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***INFORMAL REPORT***

DATA COLLECTION QUALITY ASSURANCE PLAN  
FOR TAN GROUNDWATER RCRA FACILITY INVESTIGATION

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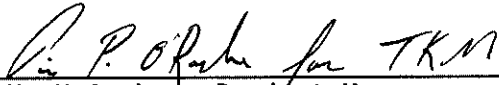
DATA COLLECTION QUALITY ASSURANCE PLAN  
FOR TAN GROUNDWATER  
RCRA FACILITY INVESTIGATION

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DATA COLLECTION QUALITY ASSURANCE PLAN  
FOR TAN GROUNDWATER RCRA  
FACILITY INVESTIGATION

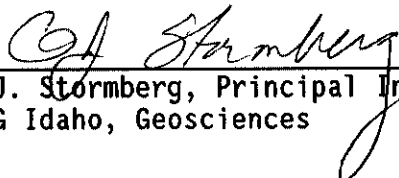
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DATA COLLECTION QUALITY ASSURANCE PLAN  
FOR TAN GROUNDWATER  
RCRA FACILITY INVESTIGATION

1. INTRODUCTION

The objective of the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) is to thoroughly evaluate the nature and extent of the release of hazardous waste and hazardous constituents and to gather all necessary data to support a Corrective Measures Study. During the course of the investigation at the Test Area North (TAN) of the Idaho National Engineering Laboratory (INEL), laboratory and field data will be used to determine the extent of the migration of hazardous constituents hydrocarbons from the Technical Support Facility (TSF) Injection Well. Therefore, the primary goal of this Data Collection Quality Assurance Plan (DCQAP) is to define the procedures that will ensure the quality and integrity of the samples, accuracy and precision of the analyses, representativeness of the results, and completeness of the information. The information gathered during this RFI will enable the project team to design and implement effective corrective action measures for removal of volatile chlorinated hydrocarbons from the groundwater at TAN. This portion of Task III describes data quality objectives and procedures associated with sample collection, laboratory analysis, sample custody, initial and continuing instrument/equipment calibration, internal quality assurance (QA) checks, performance and system audits, preventive maintenance and scheduling, data quality assessment, corrective action, and applicable QA reports to management.

## 2. PROJECT DESCRIPTION

The INEL is an 890-square-mi area managed by the Idaho Operations Office of the United States Department of Energy (DOE-ID). In 1987, the U.S. Geological Survey (USGS) surveyed the groundwater wells on the INEL Site. Trichloroethylene (TCE) was detected in five wells at concentrations between 0.5 and 7.7  $\mu\text{g/L}$ , and in the TSF Injection Well at 35,000  $\mu\text{g/L}$ . Trichloroethylene was detected in two wells (TAN-1 and TAN-2) which supply drinking water to the TSF at 7.7 and 5.4  $\mu\text{g/L}$ , respectively. Tetrachloroethene (PCE) has also been detected in TAN-1 at 1.4  $\mu\text{g/L}$ .

The suspected source of the groundwater contamination at TSF is the TSF Injection Well, where waste water was disposed of from 1953 to 1972. A soil gas survey and surface soil investigation of the solid waste management units at TSF indicate that no significant soil contamination from TCE or PCE has occurred. Therefore, this investigation has been developed to identify and quantify the extent and magnitude of the TCE and groundwater contamination from the TSF Injection Well. The investigation will also be useful in identifying, developing, and implementing appropriate remedial action measures. The following components will be part of the investigation:

