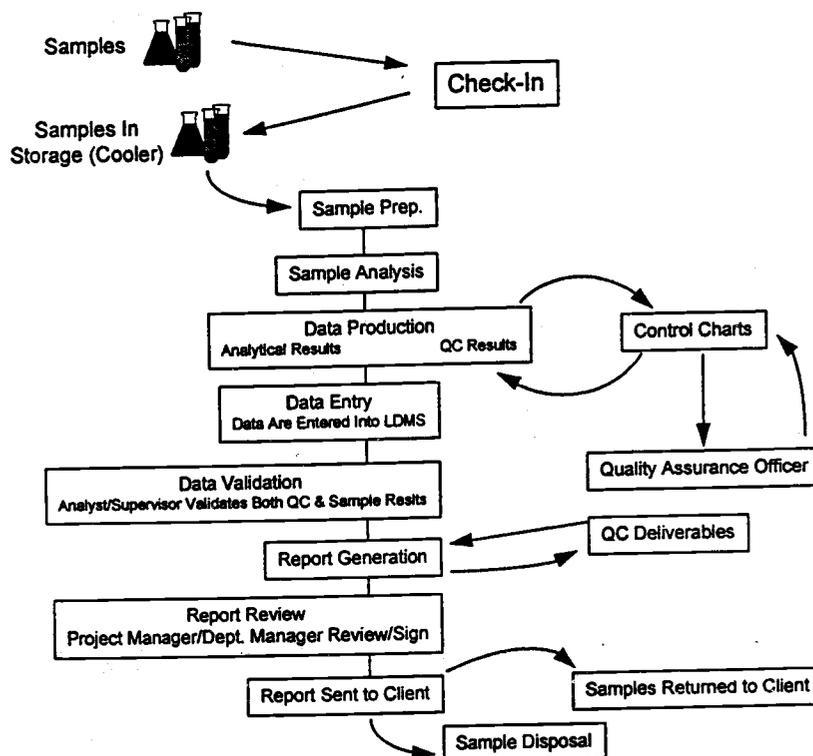
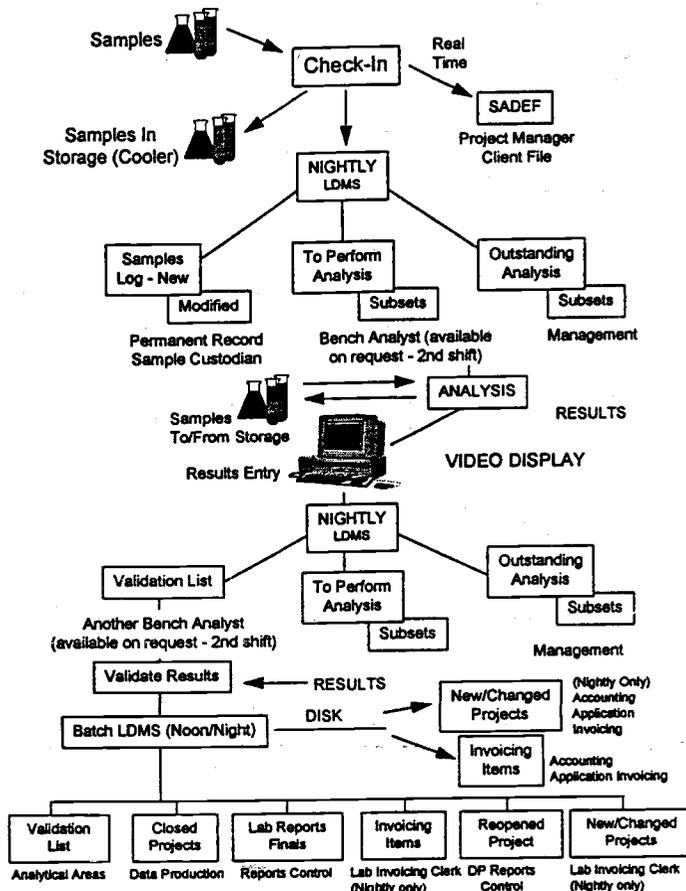


FIGURE 10.3
Information Flow Schematic



11.0 QUALITY CONTROL PROCEDURES

A quality control (QC) program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing expected control limits, using these limits to detect errors or out-of-control events, and requiring corrective action measures to prevent or minimize the recurrence of these events. QC procedures are implemented to ensure that sample data meet the quality objectives of the laboratory and the client. An effective QC program must be able to control the quality of the data through the monitoring of QC indicators. Criteria frequently applied to environmental QC data include measurements of accuracy and precision. Precision measures the randomness associated with an analytical measurement and reflects the inherent variability in that measurement system. Accuracy reflects the degree to which the measured value approximates the actual or "true" value for a given parameter and reflects the influence of systematic biases in the measurement. Thus, the "quality" of QC data can be said to be a measure of both the randomness and biases in a specific measurement system.

This section addresses the specific QC procedures applied to representative analytical methods performed at Pace. Table 11.1 presents method-specific information about QC procedures, acceptance criteria, and required corrective actions for the various analysis types.

11.1 ACCURACY AND PRECISION MEASUREMENT CONVENTIONS

The results of quality control samples created in the laboratory represent estimates of accuracy and precision for the preparation and analysis steps of sample handling. This section describes the quality control information provided by each of these analytical measurements. Information on the procedures to follow in preparation of the samples or spiking solutions is described for each method and matrix in the respective method Standard Operating Procedure.

Method Blank

A method blank is a volume of deionized and/or distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples, carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or weight processed. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and minimized. Optimally, a method blank should contain no greater than five times (5X) the method detection limit, or reporting limit where applicable, for common laboratory solvents and phthalate esters; less than the detection (or reporting) limit for all other parameters unless otherwise specified in the method or project QA plan. Results of method blank analyses are maintained with other QC data in the respective laboratories. If requested by the client, this data will be included in the report.

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Method Blank Frequency

Organics: The laboratory shall prepare and analyze one laboratory reagent blank (method blank) for each group of samples of a similar matrix (for water or soil samples), extracted by a similar method (separatory funnel, continuous liquid-liquid extraction, or sonication), and a similar concentration level (when low vs. medium level analyses are available) for:

- every 20 samples, or
- whenever samples are extracted

- whichever is more frequent.

Inorganics: At least one preparation blank (method blank), consisting of blank reagent water processed through each sample preparation and analysis procedure, shall be prepared and analyzed with every group of 20 samples, or with each batch (a group of samples prepared at the same time, e.g. daily) of samples digested, extracted, prepared or directly analyzed, whichever is more frequent.

Accuracy Measurements

Laboratory Control Samples (LCS) consist of aliquots of laboratory blank matrices (water, sand, etc.) spiked with analytes of interest. LCSs for methods with extensive lists of analytes that may interfere with one another may include a limited number of analytes, but the analytes included must be representative of as many analytes as is practical. In the case of metals analysis, all analytes of interest must be included. Laboratory pure water is used to prepare most LCSs for methods for analysis of water. Highly characterized solids, where available, are used for LCSs for methods for analysis of solids. Where no such solid LCS is available, spiked laboratory pure water or spiked reagent blanks may be substituted. LCSs provide an estimate of bias based on recovery of the compounds from a clean, control matrix. They provide evidence that the laboratory is performing the method within accepted guidelines without potential non-matrix interferences. They are prepared at a rate of one per batch of twenty or fewer samples.

For tests that are performed infrequently, an LCS shall be analyzed at least monthly if the number of samples is less than 20. This monthly requirement shall NOT apply to low-volume tests for which state certification is not sought, or for tests expected to be performed solely as part of a special project, or for tests involving study specific matrices other than water, soil, sludges and oils.

Matrix Spikes/Matrix Spike Duplicates are similar to Laboratory Control Samples except the analytes used for spiking are added to a second and third separate aliquot from the same container of selected client samples in a batch of analyses. They

enable one to assess sample matrix effects and field conditions. MS/MSDs are routinely prepared at a frequency of 5% (one set per twenty samples) when adequate sample volume is provided or once every 14 days, whichever is more frequent. An LCS/LCSD pair shall be substituted when sufficient sample volume is not available to prepare an MS/MSD sample set.

Surrogates provide an estimate of bias based on recovery of chemically similar compounds which are not expected to be in the sample, to the compounds of interest for each sample, incorporating sample matrix effects and field conditions. Surrogates are added to all samples analyzed by GC/MS and certain GC analyses prior to sample preparation.

An **Internal standard** is an analyte that has the same characteristics as the surrogate, but is added to each sample in a batch, just prior to analysis and is used for quantitation. It corrects for bias or change in instrument performance from sample to sample, incorporating matrix effects associated with the analytical process only.

Accuracy is expressed as % Recovery. For LCSs, Surrogate, and Blank Spike samples, percent recovery (%R) is calculated as:

$$\%R = (SR / SA) \times 100$$

Where: **SR** is the concentration determined
SA is the concentration spiked

For the matrix spike samples, the percent recovery is calculated as:

$$\%R = (SSR - SR) / SA \times 100$$

Where: **SSR** is the spiked sample determined result
SR is the original sample determined result
SA is the amount of spike added (expected)

Precision Measurements

A **Sample Duplicate** is a sample that has been homogenized and split into two equal portions before the method sample preparation process. It measures sample precision associated with the preparation through analysis and is prepared and analyzed at a rate of one per batch or one per twenty samples or once every 14 days whichever is greater in the inorganic laboratories. For organic analyses the MS/MSDs fulfill this function and provide a measure of overall precision.

The comparison of the values determined for a sample and its duplicate (MS/MSD) is expressed as relative percent difference (RPD). This calculation is as follows:

$$RPD = \frac{|S-D|}{[(S+D)/2]} \times 100$$

The vertical bars in the above equation indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

11.2 CONTROL CHARTS

Control charts are quality control tools which graphically display the QC parameters over time. Accuracy (Figure 11.1) and precision (Figure 11.2) control charts are generally maintained for each method; however, for certain methods tabulated control limits are used to monitor acceptability of quality control measurements. Each chart can be broken into three parts: sample identification, sample response/calculation, and graphic representation (the plot).

11.2.1 Accuracy

Accuracy charts are maintained for Surrogate and Laboratory Control Sample recovery. Each sample is identified by the date it was analyzed and its Pace sample number.

The percent recovery is plotted onto the graph where:

- The x-axis is the sample ID.; and
- The y-axis is the range of percent recoveries.

11.2.2 Precision

In cases where precision charts are maintained, the relative percent difference is plotted on the graph where:

- The median, zero, represents 0% difference
- The x-axis is the number of data points per chart; and
- The y-axis is the range of relative percent differences.

Both samples are identified by the date(s) analyzed and their Pace number.

11.2.3 Limits

Both upper and lower warning limits and upper and lower control limits are established to interpret performance. Warning limits express a narrower confidence interval and are used to warn the analyst or supervisor of possible system inconsistencies or failures, before an out-of-control event

occurs. Control limits express the outer limits of accepted method variability. Control limits and warning limits are reviewed periodically against performance. Based on statistical considerations, an evaluation is made to determine whether the control limits need to be revised.

Warning Limits

When not otherwise mandated by the method, Pace adopts warning limits to be the mean ± 2 standard deviations or a 95% confidence interval, where:

$$\text{Mean } \bar{x} = \frac{1}{n} \sum_{i=1}^n x_i$$

Standard Deviation

$$s = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad \text{or} \quad \sqrt{\frac{\sum_{i=1}^n x_i^2 - (\sum_{i=1}^n x_i)^2/n}{n-1}}$$

In this equation, n = population size
 x_i = ith observation in the sample
 \bar{x} = sample mean

Control Limits

Unless otherwise stipulated in a particular method or program, acceptance limits (control limits) will be statistically derived from laboratory generated data. Control limits will be based upon $\pm 3s$ (i.e., 99% confidence interval) from the mean and warning limits established at $\pm 2s$ from the mean (i.e., 95% confidence interval). All data used to generate these limits will undergo a Dixon Outlier test to reject outlier data points. The use of hardcoded limits nonstatistically derived from current laboratory generated data (e.g., CLP limits) shall be limited to programs which specifically permit their application. Control limits shall be updated annually at a minimum and at a maximum interval of once every 20 data points generated. At a minimum, tabulated control limits shall be available and followed by all analysts performing the associated test. However, control charts are the preferred mechanism for monitoring quality control measurements on a real

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time basis. Where interlaboratory expected ranges have been determined, Pace's goal is for their control limits to fall within these multi-laboratory expected ranges for that method.

Suspicious/Out-of-Control Events

Plotting and connecting successive data points on control charts enables the laboratory to detect many types of suspicious and out-of-control situations. These events can be caught by monitoring the following: outliers (suspicious and out-of-control), runs (suspicious), trends (suspicious), and periodicity (suspicious).

Excursions

There are two types of excursions: any particular point that falls outside the control limits or any point that falls outside the warning limits. A point that falls outside the control limits is classified as an out-of-control event; a point that falls outside the warning limits is classified as a suspicious event.

Runs

A run is defined as a series of points that line up on one side of the central line (the mean). Any run that has a length of seven points is indicative of a potential abnormality in the process, a suspicious event. A run can suggest several potential problems such as a leak in the system, elevated contamination, or incorrect dilutions of standards.

Trends

A trend is defined as a series of points that are marked by an unbroken rise or fall. Any trend with a length of five points (may vary up to seven points) is classified as a suspicious event. A trend may indicate a change in instrument sensitivity due to a dirty source or injection port or standard degradation, to name a few.

Periodicity

Periodicity is a term used to describe a recurring pattern of change over equal intervals. This occurrence may be of any length or amplitude; thus, careful observation of the control chart is necessary.

11.3 QC BATCH DEFINITION

Organics: The laboratory will perform:

- One spiked sample analysis (matrix spike), and

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-
- **One duplicate spiked sample analysis (matrix spike duplicate)**

for each group of samples of a similar matrix (for water or for soil samples) and concentration level (when low vs. medium level analyses are available) for:

- every 20 samples, or
- each 14 calendar day period during which field samples were received (said period beginning with the receipt of the first sample by the laboratory),

whichever is more frequent.

- **One spiked laboratory control sample (LCS) must be processed each time a group of 20 samples or less are extracted, prepared, or directly analyzed (Note: an organic LCS is processed at the same frequency as a laboratory reagent blank (method or prep blank).**

Inorganics: The laboratory will perform:

- **One spiked sample analysis (matrix spike), and**
- **One straight sample duplicate analysis (duplicate)**

for each group of samples of a similar matrix (for water or for soil samples) and concentration level (when low vs. medium level analyses are available) for:

- every 20 samples, OR
- each 14 calendar day period during which field samples were received (said period beginning with the receipt of the first sample by the laboratory),

whichever is more frequent.

- **One spiked laboratory control sample (LCS) must be processed each time a group of 20 samples or less are digested, extracted, prepared, or directly analyzed (Note: an inorganic LCS is processed at the same frequency as a laboratory reagent blank (method or prep blank).**

The frequencies listed above for matrix spiking applications are to be followed regardless of whether or not clients have committed to "paying for QC." Laboratory operations must make a conscious effort to periodically request or collect as part of a field sampling event (i.e., sampling performed by Pace personnel) sufficient quantities of samples for those analyses (e.g., method 418.1 TPH) that are routinely analyzed under QC batches that do not contain matrix spike applications because "insufficient sample volume was received." Samples selected for QC which contain limited volume or quantity should be evaluated based upon the type of analysis to be performed to

establish whether modifications can be made to allow for using smaller initial sample size than normally applied. For example, most organic extraction procedures require that a 1 liter sample size be analyzed for aqueous samples. However, often only a single 1 liter sample bottle remains following the extraction of the sample thus leaving insufficient volume to perform both an MS and MSD on the sample because each normally require a full liter of sample. In most cases, the usability of the QC data for assessing the accuracy and precision of the analysis is not adversely impacted if the remaining liter of sample is split into two 500 mL aliquots, spiked and carried through the procedure. While following this modification would result in a two fold increase in MDLs, since the spiked compounds are present at concentrations which are close to the midpoint of the calibration curve, the elevated MDLs will have no effect on determining recovery and RPD values. In this example, a further method modification of concentrating the final volume of the MS/MSD extracts to 0.5 mL (versus the normal 1.0 mL final volume) would provide the 1000 fold concentration requirement of the method. Any options, such as the example given above, contemplated for use to overcome limited sample size when applying QC applications must be discussed with clients prior to their implementation. Finally, for methods in which no similar technical justification can be made for decreasing the initial sample size or changing the analysis process, when none of the associated samples in a QC batch contain sufficient sample volume or quantity to permit matrix spiking to be performed, laboratory control samples should be analyzed in duplicate (LCS/LCSD) to afford assessment of both the accuracy and precision of the test.

11.4 UTILIZATION OF QUALITY CONTROL DATA

The purpose for preparing and analyzing quality control samples is to demonstrate, through the known entities, how accurate and precise the investigative sample data are. Table 11-1 summarizes the quality control assessment criteria by matrix for the most commonly used methods by Pace. Different criteria may be dictated by different methods or by project QA plans.

11.5 SAMPLING QUALITY CONTROL

Quality control is an integral part of sample collection as well as laboratory operations. Sample collection protocols must include checks to ensure that the sample collected is representative of the site from which it was collected and free from collection-related contamination or biases. Although different laboratory procedures will be used to analyze for the various parameters of interest, certain general QC procedures are applicable to most sampling methods. QC procedures frequently applied in the field are described below. The analysis types and frequency of collection for each of these field QC samples are detailed in each project's sampling and analysis plan (SAP).

- 11.5.1 Field Blanks - Field blanks are QC samples consisting of blank water that are prepared in the field. This type of QC sample serves to check for potential contamination that may be present in the environment where field samples are collected.

- 11.5.2 Trip Blanks - Trip blanks are similar to field blanks except that they are prepared in the laboratory before the sampling event. These blank samples accompany the other sample containers to the field and then accompany the collected samples back to the lab. Trip blanks serve to check for potential contamination that samples and sample containers may be exposed to during transportation to and from the field.
- 11.5.3 Equipment Rinsate Blanks - These field QC samples consist of rinsates of the equipment used to collect field samples using blank water provided by the laboratory. Equipment rinsate blanks serve to check the adequacy of equipment cleaning between successive sample collections. Inadequate cleaning of sample collection equipment after the collection of a sample could result in the contamination of the next sample collected.
- 11.5.4 Matrix Spike/Matrix Spike Duplicate Samples - At a minimum frequency of one set per 20 samples, split sample volumes are collected to be used for matrix spike (MS) and matrix spike duplicate (MSD) analyses by the laboratory.

11.6 LABORATORY QUALITY CONTROL

In addition to the sampling-related QC procedures described above, additional QC procedures are performed in the laboratory as part of routine analytical protocol. These procedures are described below for representative analytical methods.

11.6.1 GC Methods

Analytical quality control procedures for GC analyses are described in Method 8000A of SW-846, 3rd Edition, Final Update 1 and 2, and the EPA CLP Organic SOW. They include the following:

- Initial demonstration of proficiency
- Retention time window determination
- Surrogate spiked sample analysis
- Method blank analysis
- Matrix spike/matrix spike duplicate analysis
- Laboratory control sample analysis

The application of each of these analyses is described below.

Initial Demonstration of Proficiency (SW-846) - Before sample analysis can begin, the laboratory must perform a one-time demonstration of the ability to generate data with acceptable accuracy and precision. This is accomplished by analyzing four aliquots of a QC check sample by the same procedure used to analyze samples. The calculated average recovery and standard deviation

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for each analyte of interest are compared to acceptance criteria provided in the specific SW-846 method. If the calculated accuracy and precision data are within acceptance limits, analysis of samples may proceed. If not, remedial action must be taken to improve system performance and the proficiency test must be repeated.

Retention Time Window Determination - Retention time (RT) windows are calculated for each target analyte peak(s) and for each GC column used for sample analysis. To establish RT windows, the laboratory measures the RTs of each analyte peak (or of each selected peak for multi-component analytes) from three analyses of the continuing calibration standard over a 72-hour period. The RT window is determined as ± 3 times the standard deviation of the three measured RTs. Daily RT windows are established for each analyte peak using the RT in the daily calibration verification standard as the centerpoint of the window determined above. In successive continuing calibration standards, the RT of each analyte peak must fall within the prescribed RT window for the analysis sequence to continue. RT windows must be recalculated whenever a new GC column is installed.

Surrogate Spiked Sample Analysis - Surrogates are compounds that have similar chemical properties to analytes of interest except that they are not expected to occur naturally in environmental samples. The use of surrogate compounds may be project dependent and limited by the ability to select a suitable surrogate for a particular analytical method. Representative surrogate compounds and surrogate recovery acceptance limits for GC methods are given in Section 5, Tables 5.1 to 5.4. For these methods, corrective action must be taken if the surrogate spike recoveries in any analysis fall outside the prescribed acceptance limits. Corrective actions include:

- Checking for errors in the calculation or preparation of the surrogate or standard solutions.
- Checking instrument performance.
- Recalculating the data and/or reanalyzing the sample or extract if any of the above checks reveal a problem.
- Re-extracting and reanalyzing the sample if none of the above are determined to be the problem.

Method Blank Analysis - For analysis by purge-and-trap methods, a method blank must be analyzed each day of analysis. For extraction methods (including methanol extraction of volatiles for purge-and-trap analysis), at least one method blank must be extracted and analyzed for each batch or sub-batch of samples extracted to demonstrate that both the extraction and analytical systems are free from contamination. Blank samples are carried through all stages of sample preparation and analysis. Lack of contamination is demonstrated if no target analytes are present at concentrations at or

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above their reporting limits (or contract required quantitation limits (CRQLs) for CLP).

Holding Blanks - Holding or refrigerator blanks are prepared in the laboratory and stored in the refrigerators where VOA samples reside. Holding blanks are analyzed each week and are used to monitor the potential of laboratory contamination.

Matrix Spike/Matrix Spike Duplicate Analyses - At a minimum frequency of one set per batch of up to 20 samples of similar matrix, replicate aliquots of one of the samples are spiked with a mix of target analytes and the resulting matrix spike (MS) and matrix spike duplicate (MSD) samples are analyzed to evaluate the percent recovery of the spiked compounds. Representative limits for percent recovery are shown in Section 5, Tables 5.1 to 5.4. Recovery data falling outside the acceptance limits may indicate a problem in sample preparation or in the analytical system, or may be due to sample matrix interference. Analysis of laboratory control samples (LCSs) in conjunction with MS/MSD samples aids in determining whether or not the problem is sample matrix related. Acceptable recoveries of LCS spike analytes indicate that the analytical system is in control and that problems with associated recoveries in the MS and MSD samples are likely due to sample matrix interference.

Laboratory Control Sample Analysis - The laboratory control sample (LCS) consists of a subset of target analytes of interest (typically the same as in the MS and MSD samples) spiked at concentrations in the mid-calibration range. The LCS is prepared along with the samples for analysis and is used to verify that the analytical system is in control. LCS recovery data are plotted on blank spike control charts to monitor the analytical system for trends or events that indicate a change in method/instrument performance.

11.6.2 GC/MS Methods

Analytical quality control procedures for GC/MS analyses are described in methods 8000A, 8240B, and 8270B in SW-846 or in the EPA CLP Organic SOW. They include the following:

- Initial demonstration of proficiency
- Mass spectrometer sensitivity check
- Daily GC/MS performance test
- Surrogate spiked sample analysis
- Method blank analysis
- Matrix spike/matrix spike duplicate analysis
- Laboratory control sample analysis

The application of each of these analyses is described below.

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Initial Demonstration of Proficiency (SW-846) - Before sample analysis can begin, the laboratory must perform a one-time demonstration of the ability to generate data with acceptable accuracy and precision. This is accomplished by analyzing four aliquots of a QC check sample by the same procedure used to analyze samples. The calculated average recovery and standard deviation for each analyte of interest are compared to acceptance criteria provided in the specific SW-846 method. If the calculated accuracy and precision data are within acceptance limits, analysis of samples may proceed. If not, remedial action must be taken to improve system performance and the proficiency test must be repeated.

Mass Spectrometer Sensitivity Check - If the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two compared to the daily calibration verification standard, the mass spectrometer must be inspected for malfunctions and corrective action taken. Samples analyzed while the system was malfunctioning must be reanalyzed.

Daily GC/MS Performance Tests - Each day that analyses are performed, the GC/MS system must be checked using bromofluorobenzene (BFB) for volatiles analysis or decafluorotriphenylphosphine (DFTPP) for semivolatiles analysis. The acceptance criteria presented in Section 9 must be met prior to performing any sample analyses. If all criteria are not met, the instrument must be retuned and the test repeated until all criteria are met.

Surrogate Spiked Sample Analysis - All samples are spiked with surrogate standards as described in the specific methods in SW-846 and the CLP SOW. The surrogate compounds and representative surrogate recovery acceptance limits for GC/MS methods are shown in Tables 5.5 and 5.6. If the surrogate spike recovery in any analytical run is not within limits, the following steps must be taken:

- Check for errors in the calculation or preparation of the surrogate or standard solutions.
- Check instrument performance.
- Recalculate the data and/or reanalyze the sample or extract if any of the above checks reveal a problem.
- Re-extract and reanalyze the sample if none of the above are determined to be the problem.

Method Blank Analysis - For volatiles analysis by GC/MS, a method blank must be analyzed within each 12 hour run sequence. For semivolatiles analysis, at least one method blank must be prepared and analyzed for each batch or sub-batch of samples extracted, to demonstrate that both the extraction and analytical systems are free from contamination. Blank samples are carried through all stages of sample preparation and analysis.

Lack of contamination is demonstrated if no target analytes (with the exception of common laboratory solvents) are present at concentrations at or above their reporting limits (CRQLs for CLP). For volatile analyses, common laboratory contaminants, such as methylene chloride, acetone, 2-butanone, and toluene, must not exceed five times the CRQL for CLP or five times the reporting limit for SW-846. For semivolatile analyses, the concentrations of the most commonly encountered laboratory contaminants, phthalate esters, must not exceed five times the reporting limit or CRQL.

Holding Blanks - Holding or refrigerator blanks are prepared in the laboratory and stored in the refrigerators where VOA samples reside. Holding blanks are analyzed each week and are used to monitor the potential of laboratory contamination.

Matrix Spike/Matrix Spike Duplicate Analysis - A minimum of one set of matrix spike (MS) and matrix spike duplicate (MSD) samples is prepared for each analytical batch of up to 20 samples of similar matrix. Acceptance limits for percent recovery are shown in Section 5, Tables 5.5 and 5.6. Recovery data falling outside the prescribed acceptance limits may indicate a problem in sample preparation or the analytical system, or may be due to sample matrix interference. Analysis of laboratory control samples (LCSs) in conjunction with MS/MSD samples aids in determining whether or not the problem is sample matrix related. Acceptable recoveries of LCS spike compounds indicate that the analytical system is in control and that problems with associated recoveries in the MS and MSD samples are likely due to sample matrix interference.

Laboratory Control Sample Analysis - The laboratory control sample (LCS) consists of a subset of the target analytes (typically the same as in the MS and MSD samples) spiked at concentrations in the mid-calibration range. The LCS is prepared along with the samples for analysis and is used to verify that the analytical system is in control. LCS recovery data are plotted on blank spike control charts to monitor the analytical system for trends or events that indicate a change in method/instrument performance.

11.6.3 Metals Analysis

The quality control procedures applied to metals analysis by ICPS are described in SW-846 Method 6010A and in the CLP Inorganic SOW. Quality control procedures for atomic absorption analyses are described in SW-846 Method 7000 series and the CLP Inorganic SOW. These procedures include the analysis of:

- An initial calibration blank
- A continuing calibration blank
- A preparation blank

- MS/MSD/duplicate samples
- An instrument check standard
- A laboratory control sample
- An interference check standard

Each of these analyses is described below.

Initial Calibration Blank Analysis - Following the analysis of the initial calibration verification standard (ICV) and prior to the analysis of samples, an initial calibration blank (ICB) is analyzed to demonstrate that the analytical system is free from contamination. This blank analysis must be free from all elements of interest at or above the reporting limits (CRDLs for analysis by CLP protocol), or the instrument must be recalibrated before sample analysis may begin.

Continuing Calibration Blank Analysis - Following the analysis of each continuing calibration standard (CCV) in an analytical sequence, a continuing calibration blank (CCB) is analyzed to demonstrate that the analytical system is free from contamination throughout the course of that sequence. This blank analysis must be free from all elements of interest at or above the reporting limits (CRDLs for analysis by CLP protocol), or sample analysis must be discontinued and the previous 10 samples must be reanalyzed under a new calibration.

Preparation Blank Analysis - A preparation blank, containing all of the reagents and volumes used in the processing of samples and carried through the complete preparation and analysis procedure, is analyzed at a minimum frequency of one per sample batch or sub-batch. The preparation blank is analyzed to demonstrate that the sample preparation procedure is free from contamination. This blank must be free of all elements of interest at or above the reporting limits (CRDLs for analysis by CLP protocol), or the entire sub-batch of samples must be reprepared and reanalyzed.

Laboratory Control Sample Analysis - Control samples may be obtained from commercial vendors or prepared from suitable reference materials, but must be prepared independently from the calibration standards. The LCS is prepared along with the samples for analysis and is used to verify that the analytical system is in control. LCS recovery data are plotted on blank spike control charts to monitor the analytical system for trends or events that indicate a change in method/instrument performance.

Matrix Spike/Matrix Spike Duplicate or Sample Duplicate Analysis - For each analytical batch and sample matrix type, a matrix spike sample and either a matrix spike duplicate sample or duplicate matrix sample are analyzed at a minimum frequency of one set per batch. Matrix spike recoveries should fall within 75-125% (or within lab derived limits if applicable) (or within lab

derived limits of applicability of the spike concentration for water and soil matrices. If the spike is not recovered within the specified limits, the data should be flagged as suspect due to sample matrix effects. Depending upon the project, provisions should be established to determine when the method of standard addition (MSA) should be employed to compensate for matrix effects.

Interference Check Standard Analysis (ICPS) - The interference check standard is analyzed at the beginning and end of the analytical sequence and at intervals during the sequence. This standard contains the analytes of interest at minimal concentrations and by known concentration of interfering elements. If results exceed $\pm 20\%$ of the expected value, the instrument must be recalibrated before sample analysis may proceed.

11.6.4 Cyanide Analysis

Inorganic cyanide is determined colorimetrically by method 9010A, method 9012 or the CLP Inorganic SOW. Quality control procedures for this analysis include the analysis of:

- A preparation blank
- A laboratory control sample
- A matrix spike sample
- A matrix spike duplicate or sample duplicate

Each of these analyses is described below.

Preparation Blank Analysis - A preparation blank, containing all of the reagents and volumes used in the processing of samples and carried through the complete preparation and analysis procedure, is analyzed at a minimum frequency of one per sample batch or sub-batch. The preparation blank is analyzed to demonstrate that the sample preparation procedure is free from contamination. This blank must be free of cyanide at or above the reporting limit (CRDL for analysis by CLP protocol), or the entire sub-batch of samples must be reprepared and reanalyzed.

Laboratory Control Sample Analysis - Control samples may be obtained from commercial vendors or prepared from suitable reference materials, but must be prepared independently from the calibration standards. The LCS is prepared along with the samples for analysis and is used to verify that the sample preparation and analysis steps are in control. LCS recovery data are plotted on blank spike control charts to monitor the analytical system for trends or events that indicate a change in method/instrument performance.

Matrix Spike/Matrix Spike Duplicate or Sample Duplicate Analysis - For each analytical batch and sample matrix type, a matrix spike sample and either a matrix spike duplicate sample or duplicate matrix sample are analyzed at a

minimum frequency of one set per batch. Matrix spike recoveries should fall within 75-125% (or within lab derived limits if applicable) (or within lab derived limits if applicable) of the spike concentration for water and soil matrices. If the spike is not recovered within the specified limits, the data should be flagged as suspect due to sample matrix effects.

11.6.5 Anion Analysis

Anions, including chloride, nitrite, nitrate, o-phosphate, bromide, and sulfate, may be analyzed by ion chromatography as described by Method 300.0. Quality control procedures for this method include the analysis of:

- A preparation blank
- A laboratory control sample
- A matrix spike sample
- A matrix spike duplicate or sample duplicate

Each of these analyses is described below.

Preparation Blank Analysis - A preparation blank, containing all of the reagents and volumes used in the processing of samples and carried through the complete preparation and analysis procedure, is analyzed at a minimum frequency of one per sample batch or sub-batch. The preparation blank is analyzed to demonstrate that the sample preparation procedure is free from contamination. This blank must be free of the anions being measured at or above the reporting limits, or the entire sub-batch of samples must be reprepared and reanalyzed.

Laboratory Control Sample Analysis - Control samples may be obtained from a commercial vendor or prepared from suitable reference materials, but must be prepared independently from the calibration standards. The LCS is prepared along with the samples for analysis and is used to verify that the sample preparation and analysis steps are in control. LCS recovery data are plotted on blank spike control charts to monitor the analytical system for trends or events that indicate a change in method/instrument performance.

Matrix Spike/Matrix Spike Duplicate or Sample Duplicate Analysis - For each analytical batch and sample matrix type, a matrix spike sample and either a matrix spike duplicate sample or duplicate matrix sample are analyzed at a minimum frequency of one set per batch. Matrix spike recoveries should fall within 75-125% (or within lab derived limits if applicable) (or within lab derived limits if applicable) of the spike concentration for water and soil matrices. If the spike is not recovered within the specified limits, the data should be flagged as suspect due to sample matrix effects. Duplicate analyses should agree within 20% RPD.

11.6.6 Fluoride Analysis

Fluoride is determined potentiometrically by method 340.2. Quality control procedures include the analysis of:

- A preparation blank
- A laboratory control sample
- A matrix spike sample
- A matrix spike duplicate or sample duplicate

Each of these analyses is described below.

Preparation Blank Analysis - A preparation blank, containing all of the reagents and volumes used in the processing of samples and carried through the complete preparation and analysis procedure, is analyzed at a minimum frequency of one per sample batch or sub-batch. The preparation blank is analyzed to demonstrate that the sample preparation procedure is free from contamination. This blank must be free of fluoride at or above the reporting limit, or the entire sub-batch of samples must be reprepared and reanalyzed.

Laboratory Control Sample Analysis - Control samples may be obtained from commercial vendors or prepared from suitable reference materials, but must be prepared independently from the calibration standards. The LCS is prepared along with the samples for analysis and is used to verify that the sample preparation and analysis steps are in control. LCS recovery data are plotted on blank spike control charts to monitor the analytical system for trends or events that indicate a change in method/instrument performance.

Matrix Spike/Matrix Spike Duplicate or Sample Duplicate Analysis - For each analytical batch and sample matrix type, a matrix spike sample and either a matrix spike duplicate sample or duplicate matrix sample are analyzed at a minimum frequency of one set per batch. Matrix spike recoveries should fall within 75-125% (or within lab derived limits if applicable) of the spike concentration for water and soil matrices. If the spike is not recovered within the specified limits, the data are flagged as suspect due to sample matrix effects. Duplicate analyses should agree within 20% RPD.

11.6.7 Total Organic Carbon Analysis

Combustion of organic carbon and detection of carbon dioxide by IR spectrometry is performed by method 9060 or 415.1. Quality control procedures include the following analyses:

- A preparation blank
- A laboratory control sample
- A matrix spike sample

- A matrix spike duplicate or sample duplicate

Each of these analyses is described below.

Preparation Blank Analysis - A preparation blank, containing all of the reagents and volumes used in the processing of samples and carried through the complete preparation and analysis procedure, is analyzed at a minimum frequency of one per sample batch or sub-batch. The preparation blank is analyzed to demonstrate that the sample preparation procedure is free from contamination. This blank must be free of organic carbon at or above the reporting limit, or the entire sub-batch of samples must be reprepared and reanalyzed.

Laboratory Control Sample Analysis - Control samples may be obtained from a commercial vendor or prepared from suitable reference materials, but must be prepared independently from the calibration standards. The LCS is prepared along with the samples for analysis and is used to verify that the sample preparation and analysis steps are in control. LCS recovery data are plotted on blank spike control charts to monitor the analytical system for trends or events that indicate a change in method/instrument performance.

Matrix Spike/Matrix Spike Duplicate or Sample Duplicate Analysis - For each analytical batch and sample matrix type, a matrix spike sample and either a matrix spike duplicate sample or duplicate matrix sample are analyzed at a minimum frequency of one set per batch. Matrix spike recoveries should fall within 75-125% (or within lab derived limits if applicable) of the spike concentration for water and soil matrices. If the spike is not recovered within the specified limits, the data are flagged as suspect due to sample matrix effects. Duplicate analyses should agree within 20% RPD.

11.6.8 Oil and Grease Analysis

Total oil and grease is determined gravimetrically by methods 9070/9071A and 413.1 and spectrophotometrically (IR) by method 413.2. Quality control procedures include the following analyses:

- A preparation blank
- A laboratory control sample
- A matrix spike sample
- A matrix spike duplicate sample

Each of these analyses is described below.

Preparation Blank Analysis - A preparation blank, containing all of the reagents and volumes used in the processing of samples and carried through the complete preparation and analysis procedure, is analyzed at a minimum

frequency of one per sample batch or sub-batch. The preparation blank is analyzed to demonstrate that the sample preparation procedure is free from contamination. This blank must be free of oil and grease at or above the reporting limit, or the entire sub-batch of samples must be reprepared and reanalyzed.

Laboratory Control Sample Analysis - Control samples may be obtained from a commercial vendor or prepared from suitable reference materials, but must be prepared independently from the calibration standards. The LCS is prepared along with the samples for analysis and is used to verify that the sample preparation and analysis steps are in control. LCS recovery data are plotted on blank spike control charts to monitor the analytical system for trends or events that indicate a change in method/instrument performance.

Matrix Spike/Matrix Spike Duplicate Analysis - For each analytical batch and sample matrix type, a matrix spike sample and a matrix spike duplicate sample are analyzed at a minimum frequency of one set per batch. Matrix spike recoveries should fall within 75-125% (or within lab derived limits if applicable) of the spike concentration for water and soil matrices. If the spike is not recovered within the specified limits, the data are flagged as suspect due to sample matrix effects. Duplicate analyses should agree within 20% RPD.

11.6.9 Total Recoverable Petroleum Hydrocarbons (TRPH) Analysis

TRPH is determined spectrophotometrically (IR) by method 418.1. Quality control procedures include the following analyses:

- A preparation blank
- A laboratory control sample
- A matrix spike sample
- A matrix spike duplicate sample

Each of these analyses is described below.

Preparation Blank Analysis - A preparation blank, containing all of the reagents and volumes used in the processing of samples and carried through the complete preparation and analysis procedure, is analyzed at a minimum frequency of one per sample batch or sub-batch. The preparation blank is analyzed to demonstrate that the sample preparation procedure is free from contamination. This blank must be free of TRPH at or above the reporting limit, or the entire sub-batch of samples must be reprepared and reanalyzed.

Laboratory Control Sample Analysis - Control samples may be obtained from commercial vendors or prepared from suitable reference materials, but must be prepared independently from the calibration standards. The LCS is

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prepared along with the samples for analysis and is used to verify that the sample preparation and analysis steps are in control. LCS recovery data are plotted on blank spike control charts to monitor the analytical system for trends or events that indicate a change in method/instrument performance.

Matrix Spike/Matrix Spike Duplicate Analysis - For each analytical batch and sample matrix type, a matrix spike sample and a matrix spike duplicate sample are analyzed at a minimum frequency of one set per batch. Matrix spike recoveries should fall within 75-125% (or within lab derived limits if applicable) of the spike concentration for water and soil matrices. If the spike is not recovered within the specified limits, the data are flagged as suspect due to sample matrix effects. Duplicate analyses should agree within 20% RPD.

11.6.10 California Assessment Manual Waste Extraction Test (CAM WET)/Extraction Procedure Toxicity Test Method (EP-Tox)/Toxicity Characteristic Leaching Procedure (TCLP)

Waste extraction is performed according to the procedure described in the appropriate waste extraction regulation. Quality control procedures include the following analyses:

- A preparation blank
- A duplicate sample

Each of these analyses is described below.

Preparation Blank Analysis - A minimum of one method blank per sample batch of up to 20 samples is analyzed to demonstrate the absence of contamination above reporting limits.

Duplicate Sample Extraction - A duplicate sample extraction is performed with each batch of up to 20 samples. Results of analyses of the duplicate extracts are used to estimate overall measurement variability and generally should agree within 20% RPD.

11.7 STANDARDS

The term standard shall apply to any analyte solution of known concentration which is traceable to a certified reference material. This includes calibration standards, spiking solutions and laboratory control samples.

Upon receipt, all purchased standard reference materials (neat and stock solutions) are recorded into section-specific standards logbooks. Standard logbook entries include Pace unique ID, name of the neat compound or solution, manufacturer, manufacturer's lot number, certified purity, and expiration date. Subsequent

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preparations of stock, intermediate, and working solutions are also documented in the standards logbooks. These entries must include all discrete measurements made during preparation, sources of materials, solvent(s) and a Pace ID number.

The standard vial should have a reference label affixed containing the following information (if sPace permits):

- Standard ID number
- Name of standard
- Preparation date
- Preparer's initials
- Solvent
- Preservation, if applicable
- Expiration date

The Standard Operating Procedure (#MN-P-004-B) "Standards Traceability in Laboratory and Field" contains further instructions for assigning unique ID numbers, shelf life of standards, and good laboratory practices.

All primary reference standard and standard solutions are purchased from reliable commercial sources. Standards traceable to NIST are preferred; however, ASTM or equivalent specifications are acceptable. Certification records of all standards received are retained.

Second source reference standards and standard solutions are purchased from a different supplier than the primary standard or from a different manufactured lot from the same vendor. If a second supplier is not available, the second source standard can be prepared from a different lot number of the same composition from the same supplier.

Newly prepared standard solutions (surrogate, internal, calibration, spiking) are verified against another known standard prepared from another source prior to utilization. The verification data is maintained on file in the respective area. In place of performing in-house standard verification, laboratories can purchase verified second source standards from a vendor which supplies a data package demonstrating verification.

11.8 SOLVENT LOT/ACID LOT VERIFICATION

All laboratory extraction solvents utilized are at least Pesticide Grade or better. Prior to accepting a solvent lot from the supplier, a quantity of the solvent is analyzed either by Pace or by the primary vendor under a National Qualified Materials (NQM) program to access the purity. The NQM verification program is administered by the Pace Corporate office for use by all laboratory locations. If the lot is determined to meet purity standards/requirements, the lot is sequestered for Pace laboratories. The quality of organic extraction solvents is constantly monitored through the analysis of method blanks.

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Acid lots are verified for purity prior to utilization for digestion of samples. If the acid lot is determined to meet purity criteria, the acid may be used for sample preparation digestion.

Figure 11.1

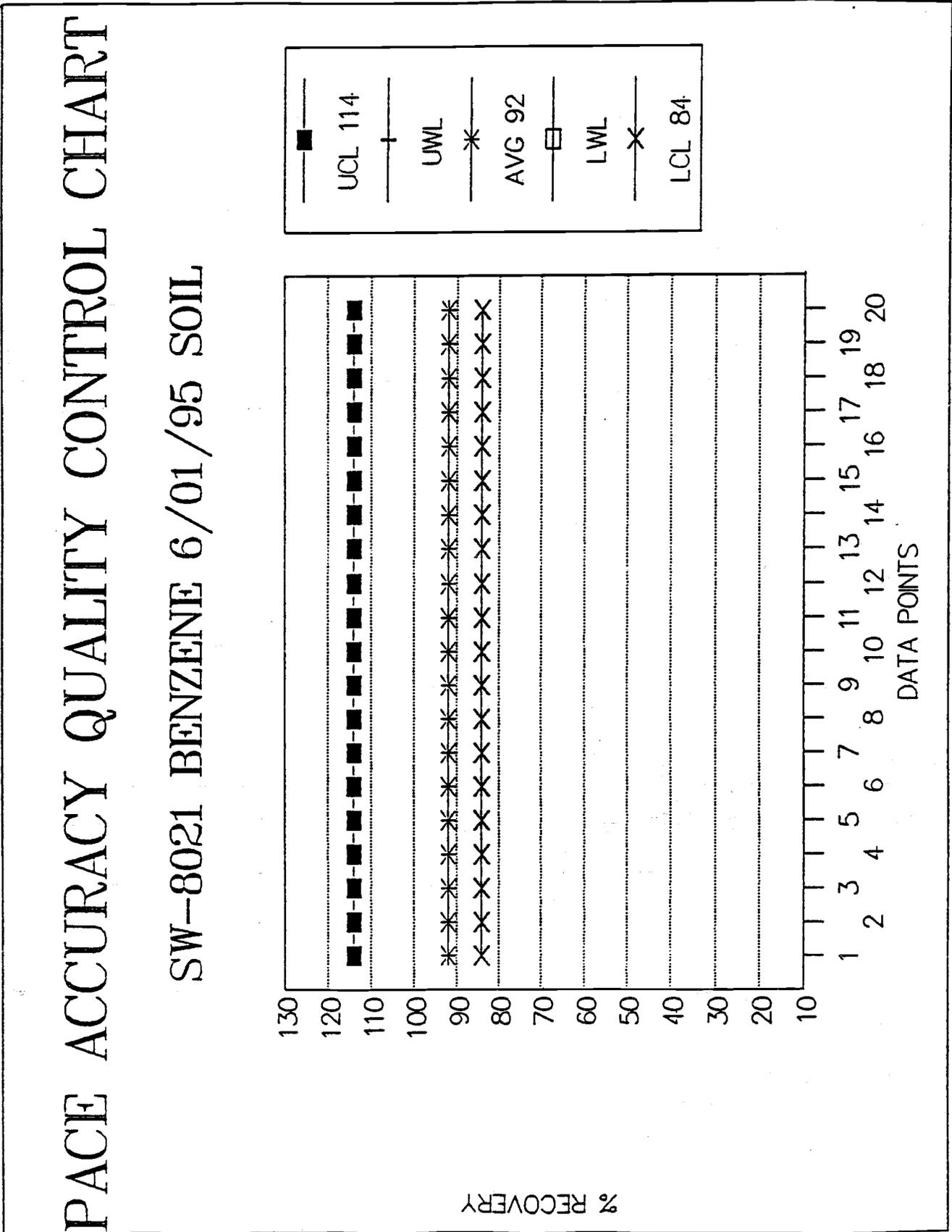


Figure 11.1 (cont.)

WATER SPIKE LIMITS-ACCURACY
BENZENE

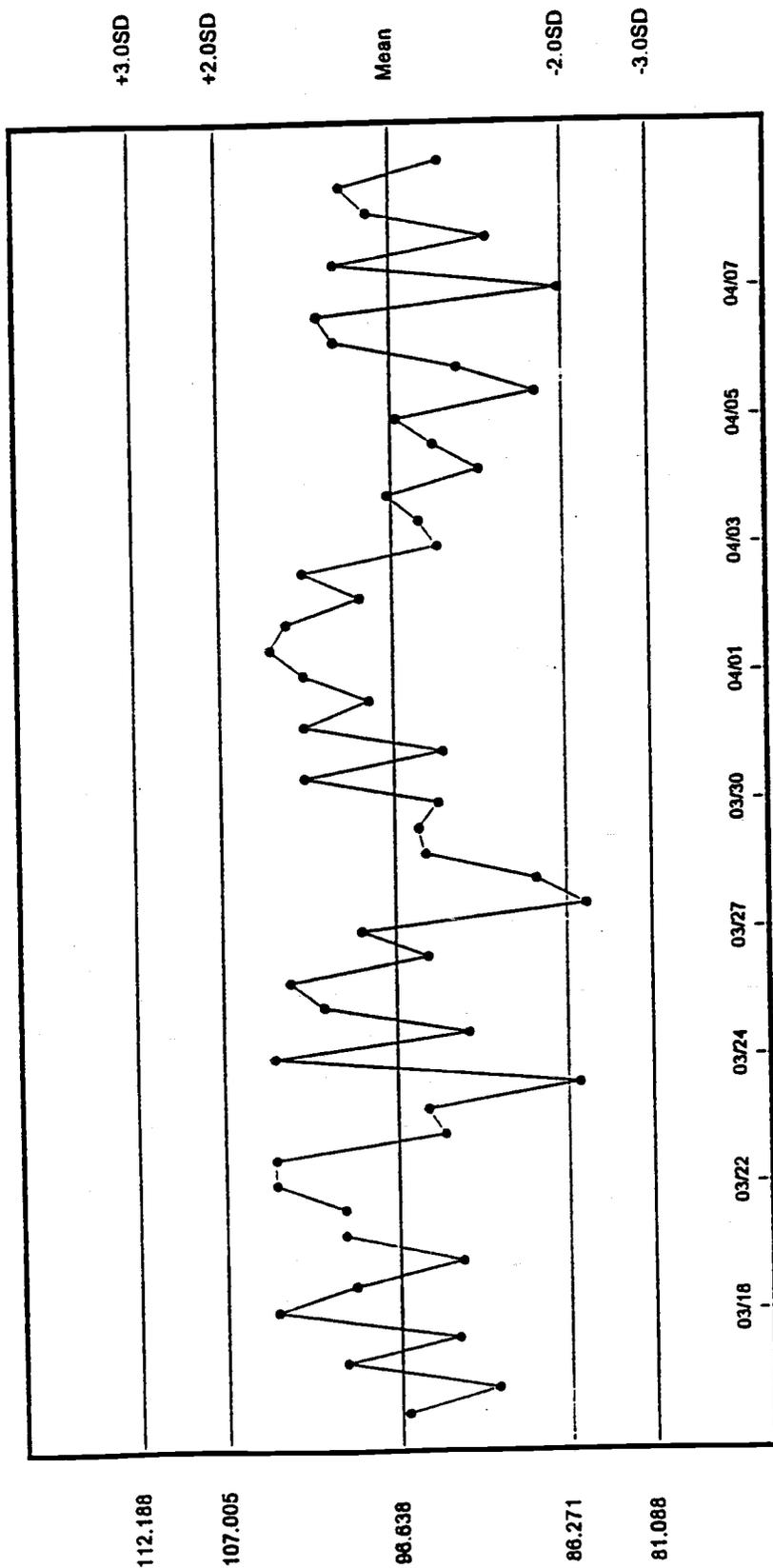


Figure 11.2

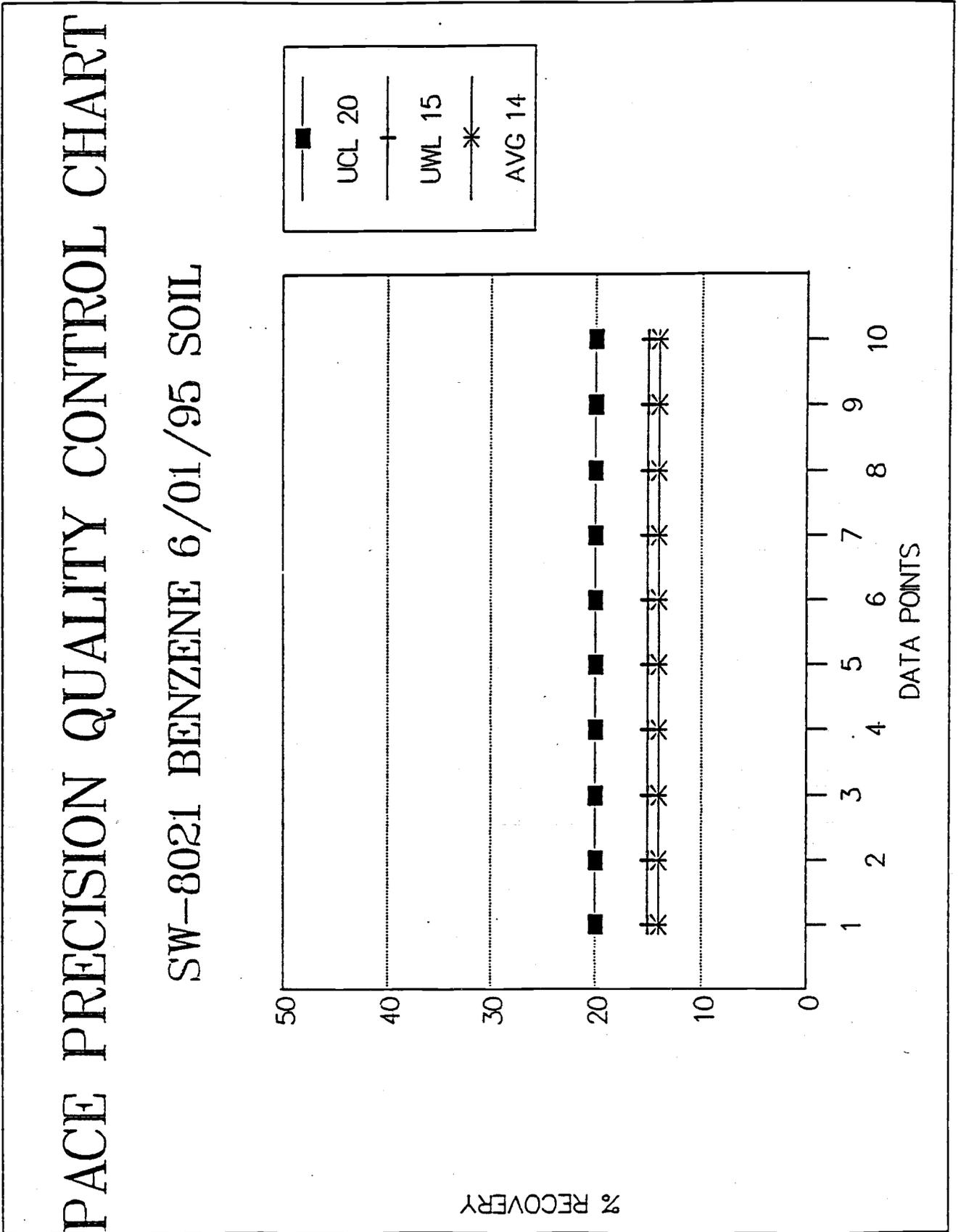
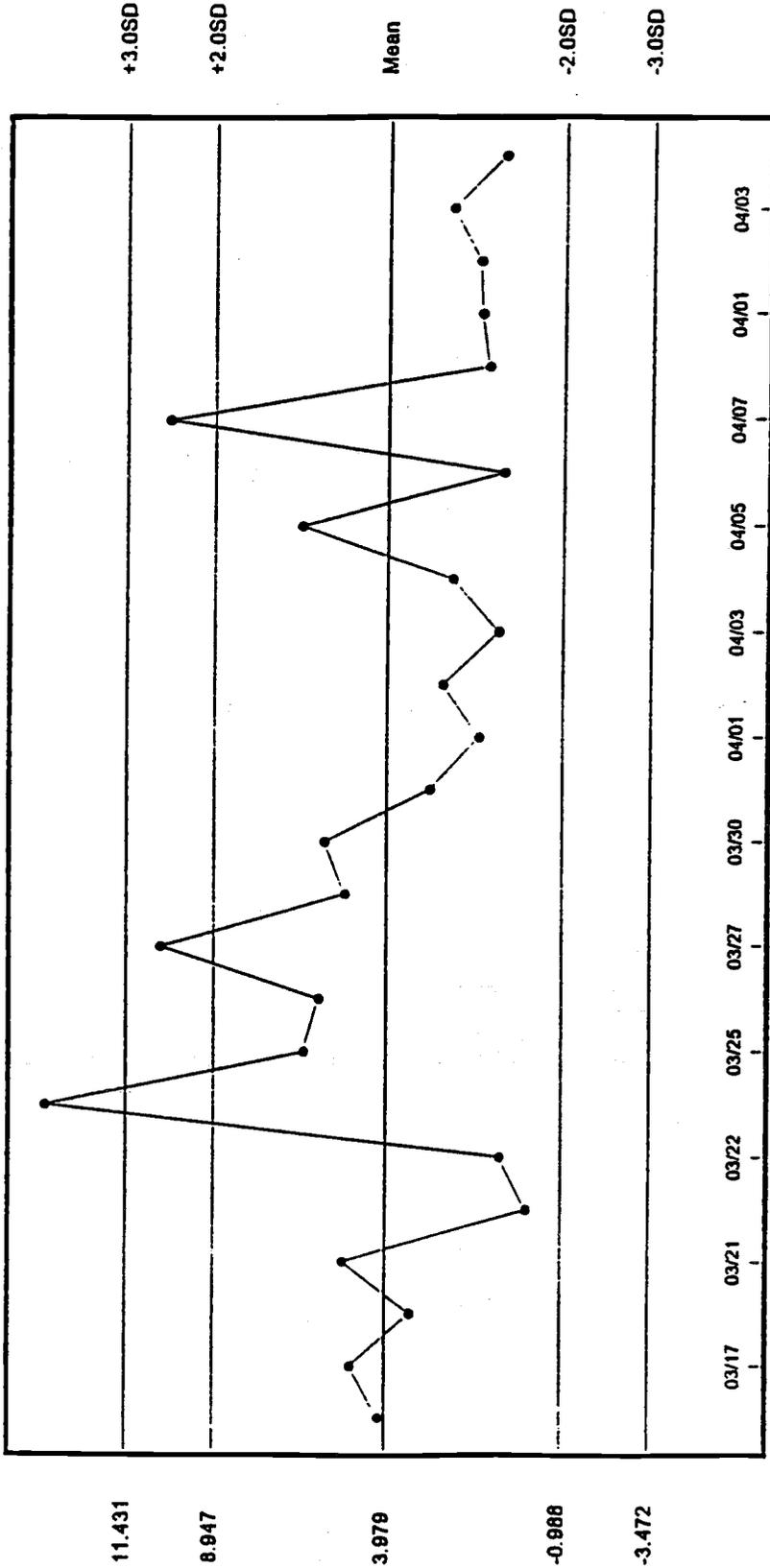


Figure 11.2 (cont.)

WATER SPIKE LIMITS-PRECISION
BENZENE



n= 25 Mean= 3.979 SD= 2.464 CV= 62.42% Min= 0.000 Max= 13.724

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
8010B	Purgeable Halocarbons	Initial 5-point calibration	As needed - Refer to method	$r \geq 0.995$	Repeat calibration
		Continuing calibration	Daily & every 10 samples	$\%D \leq 15\%$ (except gases and 2-CEVE)	1. Repeat test 2. Recalibrate
		Method blank	1 per batch & sub-batch	All analytes < reporting limit	Clean system & reanalyze sub-batch
		Surrogate spikes	Every analysis	Bromochloromethane (See Table 5.1)	Reanalyze sample
		Matrix spike & Matrix spike duplicate	1 set per batch	Method 8010B limits (See Table 5.1)	Narrate in report
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.1)	Correct problem & reanalyze sub-batch
		Second column confirmation	100% for positive results \geq reporting limit	Qualitative confirmation	N/A
		Initial 5-point calibration	As needed Refer to method	$r \geq 0.995$	Repeat calibration
8020A	Purgeable Aromatics	Continuing calibration	Daily & every 10 samples	$\%D \leq 15\%$	1. Repeat test 2. Recalibrate
		Method blank	1 per batch & sub-batch	All analytes < reporting limit	Clean system & reanalyze sub-batch

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
		Surrogate spikes	Every analysis	1,4-Bromofluorobenzene (See Table 5.1)	Reanalyze sample
		Matrix spike & Matrix spike duplicate	1 set per batch	Method 8020A limits (See Table 5.1)	Narrate in report
		Laboratory control sample (LCS)	1 per batch and	Statistical limits (See Table 5.1)	Correct problem and reanalyze sub-batch
		Second column	100% for positive results confirmation	Qualitative confirmation ≥ reporting limit	N/A
8080A CLP SOW	Organochlorine Pesticides and PCBs	Initial calibration: 3-point (CLP) 5-point (8080A)	As needed Refer to method	$r \geq 0.995$ (See method for CLP)	Repeat calibration
		Continuing calibration	Every 12 hours (CLP) Daily & every 10 samples (8080A)	%D ≤ 25% (CLP) %D ≤ 15% (8080)	1. Repeat test 2. Recalibrate
		Breakdown check (Endrin and DDT)	Every 12 hours (CLP) Daily (8080A)	≤ 20% for each compound (& ≤ 30% total for CLP)	1. Correct problem 2. Recalibrate
		Method blank	1 per batch & sub-batch	All analytes < CRQL (CLP) < Reporting limits (8080A)	Re-extract & reanalyze sub-batch
		Surrogate spikes	All analyses	Tetrachloro-m-xylene Decachlorobiphenyl (See Table 5.4)	Flag data (CLP) Reanalyze sample (8080A)

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
California LUFT Manual	Purgeable Petroleum Hydrocarbons	Matrix spike & Matrix spike duplicate	1 set per batch	Method limits (See Table 5.4)	Flag data (CLP) Narrate in report (8080A)
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.4)	Re-extract & reanalyze sub-batch
		Second column confirmation	100% for positive results \geq reporting limit	Qualitative confirmation	N/A
		Initial 5-point calibration	As needed Refer to method	$r \geq 0.995$	Repeat calibration
		Continuing calibration	Daily & every 10 samples	$\%D \leq 15\%$	1. Repeat test 2. Recalibrate
		Method blank	1 per batch & sub-batch	All analytes < reporting limit	Clean system and reanalyze sub-batch
		Surrogate spikes	Every analysis	1,4-Bromofluorobenzene (See Table 5.2)	Reanalyze sample
		Matrix spike & Matrix spike duplicate	1 set per batch	Method 8020A limits (See Table 5.2)	Narrate in report
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.2)	Correct problem and reanalyze sub-batch
		Second column confirmation	100% for BTEX results \geq reporting limit if no gasoline present	Qualitative confirmation	N/A

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
California LUFT Manual	Extractable Petroleum Hydrocarbons	Initial 5-point calibration	As needed Refer to method	$r \geq 0.995$	Repeat calibration
		Continuing calibration	Daily & every 10 samples	%D $\leq 15\%$	1. Repeat test 2. Recalibrate
	Method blank	1 per batch & sub-batch	All analytes < reporting limit	Re-extract & reanalyze sub-batch	
	Matrix spike & Matrix spike duplicate	1 set per batch	Statistical limits (See Table 5.3)	Narrate in report	
8240B CLP SOW	Volatile Organics by GC/MS	Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.3)	Re-extract & reanalyze sub-batch
		Mass scale calibration using PFTBA	Daily	N/A	N/A
	Mass spectral ion intensity check using BFB	Every 12 hours	Refer to method	1. Return Instrument 2. Repeat BFB analysis	
	Initial 5-point calibration	As needed Refer to method	RSD $\leq 20.5\%$ & min. RRF for Table 2 compounds (CLP), RSD $\leq 30\%$ for CCC & min. RRF for SPCC compounds (8240B)	Repeat calibration	

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
		Continuing calibration	Every 12 hours	%D \leq 25% & min. RRF for Table 2 compounds (CLP), %D \leq 20% for CCC & min. RRF for SPCC compounds (8240B)	1. Repeat test 2. Recalibrate
		Method blank	Every 12 hours	All analytes < CRQL, < 5X CRQL for common solvents (CLP); < Reporting limit, < 5X reporting limit for common solvents (8240B)	Clean system & reanalyze sub-batch
		Surrogate spikes	Every analysis	Method limits (See Table 5.5)	Reanalyze sample
		Internal standard	Every analysis	-50% to +100% area count of continuing calibration RT shift <30 sec. for ISs in daily std.	Reanalyze sample
		Matrix spike & Matrix spike duplicate	1 set per batch	Method limits (See Table 5.5)	Narrate in report
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.5)	Correct problem and reanalyze sub-batch
8270B CLP SOW	Semivolatile Organics by GC/MS	Mass scale calibration using PFTBA	Daily	N/A	N/A

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
		Mass spectral ion intensity check using DFTPP	Every 12 hours	Refer to method	1. Retune instrument 2. Repeat DFTPP analysis
		Initial 5-point calibration	As needed Refer to method	RSD \leq 20.5% & min. RRF for Table 5 compounds (CLP), RSD \leq 30% for CCC & min. RRF for SPCC compounds (8270B)	Repeat calibration
		Continuing calibration	Every 12 hours	%D \leq 25% & min. RRF for Table 5 compounds (CLP), %D \leq 20% for CCC & min. RRF for SPCC compounds (8270B)	1. Repeat test 2. Recalibrate
		Method blank	1 per batch & sub-batch	All analytes < CRQL, < 5X CRQL for phthalates (CLP); < Reporting limit, < 5X reporting limit for phthalates (8270B)	Re-extract & reanalyze sub-batch
		Surrogate spikes	Every analysis	Method limits (See Table 5.6)	Re-extract & reanalyze sample
		Internal standard	Every analysis	-50% to +100% area count of continuing calibration RT shift <30 sec. for ISs in daily std.	Reanalyze sample

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
		Matrix spike & Matrix spike duplicate	1 set per batch	Method limits (See Table 5.6)	Narrate in report
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.6)	Re-extract & reanalyze sub-batch
6010A	Trace Metals	Initial calibration	Daily	Refer to method	Refer to method
CLP SOW	By ICPS	Initial calibration verification (ICV)	Daily	90-110% of true value	Recalibrate
		Initial calibration blank (ICB)	Daily	All elements \leq CRDL (CLP) < Reporting limit (6010A)	Recalibrate
		Interference check (A, AB)	Beginning & end of run sequence	80-120% of true value	Recalibrate (Initial) Reanalyze samples (Final)
		CRDL check (CLP)	Beginning & end of run sequence	N/A	N/A
		Continuing calibration verification (CCV)	Every 10 samples	90-110% of true value	1. Recalibrate 2. Reanalyze samples
		Continuing calibration blank (CCB)	Every 10 samples	All elements \leq CRDL (CLP) < Reporting limit (6010A)	1. Recalibrate 2. Reanalyze samples
		Method blank	1 per batch & sub-batch	All elements \leq CRDL (CLP) < Reporting limit (6010A)	Redigest & reanalyze sub-batch

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
		Laboratory control sample (LCS)	1 per batch & sub-batch	80-120% recovery (CLP) Statistical limits (See Table 5.7, 6010A)	Redigest & reanalyze sub-batch
		Replicate exposures	Every analysis	RSD \leq 20%	Reanalyze sample
		Matrix spike	1 per batch	75-125% recovery	Narrate in report
		Matrix spike duplicate (6010A)	1 per batch	75-125% recovery	Narrate in report
		Duplicate analysis (CLP)	1 per batch	RPD \leq 10%	Flag data
		Serial dilution	1 per batch	90-110% of undiluted value	Flag data (CLP)
7041 (Sb)	Trace metals by GFAA and CVAA	Initial multipoint calibration	Daily	$r \geq 0.995$	Repeat calibration
7060A (As)		Initial calibration verification (ICV)	Daily	90-110% (GFAA) 80-120% (CVAA)	Recalibrate
7740 (Se)		Initial calibration blank (ICB)	Daily	\leq CRDL (CLP) < Reporting limit (7000)	Recalibrate
7421 (Pb)		CRA standard (CLP)	Daily	N/A	N/A
7841 (Tl)		Continuing calibration verification (CCV)	Every 10 samples	90-110% (GFAA) 80-120% (CVAA)	1. Recalibrate 2. Reanalyze samples
7470A (Hg)					
7471A (Hg)					
CLP SOW					

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
7196A	Hexavalent Chromium	Continuing calibration blank (CCB)	Every 10 samples	≤ CRDL (CLP) < Reporting limit (7000)	1. Recalibrate 2. Reanalyze samples
		Method blank	1 per batch & sub-batch	≤ CRDL (CLP) < Reporting limit (7000)	Redigest & reanalyze sub-batch
		Laboratory control sample (LCS)	1 per batch & sub-batch	80-120% recovery (CLP) Statistical limits (See Table 5.7, 7000)	Redigest & reanalyze sub-batch
		Matrix spike	1 per batch	75-125% recovery	Flag data (CLP) Narrate in report (7000)
		Matrix spike duplicate (7000)	1 per batch	75-125% recovery	Narrate in report
		Duplicate analysis (CLP) Analytical spike (GFAA) (CLP)	1 per batch Every sample	RPD ≤ 20% Refer to method	Flag data Refer to method
		Initial multipoint calibration	Daily	r ≥ 0.995	Repeat calibration
		Initial calibration verification (ICV)	Daily	90-110% of true value	Recalibrate
		Initial calibration blank (ICB)	Daily	< Reporting limit	Recalibrate

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
9010A/9012 CLP SOW	Cyanide	Continuing calibration verification (CCV)	Every 10 samples	90-110% of true value	1. Recalibrate 2. Reanalyze samples
		Continuing calibration blank (CCB)	Every 10 samples	< Reporting limit	1. Recalibrate 2. Reanalyze samples
		Method blank	1 per batch & sub-batch	< Reporting limit	Reprep & reanalyze sub-batch
		Matrix spike & Matrix spike duplicate	1 set per batch	75-125% recovery	Narrate in report
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.8)	Reanalyze sub-batch
		Initial multipoint calibration	Daily	$r \geq 0.995$	Repeat calibration
		Initial calibration verification (ICV)	Daily	85-115% of true value	Recalibrate
		Initial calibration blank (ICB)	Daily	\leq CRDL (CLP) < Reporting limit (9010A/9012)	Recalibrate
		Continuing calibration verification (CCV)	Every 10 samples	85-115% of true value	1. Recalibrate 2. Reanalyze samples
		Method blank	1 per batch & sub-batch	\leq CRDL (CLP) < Reporting limit (9010A/9012)	Reprep & reanalyze sub-batch

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
300.0	Anions by Ion Chromatography	Laboratory control sample (LCS)	1 per batch & sub-batch	80-120% recovery (CLP) Statistical limits (See Table 5.8)	Reprep & reanalyze sub-batch
		Matrix spike	1 per batch	75-125% recovery	Flag data (CLP) Narrate in report (9012)
		Matrix spike duplicate (9010A/9012)	1 per batch	75-125% recovery	Narrate in report
		Duplicate analysis (CLP)	1 per batch	RPD \leq 20%	Flag data
		Initial multipoint calibration	Daily	$r \geq 0.995$	Repeat calibration
		Initial calibration verification (ICV)	Daily	90-110% of true value	Recalibrate
		Initial calibration blank (ICB)	Daily	All analytes < reporting limit	Recalibrate
		Continuing calibration verification (CCV)	Every 10 samples	85-115% of true value	1. Recalibrate 2. Reanalyze samples
		Continuing calibration blank (CCB)	Every 10 samples	All analytes < reporting limit	1. Recalibrate 2. Reanalyze samples
		Method blank	1 per batch & sub-batch	All analytes < reporting limit	Reanalyze sub-batch

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
120.1	Conductance	Matrix spike & Matrix spike duplicate	1 set per batch	75-125% recovery ≤20% RPD	Narrate in report
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.8)	Reanalyze sub-batch
		Multipoint calibration	Daily	98-102% of true value for each standard	Repeat calibration
		Calibration verification	Daily	90-110% recovery	1. Repeat check 2. Recalibrate 3. Reanalyze batch
130.2	Hardness	Duplicate analysis Laboratory control sample (LCS)	1 per batch 1 per batch & sub-batch	RPD ≤20% Statistical limits (See Table 5.8)	Narrate in report 1. Restandardize titrant 2. Reanalyze sub-batch
		Method blank	1 per batch & sub-batch	< Reporting limit	Reanalyze batch
		Duplicate analysis	1 per batch	RPD ≤20%	Narrate in report
150.1	pH	2 to 3-point calibration	Daily	±0.05 to > ±0.1 pH units	Repeat calibration 1. Recalibrate
		Calibration verification	Daily	±0.05 to > ±0.1 pH units	2. Reanalyze batch
		Duplicate analysis	1 per batch	±0.05 to > ±0.1 pH units	Narrate in report

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
160.1	Total Dissolved Solids	Method blank	1 per batch & sub-batch	± 0.5 mg	Reprep and reanalyze batch
		Duplicate analysis	1 per batch	RPD $\leq 20\%$	Narrate in report
160.2	Total Suspended Solids	Method blank	1 per batch & sub-batch	± 0.5 mg	Reprep and reanalyze batch
		Duplicate analysis	1 per batch	RPD $\leq 20\%$	Narrate in report
310.1	Carbonate/ Bicarbonate	3-point calibration	Daily	± 0.05 to ± 0.1 pH units	Repeat calibration
		Calibration verification	Daily	85-115% recovery	1. Recalibrate 2. Reanalyze batch
		Method blank	1 per batch & sub-batch	< Reporting limit	Reanalyze batch
		Duplicate analysis	1 per batch	RPD $\leq 20\%$	Narrate in report
340.2	Fluoride	Matrix spike & Matrix spike duplicate	1 set per batch	75-125% recovery	Narrate in report
		Multipoint calibration	Daily	$r \geq 0.995$	Repeat calibration
		Calibration verification	Daily	90-110% recovery	Recalibrate
		Method blank	1 per batch & sub-batch	< Reporting limit	Reanalyze batch

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	
350.3	Ammonia	Matrix spike & Matrix spike duplicate or Duplicate	1 set per batch	75-125% recovery RPD \leq 20%	Narrate in report	
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.8)	Reanalyze batch	
		Multipoint calibration	Daily	$r \geq 0.995$	Repeat calibration	
		Calibration verification	1 per batch	85-115% recovery	1. Recalibrate 2. Reanalyze batch	
		Method blank	1 per batch & sub-batch	< Reporting limit	Reanalyze batch	
		Matrix spike & Matrix spike duplicate or Duplicate	1 set per batch	75-125% recovery RPD \leq 20%	Narrate in report	
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.8)	Reanalyze batch	

TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
9060 415.1	Total Organic Carbon	Initial calibration (4 replicates of 1 point)	Daily	1125-1875 average raw instrument reading	Reprep standard & recalibrate
		Calibration verification	Daily	85-115% of true value	Recalibrate
		Method Blank	1 per batch & sub-batch	< Reporting limit	Reanalyze batch
		Duplicate analysis	1 per batch	RPD \leq 50%	Narrate in report
		Matrix spike	1 set per batch	75-125% recovery	Narrate in report
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.8)	Reanalyze batch
9070/9071 413.1	Oil & Grease Gravimetric	Balance calibration check at 1 g & 100 g	Beginning & end of analytical sequence	\pm 0.001 g of true weight	Recalibrate balance
		Method blank	1 per batch & sub-batch	< Reporting limit	1. Redesiccate & reweigh samples 2. Re-extract sub-batch
		Matrix spike & Matrix spike duplicate or Duplicate	1 set per batch	75-125% recovery RPD \leq 20%	Narrate in report
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.8)	Re-extract & reanalyze sub-batch
413.2	Oil & Grease by IR	Initial 5-point calibration	Daily	$r \geq 0.995$	Repeat calibration

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TABLE 11.1 SUMMARY OF CALIBRATION AND QUALITY CONTROL PROCEDURES

ANALYTICAL METHOD	APPLICABLE PARAMETER	QUALITY CONTROL CHECK	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
418.1	Total Petroleum Hydrocarbons by IR	Continuing calibration	Every 10 samples	80-120% of true value	1. Repeat test 2. Recalibrate
		Method blank	1 per batch & sub-batch	< Reporting limit	1. Clean system & recheck 2. Re-extract & reanalyze sub-batch
		Matrix spike & Matrix spike duplicate or Duplicate	1 set per batch	75-125% recovery RPD \leq 20%	Narrate in report
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.8)	Re-extract & reanalyze sub-batch
		Initial 5-point calibration	Daily	$r \geq 0.995$	Repeat calibration
		Continuing calibration	Every 10 samples	80-120% of true value	1. Repeat test 2. Recalibrate
		Method blank	1 per batch & sub-batch	< Reporting limit	1. Clean system & recheck 2. Re-extract & reanalyze
		Matrix spike & Matrix spike duplicate or Duplicate	1 set per batch	75-125% recovery RPD \leq 20%	Narrate in report
		Laboratory control sample (LCS)	1 per batch & sub-batch	Statistical limits (See Table 5.8)	Re-extract & reanalyze sub-batch

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12.0 QUALITY ASSURANCE AUDITS AND PERFORMANCE EVALUATIONS

Integral to Pace's quality assurance program is a program of internal audits designed to provide feedback about the effectiveness and completeness of the various quality control and quality assurance systems in the laboratory. This section describes the types of audits conducted at Pace and discusses the roles and responsibilities of Pace personnel related to these audits.

12.1 INTERNAL AUDITS

12.1.1 Quality Assurance Auditor

The QA auditor is responsible for designing and/or performing QA performance and systems audits. Since QA audits represent an independent assessment of laboratory functions, the auditor must be functionally independent from laboratory operations to ensure objectivity. However, the auditor must be familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The auditor's ability to identify components of systems that are critical to overall data quality is especially important.

Internal audits are typically conducted by the laboratory QA Officer, who may be assisted by other laboratory personnel. The QA Officer reports directly to the Laboratory General Manager, and therefore is independent of laboratory operations. The QA Officer evaluates audit observations and verifies the completion of corrective actions.

12.1.2 Scope and Frequency of Internal Audits

Internal systems audits are conducted at a minimum frequency of one per quarter. The scope of these audits may include the examination of the operations of a specific analytical department or may focus on the evaluation of a specific quality-related system as applied throughout the laboratory.

Examples of system-wide elements which can be audited include:

- Standard operating procedures, including system of review, issue, filing, maintenance, training, understanding, documentation of deviations and implementation of SOPs.
- Adherence to standard operating procedures, the QAP and regulations.
- Personnel and training files, including job descriptions, resumes, documented training and training file maintenance.
- General laboratory safety, including appropriate clothing, waste disposal, health and safety plan review, obvious safety concerns.
- Labeling of reagents, solutions, standards, and associated documentation.
- Equipment and instrumentation documentation, calibration/ maintenance records, operating manuals.

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- Sample handling, storage and disposal including storage locations, security, tracking/chain-of-custody, disposal practices and records, labeling and retention.
 - Documentation of sample analysis, methodologies, quality control requirements.
 - Documentation of discrepancy reports and corrective action.
 - General procedures for data security, review, documentation, reporting and archiving.

When the operations of a specific department are evaluated, a number of functions are reviewed, such as:

- Documentation of technical training and analyst proficiency
- Method detection limit studies
- Internal chain-of-custody documentation
- Nonconformance documentation
- Documentation of standard preparations
- Instrument maintenance documentation
- Standard operating procedures
- Control charts
- Documentation of sample preparation and analysis
- Documentation of data review

As required on specific projects, internal audits are performed to ensure laboratory conformance to site workplans, sampling and analysis plans, QAPP, etc. Project audits can include review of the following items of concern:

- Sample log-in and chain-of-custody records
- Sample storage procedures and records
- Sample preparation and analysis procedures
- Method validation (where applicable)
- Conformance to QAPP
- Control charts (if applicable)
- Precision and accuracy assessment
- Method blanks, reagent blanks, duplicates, check samples, fortifications, surrogates, etc.
- Calibration
- Data packages
- Analyst qualifications
- Data validation and reporting

12.1.3 Internal Audit Reports and Corrective Action Plans

A full description of the audit, including the identification of the department or operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations made during the course of the audit, are summarized in an internal audit report. Although other personnel may assist with

the performance of the audit, the QA Officer writes and issues the internal audit report. The QA Officer identifies which audit observations are deficiencies that require corrective action.

Once completed, the internal audit report is issued jointly to the laboratory general manager and the manager(s)/supervisor(s) of the audited department(s) or operation(s). The responsible manager(s)/supervisor(s) respond with a plan to correct all of the deficiencies cited by the due date specified in the audit report. Each response must include timetables for completion of all proposed corrective actions.

The QA Officer reviews and accepts the audit responses. If the response is accepted, the QA Officer uses the action plan(s) and timetable(s) as a guideline for verifying completion of the corrective action(s). If the QA Officer determines that the audit response does not adequately address the correction of cited deficiencies, the response will be returned for modification.

To complete the audit process, the QA Officer performs a re-examination of the areas where deficiencies were found to verify that all proposed corrective actions have been implemented. An audit deficiency is considered closed once implementation of the necessary corrective action has been verified. If corrective action cannot be verified, the associated deficiency remains open until that action is completed.

12.2 EXTERNAL AUDITS

Pace is audited as required by regulatory agencies to maintain laboratory certifications, and by various commercial clients. External audits include those by state laboratory certification agencies, USEPA, Army Environmental Center (AEC), Army Corps of Engineers, and other appropriate federal, state and private agencies (e.g., MITRE).

Audit teams external to the company review the laboratory to assess the existence of systems, implementation of the systems, and degree of technical expertise. QA staff host the audit team and collect notes during the audit process. These notes are communicated to the General Manager, Laboratory Manager and the supervisor. Generally, the auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. In some cases, in lieu of an official report, items of concern are discussed during a debriefing convened at the end of the on-site review process. The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the QAO. The laboratory manager provides the necessary resources for staff to develop and implement the corrective action plans. The QAO collates this information and provides a written report to the audit team. The report contains the corrective action plan and expected completion dates for each element of the plan. QAO staff follow-up with the laboratory staff to ensure that corrective actions are implemented.

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12.3 TOTAL QUALITY SYSTEM AUDIT

The Corporate Quality Office coordinates on-site audits of each laboratory facility. The corporate audit is conducted by the Vice President of Quality with the assistance of Pace QAO's. This audit is designed to evaluate all aspects of facility operations and is not limited to only laboratory operations. Audits may either be system related or technical in nature, depending on the type of information needed for making quality improvements.

Assessment of quality/technical practices within laboratory operations involves three types of review.

1. Documentation - On-going monitoring of quality issues in laboratory offices requires that the corporate quality office receive the following hardcopy information on an "as released" basis, supplied as part of the Quarterly Quality Report to management.
 - PE scores (e.g., WP, WS, CLP, COE, AEC, EML, EMSL, NIOSH, etc.)
 - Certification/parameter list approvals
 - External audit reports and responses
 - Internal audit reports
 - Copies of all newly developed SOPs (also critical for the MRD program)
 - Periodic data validation report reviews

2. Pre-audit Documentation
 - Verify methods capability matrix information (completed for each method routinely performed in the laboratory)
 - Sample report for each reporting deliverable level used by the facility
 - Facility and equipment inventory
 - Current organization chart
 - LIMS sample management reports

3. Facility Audit Process
 - Checklist approach with minimal textual report
 - Objective scoring system based on checklist results
 - Technical review based upon compliance to MRDs and associated SOPs, published methods, federal program requirements
 - "GLP" documentation trail (retrace the path of a sample through the laboratory)
 - Non-analytical documentation (e.g., training logs, discrepancy/corrective action reports, preventative maintenance, standards traceability, etc.)
 - Review of QA procedures (relative to the Pace Generic Quality Assurance Plan)
 - Credential/certification verification
 - Instrumentation and facility utilization (e.g., automation, workflow)

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- Health and safety, waste disposal programs (reviewed in conjunction with technical audit)
 - Debriefing meeting
 - Issue final audit report and establish corrective action schedule

12.4 PERFORMANCE EVALUATION AUDITS

12.4.1 Pace PE Samples

Double blind performance evaluation (PE) samples are submitted periodically to all Pace laboratories to evaluate all areas of the laboratory. The program is identified as the Pace Interlaboratory Testing Survey (PITS). Results from internal PE sample analyses are processed by the analytical departments and reported to the responsible Project Manager. The Project Manager issues a standard analytical report to a fictitious client (PE vendor) in a manner identical to that done for all other client work orders.

Evaluated PE results are given to the laboratory for review. For parameters where the reported results fall within the defined acceptance limits, no further action is required. For parameters where the reported results fall outside acceptance limits, the responsible Department Manager/Supervisor must investigate in an effort to find the root cause of each problem. For each missed quantitation, the Manager/Supervisor summarizes the findings of their investigation in a PE investigation report. Each report must include a description of the corrective action(s) that will be taken to prevent recurrence of the problem. Completed reports are passed to the QA Officer for review, then forwarded to Pace's corporate quality office. The internal auditing process is used to verify implementation of corrective actions.

12.4.2 EPA WP and WS PE Studies

Pace labs routinely participate in EPA's Water Pollution (WP) and Water Supply (WS) round-robin PE studies. Each of these studies is conducted twice per year. Generally, all analytical sections of the laboratory participate in the WP and WS PE studies. Examples of parameters analyzed under each study are listed below. Satisfactory performance on these studies is essential as they are a fundamental requirement of state accreditation programs.

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- EPA Performance Evaluations - Water Supply - Semiannual (April and September)
 - Trace Metals
 - Nitrate/Nitrite/Fluoride
 - Insecticides
 - Herbicides
 - PAHs
 - Adipate/Phthalates
 - Trihalomethanes (THMs)
 - Volatile Organic Compounds
 - Turbidity
 - Total Filterable Residue
 - Calcium (as CaCO₃)
 - pH
 - Alkalinity
 - Corrosivity
 - Sodium
 - Sulfate
 - Total Cyanide

 - EPA Performance Evaluations - Water Pollution - Semiannual (February and August)
 - Trace Metals
 - Minerals
 - Nutrients
 - Demand
 - PCBs
 - PCBs in Oil
 - Pesticides
 - Volatile Halocarbons
 - Volatile Aromatics
 - Total Cyanide
 - Non-Filterable Residue
 - Oil and Grease
 - Total Phenolics

For PE quantitations evaluated as being outside the acceptance ranges, investigations must be performed by the responsible Manager(s)/Supervisor(s) and reports must be completed as described above for the Pace PE samples. The reports are reviewed by the QA Officer, then forwarded to the various state accreditation agencies for their review.

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12.4.3 Other PE Studies

Other PE samples may be performed by Pace in conjunction with a specific program or contract. Examples include:

- PE samples distributed by the US Army Corps of Engineers, Missouri River Division, as part of their laboratory evaluation process
- Quarterly blind (QBs) PE samples distributed to laboratories participating in EPA's Contract Laboratory Program

In addition, clients may arrange for PE samples to be analyzed, either as part of a laboratory evaluation process or as a periodic performance check.

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13.0 PREVENTIVE MAINTENANCE

The objective of Pace's preventive maintenance program is to establish a system of instrument care that prevents the loss of analytical quality control and results in a minimum of lost productivity due to instrument failure. This program includes a system for documenting all routine and non-routine instrument maintenance and repairs.

Pace maintains service contracts for most major analytical equipment including chromatographic instruments, balances, atomic absorption, and inductively coupled plasma instruments. All equipment and instruments generating analytical results have calibration and maintenance records.

13.1 MAINTENANCE RESPONSIBILITIES

The Laboratory Operations Manager and Supervisors are responsible for providing technical leadership to all staff involved with chemical analysis. This leadership role includes serving as a technical resource to help solve equipment and method problems, evaluating and recommending investments in new technologies, improving efficiency, and coordinating instrument repair and maintenance.

The primary responsibility for the maintenance of instruments and equipment rests with each analytical Department Manager/Supervisor. The Department Manager/Supervisor is further responsible for developing procedures and schedules for maintaining each major instrument or piece of equipment and for delegating specific maintenance responsibilities to department staff.

13.2 MAINTENANCE SCHEDULES

The effectiveness of the maintenance program relies heavily on adherence to prescribed schedules for maintaining each instrument or piece of equipment. A schedule is established for all routine maintenance. Other maintenance activities may also be identified as requiring attention on an as-needed basis. Manufacturers' recommendations provide the primary basis for developing these schedules, and manufacturers' service contracts provide primary maintenance for some major instruments.

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument. SOPs are written for each instrument that cover basic operation and maintenance procedures. The following are brief summaries of maintenance for each type of major instrumentation. This information is also listed by major instrumentation system in Table 13.1.

13.2.1 Preventive Maintenance - GC/MS

Regularly performed maintenance includes, but is not limited to the following for GC/MS instrumentation:

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- Hard tune with calibration gas (pftba)
 - Removal of 2-3 inches from the injection end of the capillary columns
 - Replacement of 2-3 inches of column packing from the injection end of packed columns
 - Injection port liner replacement
 - Replace injection port septum
 - Clean ion source as needed
 - Check vacuum pump oil level
 - Check carrier gas tanks
 - Replace or recondition vent traps

13.2.2 Preventive Maintenance - GC

Regularly performed maintenance includes, but is not limited to the following for extractable GC instrumentation:

- Removal of 5-10 inches of guard column (if applicable) and 2-3 inches from the injection end of the capillary columns
- Replacement of 2-3 inches of column packing from the injection end of packed columns
- Injection port liner and RP seal replacement
- Replacement of septum
- Fill solvent rinse bottles in auto sampler
- Check carrier and support gases
- NRC wipe test ECD

Regularly performed maintenance includes, but is not limited to, the following for volatile organics GC instrumentation:

- Clean and bake sparge tubes
- Replace trap as needed
- Check carrier and support gases
- Replace transfer line as needed
- Replace nickel tube as needed
- Clean or replace PID as needed

13.2.3 Preventive Maintenance - ICP

- Check liquid argon tank level
- Change pump tubing
- Clean nebulizer and spray chamber as needed
- Replace and realign plasma torch when required
- Check cooling system water level
- Empty waste reservoir when full

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13.2.4 Preventive Maintenance - AA Graphite Furnace

- Check and align source lamps
- Clean and inspect graphite tube, replacing when surface appears excessively burnt or cracked
- Clean and inspect contact ring, replacing when excessively worn
- Clean mirrors for optical sensor and sample compartment windows
- Check autosampler injector alignment and deposition

13.2.5 Preventive Maintenance - Mercury Analyzer

- Check and align source lamp
- Remove and clean sample cell and connecting tubes
- Check sparger for proper operation
- Clean sample compartment windows

13.2.6 Preventive Maintenance - General Laboratory Areas

- Calibrate automatic pipets and burets monthly
- Clean, check, calibrate to manufacturers specifications all pH, DO, conductivity and, turbidity meters, and spectrophotometers annually
- General housekeeping: keep counter tops, hoods, and floors clean and keep safety equipment accessible
- Check airflow in hoods once a quarter

13.2.7 Preventive Maintenance**Thermometers, Refrigerators, Ovens and Balances:**

Laboratory thermometers are calibrated against NIST traceable thermometers annually. Digital thermometers are calibrated quarterly. The results are recorded in a logbook specific to that purpose. Correction factors are recorded on the thermometer tags, along with the unique thermometer identification number and calibration date, and are used by Pace personnel to correct actual temperature measurements. The correction factor is applied to each reading until the thermometer is calibrated again. Use of thermometers with a correction of $> 5^{\circ}\text{C}$ is avoided. Pace minimizes the need to apply corrections by utilizing the correct media, thermometers and procedures during calibration.

Refrigerators, freezers and ovens are monitored once or twice daily or as used, dependent upon the function of the unit. Logbooks are maintained for documentation of readings and corrective actions. If a unit fails acceptance criteria, monitoring is continued until the temperature stabilizes within the range or appropriate corrective actions are taken. Monitoring occurs at one hour intervals for a maximum four hour period; if the reading following the temperature control adjustment is out, the unit is considered "out of order", and

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is emptied and serviced. It is not put back into service until shown to be stable at the required temperature range.

Analytical balances are calibrated annually (or more frequently if required under a specific program) by an outside service. A dated sticker, certifying the calibration, is placed on each balance. Records for balance calibration/servicing are maintained in Pace QA files. Multi and single point calibration checks are regularly performed to ensure the accuracy of each balance. The results are recorded in dedicated logbooks that are maintained at each balance location. Balances that do not satisfy specifications are taken out of service for replacement or repair. ASTM Class "1" weights must be verified/calibrated every two years.

13.3 MAINTENANCE DOCUMENTATION

All routine and non-routine instrument maintenance is documented in maintenance logbooks assigned to each instrument. To provide a clear and complete history of repairs and maintenance associated with each instrument, each maintenance entry must include the following elements:

1. An explanation of the reason for the maintenance or repair, e.g., was this action taken to fix a problem or was it part of routine instrument maintenance
2. A full description of the maintenance or repair actions taken
3. A description of how the analyst demonstrated that the analytical system was operating in control after completion of the maintenance actions, but before the resumption of sample analysis

When maintenance is performed to repair an instrument problem, the entry should include a description of the symptoms or problem that precipitated the maintenance actions. Depending on the initial problem, demonstration of return to control may be satisfied by the successful analysis of a reagent blank or continuing calibration standard. The entry must include a summary of the results of that analysis and a verification by the analyst that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance logbook, if different from the date(s) of the maintenance.

13.4 SPARE PARTS

Along with the development of maintenance schedules, an adequate inventory of spare parts is required to minimize equipment downtime. This inventory should emphasize those parts and supplies that:

- are subject to frequent failure,

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-
- have limited useful lifetimes, or
 - cannot be obtained in a timely manner should failure occur.

Department Manager/Supervisors are responsible for maintaining an adequate inventory of necessary spare parts for all major instruments and equipment items. Examples of spare parts maintained for major instrumentation systems are listed in Table 13.1.

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TABLE 13.1

**Scheduled Maintenance Procedures and Representative Spare Parts
for Major Instrumentation**

INSTRUMENT	MAINTENANCE PROCEDURE	SPARE PARTS
Gas Chromatography Mass Spectrometry (GC/MS)	<ol style="list-style-type: none"> 1. Change septa and inserts as needed 2. Clip column 3. Replace pump oils as needed 4. Change gas line dryers as needed 5. Clean source as needed 6. Replace electron multiplier as needed 	<ol style="list-style-type: none"> 1. Syringes 2. Septa and inserts 3. GC columns 4. Various electronic components 5. Plumbing supplies - tube fittings
Gas Chromatography (GC)	<ol style="list-style-type: none"> 1. Change septa and inserts as needed 2. Clip column 3. Change gas line dryers as needed 4. Leak check when installing new analytical column 5. Check inlet system for residue buildup periodically 	<ol style="list-style-type: none"> 1. Syringes 2. Septa and inserts 3. GC columns 4. Various electronic components 5. Plumbing supplies - tube fittings
Purge and Trap Sample Concentrator	<ol style="list-style-type: none"> 1. Replace trap as needed 2. Decontaminate system as required by blank analysis 3. Leak check system 4. Measure flowrates for each sparging position monthly 	<ol style="list-style-type: none"> 1. Spare traps 2. Various electronic components and circuit boards 3. Plumbing supplies - tube fittings
Inductively Coupled Argon Plasma Spectrometer (ICP)	<ol style="list-style-type: none"> 1. Clean torch assembly and mixing chamber when discolored or after 8 hours of running high dissolved solids samples 	<ol style="list-style-type: none"> 1. Spare torch and mixing chamber 2. Spare coil 3. Plumbing supplies - tube fittings
Graphite Furnace Atomic Absorption Spectrophotometer	<ol style="list-style-type: none"> 1. Change graphite contact rings as needed 2. Clean quartz windows as needed 	<ol style="list-style-type: none"> 1. Contact rings 2. Graphite cups and electrodes 3. Autosampler tubing
Hg Analyzer	<ol style="list-style-type: none"> 1. Clean tubing and quartz cell as needed 2. Clean aspirator as needed 3. Replace drying tube media daily 	<ol style="list-style-type: none"> 1. Quartz cells 2. Aspirator 3. Plumbing supplies

14.0 DATA QUALITY ASSESSMENT

Data quality assessment requires the review of quality control samples for precision, accuracy, representativeness, completeness, and comparability. Precision and accuracy data are used to determine the acceptability of analytical results. Standard operating practices require the use of a minimum of 20 tabulated precision or accuracy data points to prepared quality control charts. However, preliminary control limits can be established using as few as four data points. The Shewhart technique is the statistical method used to construct the charts. These quality control charts provide a quick visual means for monitoring the daily performance of the laboratory and identifying nonconformance trends.

For every batch of samples analyzed, a series of quality control samples are analyzed to assess the precision, accuracy and validity of the analysis. These data are reviewed before release of the data. All QC data are stored at Pace and are useable for determination of method precision and accuracy. Pace makes every effort to meet or exceed the accuracy and precision data as defined within specific methodologies. However, for actual matrices these data may not be comparable.

To estimate accuracy, spiked blank samples (laboratory control samples) and matrix spike sample recoveries are evaluated. This allows for the determination of both method and actual sample batch accuracy. Precision is measured and monitored in two ways: using range control for duplicate pairs and relative percent differences. Pace uses the formulas presented in Standard Methods, SW 846 and the USEPA Quality Assurance handbooks for calculations of precision and accuracy. This section illustrates calculations for determining data quality in terms of precision and accuracy. In addition to calculations concerning precision and accuracy, those which pertain to representativeness, completeness, and comparability are used to ascertain the level at which DQOs have been satisfied. Calculations for these other data quality indicators are included as well.

14.1 PRECISION

Precision is the degree to which the measurement is reproducible. Precision can be assessed by duplicate measurements of a laboratory control sample or an environmental sample. The precision of laboratory analytical data can be expressed using one of several statistical determinations, including: 1--standard deviation, 2--range, 3--relative standard deviation, also known as the coefficient of variation, and 4--relative percent difference.

Standard deviation is a measure of the variance of individual observations from the mean. It is usually denoted as "s" and is defined as:

$$s = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n - 1}} \quad \text{or} \quad \sqrt{\frac{\sum_{i=1}^n X_i^2 - (\sum_{i=1}^n X_i)^2/n}{n - 1}}$$

In this equation,

n	=	population size
X_i	=	ith observation in the sample
\bar{X}	=	sample mean

Standard deviation can be used to determine variation among several RPD values for duplicate pairs and establish statistical limits for duplicate RPD. Range control may also be used.

Range is the largest observation in a data set minus the smallest observation in the data set, often denoted as "R".

$$R = A - B$$

$$\bar{X} = \frac{A + B}{n}$$

Where:

R	=	Range of a pair of results
\bar{X}	=	Average of a pair of results
A	=	Duplicate value 1
B	=	Duplicate value 2
n	=	2 (represents a single duplicate pair)

To graphically represent the data of numerous duplicate pairs on control charts, the following calculations are performed using statistical numbers.

$$\bar{\bar{X}} = \text{the sum of } \bar{X} / n$$

$$\bar{R} = \text{the sum of } R / n$$

Where:

$\bar{\bar{X}}$	=	Grand Mean
\bar{R}	=	Average Range
\bar{X}	=	Average of a pair of results
R	=	Range of a pair of results
n	=	2 (represents a single duplicate pair)

Control limits for ranges (R - bar chart):

$$CL = 3.27 (\bar{R})$$

$$WL = \bar{R} + 2/3 (3.27 \bar{R} - \bar{R})$$

Where:

\bar{R}	=	Average Range
CL	=	Control Limit
WL	=	Warning Limit

To determine if the proper range control chart is being used for evaluation of a duplicate pair of results, the X control chart may be used.

Control limits for averages (X - bar chart):

$$UCL = \bar{X} + 1.88 (\bar{R})$$

$$LCL = \bar{X} - 1.88 (\bar{R})$$

$$UWL = \bar{X} + 2/3 (1.88 \bar{R})$$

$$LWL = \bar{X} - 2/3 (1.88 \bar{R})$$

Where:

- \bar{X} = Grand Mean
- \bar{R} = Mean Range
- UCL = Upper Control Limit
- LCL = Lower Control Limit
- UWL = Upper Warning Limit
- LWL = Lower Warning Limit

Relative standard deviation (RSD), or coefficient of variation (CV), is a commonly used measure of variability that is adjusted for the magnitude of the values in the sample:

$$RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100\%$$

RSD is used most often when the size of the standard deviation changes with the size of the mean. Individual measurements of RSD or CV can be combined (pooled) to give an overall measure of variability for a given type of analysis or measurement:

$$\text{Pooled CV} = \sqrt{\frac{\sum_{i=1}^n X_i^2 DF_i}{\sum_{i=1}^n DF_i}}$$

In this equation,

- X_i = CV of data set i
- DF_i = degrees of freedom from data set i ($k_i - 1$)
- n = number of data sets
- k_i = number of data points in set i
- i = data set 1, 2, 3 . . . n

Relative percent difference (RPD) is another commonly used measure of variability that is adjusted for the magnitude of the measured values. It is used only when the sample

contains two observations. The results of the duplicate analyses are computed and the absolute RPD is calculated using the following equation:

$$RPD = \frac{|x_1 - x_2|}{\frac{x_1 + x_2}{2}} \times 100$$

Where:

RPD	= Relative Percent Difference
x_1	= first sample value (original)
x_2	= second sample value (duplicate)

For duplicate results RPD is directly related to RSD by:

$$RPD = \sqrt{2} RSD$$

The RPDs are tabulated, the average RPD and standard deviation are calculated, and a control chart constructed. Formulas for control limit are:

$$\begin{aligned} UCL &= \overline{RPD} + 3s \\ UWL &= \overline{RPD} + 2s \\ LCL &\text{ always equals } 0 \end{aligned}$$

14.2 ACCURACY

Accuracy measures the degree of difference between observed and true values. The actual test result is compared to the theoretical result of 100% recovery and the percent recovery calculated. The accuracy of sample data can be assessed using the laboratory control spike, the environmental sample spiked with target analytes (matrix spike), or surrogate standards. Accuracy data are evaluated against established control limits. The percent recovery is computed using the following equation:

$$\%R = \frac{M - B}{T} \times 100$$

Where:

M	= Measured concentration of analyte in spiked sample
B	= Background concentration of unspiked sample
T	= Target value (known concentration of analyte in spike)

The percent recovery data for a compound or parameter are tabulated, the average percent recovery and standard deviation calculated, and a control chart constructed.

The accuracy of laboratory analytical data can also be presented in terms of: 1--percent relative error, and 2--confidence intervals at the 95% level.

$$\text{Percent Relative Error} = \frac{\text{Measured Value} - \text{Actual Value}}{\text{Actual Value}} \times 100\%$$

$$95\% \text{ Confidence Interval} = X \pm \sqrt{\frac{t^2(a, n-1) s^2}{n}}$$

In this equation,

X	=	sample mean
s	=	sample standard deviation
n	=	sample size
a	=	risk level (0.025 for 95% confidence interval)
t(a, n-1)	=	value of the tabulated student's "t" distribution for n-degrees of freedom and risk level a

Percent recovery is related to percent relative error by:

$$\% \text{ Recovery} = \% \text{ Relative Error} + 100$$

The correlation coefficient, "r" is used to determine the acceptability of multi-point initial calibration data. The correlation coefficient value reflects the degree of fit of the calibration data with a linear or other curve function and is calculated as:

$$r = \frac{n(\sum xy) - (\sum x)(\sum y)}{\sqrt{[n(\sum x^2) - (\sum x)^2][n(\sum y^2) - (\sum y)^2]}}$$

In this equation,

x	=	concentration of the standard
y	=	instrument response (peak area)
n	=	number of calibration points (x,y data pairs)

14.3 CONTROL CHARTS

Once a minimum of 20 QC data points are tabulated, an accuracy control chart can be constructed as follows:

Compute the mean value of the tabulated points.

$$P = \frac{\sum P_i}{n}$$

Where:

P	=	Mean Value
P _i	=	Sample result
n	=	Total number of results in data set

Using the mean (P), compute the standard deviation (SD) of the data set.

$$SD = \sqrt{\frac{\sum (x - P)^2}{(n - 1)}}$$

Where:

SD	=	Standard deviation
x	=	Sample result
P	=	Mean value
n	=	Total number of results in data set

Employing the mean and standard deviation of the data set, determine the upper and lower warning and control limits are determined as described in 14.3.1 and 14.3.2.

14.3.1 Warning Limits

Warning limits represent the 95% confidence interval and are equal to the mean value for the control sample plus or minus two standard deviations (2SD). Exceeding these limits is warning that the analytical system may be approaching an out-of-control situation and should be inspected for possible sources of error. The warning limits are calculated with the following equation:

$$WL = P \pm 2SD$$

Where:

WL	=	Warning limits
P	=	Mean
SD	=	Standard deviation

14.3.2 Control Limits

Control limits represent the 99% confidence interval and are equal to the mean value of the control sample, plus or minus three standard deviations (3SD). Exceeding these limits indicates that the analytical system is out-of-control. Control limits are calculated using the following equation:

$$CL = P \pm 3SD$$

Where:

CL	=	Control limits
P	=	Mean
SD	=	Standard deviation

14.3.3 Utilization of Acceptance Limits

Once the warning and control limits are established, a control chart is constructed.

To verify the control chart, the initial data points are checked against the newly generated limits for statistical outliers. Subsequently generated QC sample

data are then plotted on the chart. The plotted points must fall within the control limits for the result to be accepted and the associated sample data validated. Outliers are evaluated for corrective action measures. Control charts are prepared for each required parameter, the limits updated at least annually and graphs produced for identification of trends.

The laboratory must also review control charts for out-of-statistical control trends. Any of the following trends is considered an out-of-control trend occurrence.

- Any three consecutive points are outside warning limits.
- Any seven consecutive points are on the same side of the mean of the central line.
- Any six consecutive points are such that each point is larger (smaller) than its immediate predecessor.
- Any obvious cyclic pattern in the points.

14.4 REPRESENTATIVENESS

Representativeness is a qualitative element related to the ability to collect a sample that reflects the characteristics of that part of the environment being assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project site. It is specifically addressed in each work plan.

14.5 COMPLETENESS

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions. It is expected that laboratories should provide data meeting QC acceptance criteria for 95% or more of the requested determinations. It is necessary for data users to identify any sample types which require 100% completeness. The mathematical formula is as follows:

$$C = \frac{V}{T} \times 100$$

Where:	C	=	Percent completeness
	V	=	Number of measurements judged valid
	T	=	Total number of measurements

14.6 COMPARABILITY

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. For example, the use of EPA approved methods and procedures ensure comparability with other data from previous of following studies using the same methods.

15.0 CORRECTIVE ACTION

This section describes the quality assurance system at Pace established to address the documentation and correction of problems encountered during sample handling and analysis. In many instances, the accurate and timely communication of sample handling and analysis problems to laboratory Managers/Supervisors, Project Managers, and/or the QA Officer can mean the difference between a situation that is corrected with little or no impact to sample data quality and one that results in resampling.

A number of problems encountered in the laboratory require defined courses of corrective action. When a problem arises, action to correct the problem must be taken promptly. For example, laboratory Managers/Supervisors must initiate corrective action whenever QC results for control parameters fail to stay within acceptance limits. In many situations, input from the client is important in deciding how problems are to be resolved.

When errors, deficiencies, unusual occurrences, or out-of control situations exist, the QA program provides systematic procedures, called "corrective actions", to resolve problems and restore proper functioning to the analytical system. Within Pace, a distinction is made between "out-of-control events" and "unusual occurrences" for the purposes of requiring corrective actions.

An out-of-control event is any event which is beyond the acceptance limits established for laboratory operation by Pace SOPs, EPA methods, or client specific contracts or protocols. This can be due to data which are outside of the accepted bounds for accuracy and/or precision, method contamination, improper instrument calibration or maintenance, or deviations from the contract or SOP detected by a QA audit.

An unusual occurrence is a situation in which the analytical system is, strictly speaking, compliant with the protocol or SOP and therefore in control but an atypical or undesirable incident has occurred which warrants further investigation. Such an occurrence could be a holding blank which is contaminated or differences in the pattern of non-spiked target compounds between a spiked and unspiked aliquot of a sample used as the matrix spike.

Both out-of-control events and unusual occurrences are formally documented. Within Pace, the formal documentation report is identified under the following designations: Corrective Action Report (CAR); Non-conformance Memo (NCM); or Discrepancy Report (DR). Each of these reports serves the same purpose (because of this, the names are used inter-changeably) of documenting whenever either type of event is noted. A representative report form is illustrated in Figure 15.1.

15.1 NON-CONFORMANCE MEMO

The primary tool for documenting deficiencies and problems is the nonconformance memo (NCM). NCMs may be initiated by an analyst, laboratory Manager/Supervisor, Project Manager, or other laboratory personnel. The NCM is used to document a specific problem or deficiency noted during sample handling or analysis. Depending on the specific

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problem or deficiency, corrective actions may be taken by the Analyst, laboratory Manager/Supervisor, Project Manager, or other laboratory personnel. Since problems encountered with sample analysis often have the potential to impact data quality, appropriate corrective action is frequently determined in communications between the laboratory Project Manager and the client.

Each NCM requires the initials of the person documenting the problem as well as those of any person documenting additional information or corrective action. Each NCM is then reviewed and initialed by the department Manager/Supervisor. If the deficiency or problem impacts client sample data, the department Manager/Supervisor passes the NCM directly to the appropriate Project Manager for their review and followup. If the nonconformance does not directly impact sample data quality or integrity, the department Manager/Supervisor passes the NCM to the QA Officer.

The NCM must describe the actions taken at each step of the review process. Where an NCM documents an analytical event that is judged to be out-of-control, evidence of return to control must also be documented. Once documentation of the problem, corrective action, and return to control is complete, the NCM is forwarded to the QA Officer for QA review. QA review is documented with the QA Officer's initials and the completed original NCM is either passed back to the appropriate Project Manager to be filed in the client project file or is filed in the QA files, depending on the nature of the nonconformance.

15.2 OUT OF CONTROL EVENTS

Out-of-control events associated with the statistical analysis and review of data are straight forward to identify. The Analyst generating the data is responsible for checking the results against the established limits. Any deviations are immediately addressed. If data are outside accepted limits, the Analyst immediately notifies the responsible Section Supervisor. If the situation can not be corrected to prevent an out-of-control condition, the Section Supervisor shall notify the Operations Manager and the Quality Assurance Officer. The Operations Manager and Group Supervisors are responsible for identifying the source of the problem and initiating corrective action. Completion of corrective action should be evidenced by the return of data to prescribed acceptable limits.

Events which do not cause an immediate obvious effect on data quality are more difficult to identify. Such events could be samples stored at an incorrect temperature or held beyond prescribed holding times, or improper maintenance of records. Everyone in the laboratory is responsible for reporting "system" problems. Analysts should report out-of-control events to their Group Supervisor, who should then in turn report the situation to the Operations Manager. Corrective action is again the responsibility of the Operations Manager and the Group Supervisors. They shall review and approve the action taken.

If an out-of-control event does occur during analysis, for instance an LCS recovery falls outside the expected range, the analyst must describe on the corrective action report the event, the investigative and corrective actions taken, the cause of the event, and notify the QA Officer. In some cases, investigation of an out-of-control event will reveal no problems. In such cases, only the event and the investigative action is recorded.

The investigative action taken is somewhat dependent on the analysis and the event. However, listed below is a progression of steps which may be taken to find the cause of an out-of-control event:

- Check calculations to ensure there are no errors
- Check standard and spiking solutions for degradation or contamination
- Check instrument performance

If the problem is with the standards or instrument performance, the analyst must recalibrate or retune the instrument before reanalyzing the sample extracts affected. If the out-of-control condition is still not remediated, the samples may require reextraction and reanalysis or data qualification.

It is occasionally necessary to qualify data when the accompanying quality control data are not within established performance criteria. The qualifying of data alert the data end user to the fact that the analysis was, to some degree, flawed and that the precision and accuracy of the data produced may not fulfill the data quality objectives (DQOs) for that particular project. Based on the project DQOs, analytical data with qualifiers may not be appropriate for the intended use.

15.2.1 Volatile Organic Analyses

Method Blanks

If target compounds are detected in the method blank above the detection limit (or reporting limit if different from the detection limit) (above 5 times the detection and/or reporting limit for methylene chloride, acetone, toluene, and 2-butanone) the corrective action consists of checking the calculations, reanalyzing the blank, qualifying the associated sample data, and investigating the source of the problem to implement corrective action for the future. When target list compounds are detected in a method blank, the following condition applies:

- When any target compound is detected in a method blank above the action levels listed earlier, but not in associated samples, then no qualifier is applied.

Surrogates

The % recovery of each surrogate is calculated for each sample, blank, and LCS. Corrective action is taken whenever one (or more) surrogate recovery is outside the acceptance criteria. The following corrective actions are taken when required as stated above:

- Check calculations to assure there are no errors;

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- Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
 - If instrument failure is indicated, reanalyze the sample;
 - If a method blank surrogate is outside of acceptance criteria, then the problem must be corrected before proceeding with sample analysis. This may include reanalysis, reextraction or recalibration;
 - If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded with the note "surrogate diluted out".
 - If all QC associated with the sample is within acceptance limits (the method blank surrogate recovery and LCS spike recovery), the problem may be attributed to a matrix effect. To identify the matrix as the problem, reanalyze the sample. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative.

Laboratory Control Samples

The % recovery of each spiked analyte in the Laboratory Control Sample (LCS) is calculated. Corrective action is taken whenever the % recovery is outside the established acceptance criteria for that analyte. The following corrective actions are taken when required as stated above:

- Check calculations to assure there are no errors;
- Check internal standard and spiking standard solutions for degradation, contamination, etc., and check instrument performance;
- Reanalyze samples associated with a failed LCS, if available;
- If that does not correct the problem, then the data is reported and a qualifying statement included in the report narrative.

For Matrix Spike and Matrix Spike Duplicates, if all QC associated with a sample is within acceptance limits (method blank and LCS spike recoveries), the problem may be attributed to a matrix effect. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative as appropriate.

Calibration

For an initial 5 point calibration curve to be valid, the % relative standard deviation of the individual relative response factors (RRF) for the Calibration

Check Compounds (CCC) shall be less than or equal to 30%. If this criteria is not met, then the calibration curve shall be reanalyzed.

For continuing calibration checks to be valid, the relative response factor for each of the System Performance Check Compounds (SPCC) should be at least 0.300 (0.250 for Bromoform) and the RRF for each of the CCC should be $\leq 20\%$ different from the average RRF from the initial calibration. If these criteria are not met, then the following corrective actions should be taken:

- Check internal standard and standard solutions for degradation, contamination, etc.,
- Check instrument for contamination at the injection port inlet and front end of the column;
- If no source of the problem is identified, then a complete 5 point initial calibration must be performed.

The SPCC and CCC for Volatiles are:

<u>SPCC</u>	<u>CCC</u>
Chloromethane	Vinyl Chloride
1,1-Dichloroethane	1,1-Dichloroethene
Bromoform	Chloroform
1,1,2,2-Tetrachloroethane	1,2-Dichloropropane
Chlorobenzene	Toluene
	Ethylbenzene

15.2.2 Semivolatile Organic Analyses

Method Blanks

If target compounds are detected in the method blank above the detection limit (or reporting limit if different from the detection limit) (above 5 times the detection limit and/or reporting for phthalate esters) the corrective action consists of the following:

- Checking the calculations;
- Reanalyzing the blank;
- Flagging the associated sample data;
- Investigating the source of the problem to implement corrective action for the future.

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When target list compounds are detected in a method blank, the following data condition applies:

- When any target compound is detected in a method blank above the action levels listed earlier but not in associated samples, then no flag is applied.

Surrogates

The % recovery of each surrogate is calculated for each sample, blank, and standard. Corrective action is taken whenever one (or more) surrogate recovery from either the base/neutral or acid fraction is outside the acceptance criteria. The following corrective actions are taken when required as stated above:

- Check calculations to assure there are no errors;
- Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
- If instrument failure is indicated, reanalyze the sample;
- If more than one method blank surrogate is outside of acceptance criteria or if one surrogate yields less than 10% recovery, then the problem must be corrected before proceeding with sample analysis. This may include reanalysis, reextraction or recalibration;
- If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded with the note "surrogate diluted out";
- If all QC associated with the sample is within acceptance limits (the method blank surrogate recovery and LCS spike recovery), the problem may be attributed to a matrix effect. If any one surrogate yields less than 10% recovery or if more than one surrogate in a fraction fails, reanalyze the sample to demonstrate matrix interference. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative.

Laboratory Control Samples

The % recovery of each spiked analyte in the Laboratory Control Sample is calculated. Corrective action is taken whenever recovery is outside the acceptance criteria. The following corrective action is taken when required as stated above:

- Check calculations to assure there are no errors;
- Check internal standard and spiking standards solutions for degradation, contamination, etc., and check instrument performance;

-
- Reanalyze all associated samples, if available;
 - If that does not correct the problem, then the data is reported and a qualifying statement regarding the laboratory control sample is included in the report narrative.

For Matrix Spike and Matrix Spike Duplicates, if all QC associated with a sample is within acceptance limits (the method blank and LCS/LCS dup spike recovery), the problem may be attributed to a matrix effect. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative as appropriate.

Calibration

For an initial 5 point calibration curve to be valid, the % relative standard deviation of the individual relative response factors (RRF) for the Calibration Check Compounds (CCC) should be less than or equal to 30%. If this criteria is not met, then the calibration curve should be reanalyzed.

For continuing calibration checks to be valid, the relative response factor for each of the System Performance Check Compounds (SPCC) should be at least 0.050 and the RRF for each of the CCC should be $\leq 20\%$ different from the average RRF from the initial calibration. If these criteria are not met, then the following corrective actions should be taken:

- Check internal standard and standard solutions for degradation, contamination, etc.,
- Check instrument for contamination at the injection port inlet and front end of the column;
- If no source of the problem is identified, then a complete 5 point initial calibration must be performed.

The SPCC and CCC for Semivolatiles are:

SPCC

n-Nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

CCC

Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
n-Nitroso-di-phenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

15.2.3 Gas Chromatography Analyses

Method Blanks

If target compounds are detected in the method blank above the detection limit (or reporting limit if different from the detection limit) the corrective action consists of the following:

- Checking the calculations;
- Reanalyzing the blank;
- Flagging the associated sample data;
- Investigating the source of the problem to implement corrective action for the future.

When target compounds are detected in a method blank, the following conditions apply:

- When any target compound is detected in a method blank above the action levels listed earlier, but not in associated samples, then no flag is applied.

Surrogates

The % recovery of each surrogate is calculated for each sample, blank, and standard. Corrective action is taken whenever one (or more) surrogate recovery is outside the acceptance criteria. The following corrective action is taken when required as stated above:

- Check calculations to assure there are no errors;
- Check standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
- If instrument failure is indicated, reanalyze the sample;

-
- If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded with the note "surrogate diluted out";
 - If all QC associated with the sample is within acceptance limits (the method blank surrogate recovery and LCS spike recovery), the problem may be attributed to a matrix effect. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative.

Laboratory Control Samples

The % recovery of each spiked analyte in the Laboratory Control Sample is calculated and corrective action is taken whenever recovery is outside the acceptance criteria. The following corrective action is taken when required as stated above:

- Check calculations to assure there are no errors;
- Check standard and spiking standard solutions for degradation, contamination, etc., and check instrument performance;
- If that does not correct the problem, then the data is reported and a qualifying statement regarding the laboratory control sample is included in the report narrative.

Calibration

For an initial 5 point calibration curve to be valid, the responses for each compound should be linear over the calibration range (generally linearity is defined as having an RSD \leq 20%). If this criteria is not met, then the calibration curve should be reanalyzed.

For continuing calibration checks to be valid, the % difference in the calibration factor for each compound in calibration should not exceed 15% from the initial calibration. If these criteria are not met, then the following corrective actions should be taken:

- Check standard solutions for degradation, contamination, etc.;
- Check instrument for contamination at the injection port inlet and front end of the column;
- If no source of the problem is identified, then a complete 5 point initial calibration must be performed.

15.2.4 Metals Analyses

Method Blanks

If target analytes are detected in the method blank above the reporting limit the corrective action consists of the following:

- Checking the calculations;
- Reanalyzing the blank;
- Investigating the source if the problem to implement corrective action for the future;
- Redigesting and reanalyzing the associated samples if the analyte concentration in the samples is less than 5 times the blank concentration and greater than the reporting limit.
- Data that cannot be regenerated acceptably is flagged as non-compliant.

When target analytes are detected in a method blank, the following data condition applies:

- When any target analyte is detected in a method blank above the action levels listed earlier but not in associated samples, then no flag is applied.

Laboratory Control Samples

The % recovery of each spiked analyte in the Laboratory Control Sample is calculated. Corrective action is taken whenever recovery is outside the acceptance criteria. The following corrective action is taken when required as stated above:

- Check calculations to assure there are no errors;
- Check standard and spiking standard solutions for degradation, contamination, etc., check instrument performance;
- Redigest and reanalyze samples if there is no indication of failure in any of the above;
- If that does not correct the problem, then the data is reported and a qualifying statement regarding the laboratory control sample is included in the report narrative.

For Matrix Spike, Matrix Spike Duplicates and Sample Duplicates, if all QC associated with a sample is within acceptance limits (method blank and LCS spike recoveries), the problem may be attributed to a matrix effect. Samples

exhibiting a matrix effect will be qualified and discussed in the report narrative as appropriate.

An exception to this criteria is allowed for matrix spike samples when the sample concentration exceeds the spike concentration by a factor of 4 or more. In that instance, the data is reported unqualified.

Calibration

For an initial and continuing instrument calibration to be valid, the responses for each analyte must be linear over the calibration range and the accuracy of calibration, as determined by analysis of an independent check standard, must be within $\pm 10\%$ of the true value for ICP analysis and within $\pm 20\%$ for cold vapor and graphite furnace AA analyses. If these criteria are not met, then the following corrective actions taken:

- Check standard solutions for degradation, contamination, etc.,
- Check instrument for contamination, incorrect operating conditions, etc.;
- If no source of the problem is identified, then a complete instrument calibration must be performed.

15.3 OUT-OF-STATISTICAL-CONTROL BLANK SPIKE CONTROL CHART DATA

In accordance with the requirements of certain federal programs (Navy's NFESC [formerly NEESA], HAZWRAP), blank spike control charts are maintained for all analyses performed for that program. Control chart data consist of recovery values derived from blank spike (LCS) data. Each control chart has five lines representing a statistical analysis of a set of percent recoveries from previously analyzed LCS samples:

1. arithmetic mean
2. upper warning limit (+2 standard deviations from the mean)
3. lower warning limit (-2 standard deviations from the mean)
4. upper control limit (+3 standard deviations from the mean)
5. lower control limit (-3 standard deviations from the mean)

Plotted blank spike recovery data are evaluated against these limits to monitor for out-of-control and out-of-statistical-control conditions.

15.3.1 Out-of-Control Blank Spike Recovery Data

For a specific parameter and matrix, the laboratory process is considered out of control if any one blank spike recovery value is outside the control limits on the respective blank spike control chart. Except where LCS control limits are defined by the client, excursions of the blank spike control limits equate to LCS recoveries falling out of control.

15.3.2 Out-of-Statistical-Control Conditions

For a specific parameter and matrix, the laboratory process is considered out of statistical control whenever one or more of the conditions described below is demonstrated by control chart monitoring:

1. Any three consecutive points are outside the warning limits
2. Any eight consecutive points are on the same side of the centerline
3. Any six consecutive points are such that the value of each is larger (or smaller) than its immediate predecessor
4. Any obvious cyclic pattern is seen in the points

The blank spike control charts serve as a mechanism both to note excursions from the prescribed control limits and for recognizing trends that may represent a degradation of an analytical system's quality control. They are valuable as an early warning indicator that corrective action is needed to prevent more serious loss of quality control.

15.3.3 Corrective Action for Out-of-Statistical-Control Conditions

In the course of plotting blank spike recovery data, if a condition is observed that meets one or more of the criteria described above as an out-of-statistical-control condition, corrective action must be taken. An NCM must be initiated that identifies the trend causing the out-of-statistical-control condition. Where known, the NCM should describe the root cause of the trend or excursion and the actions taken to prevent recurrence.

For some statistical trends, such as eight or more points on one side of the mean, the initial required corrective action may be no more than to continue monitoring future blank spike recovery data. If, however, the out-of-statistical-control condition persists, or if the condition recurs repeatedly, further corrective action, such as instrument maintenance or recalibration, must be performed before sample analysis may resume.

15.4 UNUSUAL OCCURRENCES

Whereas out-of-control events involve occurrences outside of pre-established acceptance windows, unusual occurrences are more subjective and involve incidents which may be compliant with the assessment criteria but still warrant investigation. Many of these investigations will be the result of the professional judgement of the analyst, auditor or data reviewer that the analysis was not typical or reasonable. Another example of this type of investigation is an inquiry or questioning of data received from a client or from the results of performance evaluation samples.

Date: 12/22/95

Section 15

Revision 0.01

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**FIGURE 15.1
Example**

**PACE CORRECTIVE ACTION REPORT
Revision 2 - 3/94**

CLP_ NEESA_ SW846_ NPDES_ Drinking water_ AFCEE_ ACOE_

Sample ID Number(s) Involved and QC batch #: _____

Type of Event: Out-of Control Event Attachments Unusual Occurrence

Disposition of Samples: Reprepped Reanalyzed Narrated

1) Description of Event:

- 1. LCS Failure Contamination Failure
- 2. Blank Contamination Failure
- 3. Poor Precision
- 4. Prep Error
- 5. Hold Time
- 6. Login Error
- 7. Detection Limit
- 8. Calibration Failure
- 9. Retention Time Window
- 10. Linearity
- 11. Sample
- 12. Surrogate
- 13. Matrix Spike Failure
- 14. Other (Describe Below)

2) Discussion of Known or Suspected Cause: _____

3) Corrective Action(s) Taken (include date, person and action): _____

4) Return to Control: _____

Initial and Date below please

1) Reported by ____/____/____ 2) Corrective Action Taken By: ____/____/____ 3) Corrective Action Approved ____/____/____
4) Documented Return to Control ____/____/____ 5) Supervisor ____/____/____ 6) QA ____/____/____

16.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The objective of the Pace quality assurance program is to ensure that an operational system is in place which enables management to determine the quality of all data produced within the laboratory system. An essential component of the system is the communication pathways and feedback mechanisms which ensure that management obtains quality information promptly and consistently. To achieve this objective, Pace employs informal and formal reporting processes to ensure that management is informed of operational quality. This information enables Pace to take corrective action promptly when required. Reporting occurs at the following frequency.

- Daily meetings to discuss possible quality assurance problems and proposed solutions.
- Weekly meeting with upper management to discuss laboratory performance, upcoming audits, certification programs, and past audit performances.
- Quarterly written status reports to upper management; inclusion of all quality assurance concerns and pertinent laboratory issues.
- As required, internal departmental audit reports with observations and suggested corrective action procedures.

The Quality Assurance Officer and Quality Assurance Auditor are responsible for preparing reports to management indicating effectiveness of the laboratory Quality Assurance Program.

16.1 QUALITY ASSURANCE AUDITOR

Results of audits performed by the QA staff are detailed in formal, written audit reports. These reports are distributed to the audited personnel, section supervisor, Laboratory Operations Manager, QA Officer, and General Manager for review and appropriate action. These and other QA-related reports are distributed as produced, with no set schedule.

Auditor reports will include, but not be limited to:

- Results of internal laboratory review activities
- Results of internal data review activities
- Results of Proficiency Evaluation studies
- Results of state certification applications
- Summary of holding time exceedence and data qualification
- Method detection limit study status

To demonstrate management review, the audit report will contain a page which will be signed and dated by the QA Officer and General Manager acknowledging that they have received the report and have reviewed its contents, and taken the necessary action dictated by their position.

16.2 QUALITY ASSURANCE OFFICER

The Quality Assurance Officer will issue a report of QA activities and findings on a regular basis to the General Manager. The status report will include:

- Results of internal systems or performance audits
- Corrective Action recommendations
- Discussion of QA issues raised by laboratory users
- Results of third party or external audits
- Status of laboratory certifications
- Other significant events
- Performance Evaluation Sample Results

16.3 MANAGEMENT REVIEW OF THE QUALITY ASSURANCE PROGRAM

Review of the appropriateness and adequacy of the Quality Assurance Program is ongoing. At anytime, any laboratory employee, through the Laboratory Operations Manager, may present recommended changes to the Quality Assurance Officer.

During system audits, the Quality Assurance Program should be discussed. The audit report will document recommendations made by either the Laboratory Operations Manager or the auditor for revision.

16.4 QUARTERLY QUALITY REPORTS TO MANAGEMENT

Quarterly reports are provided by the Quality Assurance Office staff to the Corporate Quality Office and the General Manager. The report summarizes quality assurance activities including details of corrective actions recommended or implemented, internal and external audit results, status of performance evaluation samples, certification status, and the status of internal procedure (evidentiary and technical) documents.

17.0 SUMMARY OF REVISIONS

On November 10, 1995, a business transaction was consummated which created a new company, Pace Analytical Services, Inc., consisting of seven laboratories of PACE Incorporated. At the time of the transaction, in order to ensure an efficient transition to forming the new company, quality systems (e.g., QA plan, SOPs, etc.) which were previously in place at each of the laboratories remained intact.

This document describes a new consolidated quality assurance program including (as appropriate) key elements of each facility's previous quality control practices. As such, this document has been designated as Revision 0.01, representing the first revision of the former QA plan of PACE Incorporated.

17.1 REVISION DESIGNATION

Revision numbers are designated by an integer followed by two decimal places. At a minimum, this document will be reviewed in its entirety on an annual basis. Annual, document-wide review/revisions are tracked by incrementing the integer by one (e.g., Rev. 0.00 issued 5/22/95 will be revised no later than 5/22/96 and designated as Rev. 1.00). The decimal places are used to track individual section or page revisions which occur in the interim between annual revisions. For example, when a section is initially revised independently of any other, the revision number indicated in the header will be listed as __.01 while the unrevised sections would remain at __.00. Subsequent revisions of the same section or page would incrementally increase the decimal designation (e.g., 0.02, 0.03, 0.04, etc.) The cover page revision number will always reflect the total number of individual revisions performed between annual revisions. At the time of the annual revision, all individual section and/or page revision numbers are returned to an initial __.00 designation, which initiates a new overall revision number.

17.2 SUMMARY OF REVISIONS

This section will provide a historical chronology of all future revisions. The listing will contain the date of the revision, the section or page revised and the new revision number.

National Certifications/Programs

Programs & Certifications	Asheville	Charlotte	Houston	Kansas City	Minnesota	New Orleans	N. California
EPA Contract Laboratory Program (CLP) Experience				●	●	●	
AFCEE Installation Restoration Program			▲	●	●		▲
Missouri River Division, U.S. Army COE				●	●	●	▲
Army Environmental Center (AEC)					●		
NFESC					●	●	
HAZWRAP					●		
Department of Agriculture Soil Import Permit			●		●	●	●
American Association of Laboratory Accreditation (A2LA)						▲	

● Established

▲ Pending or Interim

Kansas City Facility Information

Laboratory Location: Kansas City	Address: 9608 Loiret Boulevard Lenexa, KS 66219	Tel: (913) 599-5665 Fax: (913) 599-1759
General Manager: Duane Boline, Ph.D.	Marketing Contact: John Gerken	Quality Assurance Officer: Neal Hudson
Personnel Overview: Number of Personnel 45 Technical Personnel 32 Support Personnel 13 Advanced Degrees 3 BS 14	<ul style="list-style-type: none"> • 17,500 ft² facility, providing full organic and inorganic analyses • Sampling services for: <ul style="list-style-type: none"> - ground water - waste water - soil • Large project capacity 	
Major Instrumentation:		
10 Gas Chromatographs	1 Flame Atomic Absorption Spectrophotometer	
6 GC/MS Systems	2 Inductively Coupled Plasma (ICP) Emission Systems	
1 IR Spectrophotometer	1 Mercury Analyzer	
3 AA Graphite Furnaces	1 Gel Permeation Chromatography System	
Certifications, Contracts & Approvals		Expiration/Renewal Dates/Status
Air Force Center for Environmental Excellence/Installation Restoration Program (AFCEE-IRP)		Current
Missouri River Division of the U.S. Army Corps of Engineers.....		1/95
California: Hazardous Waste/Wastewater.....		8/95
Colorado: Drinking Water		4/96
Kansas: Drinking Water/Solid & Hazardous Waste/Wastewater		4/95
Michigan: Drinking Water		6/94
North Dakota: Drinking Water/Wastewater/Solid & Hazardous Waste		4/95
Oklahoma: Drinking Water/Wastewater/Solid & Hazardous Waste		5/95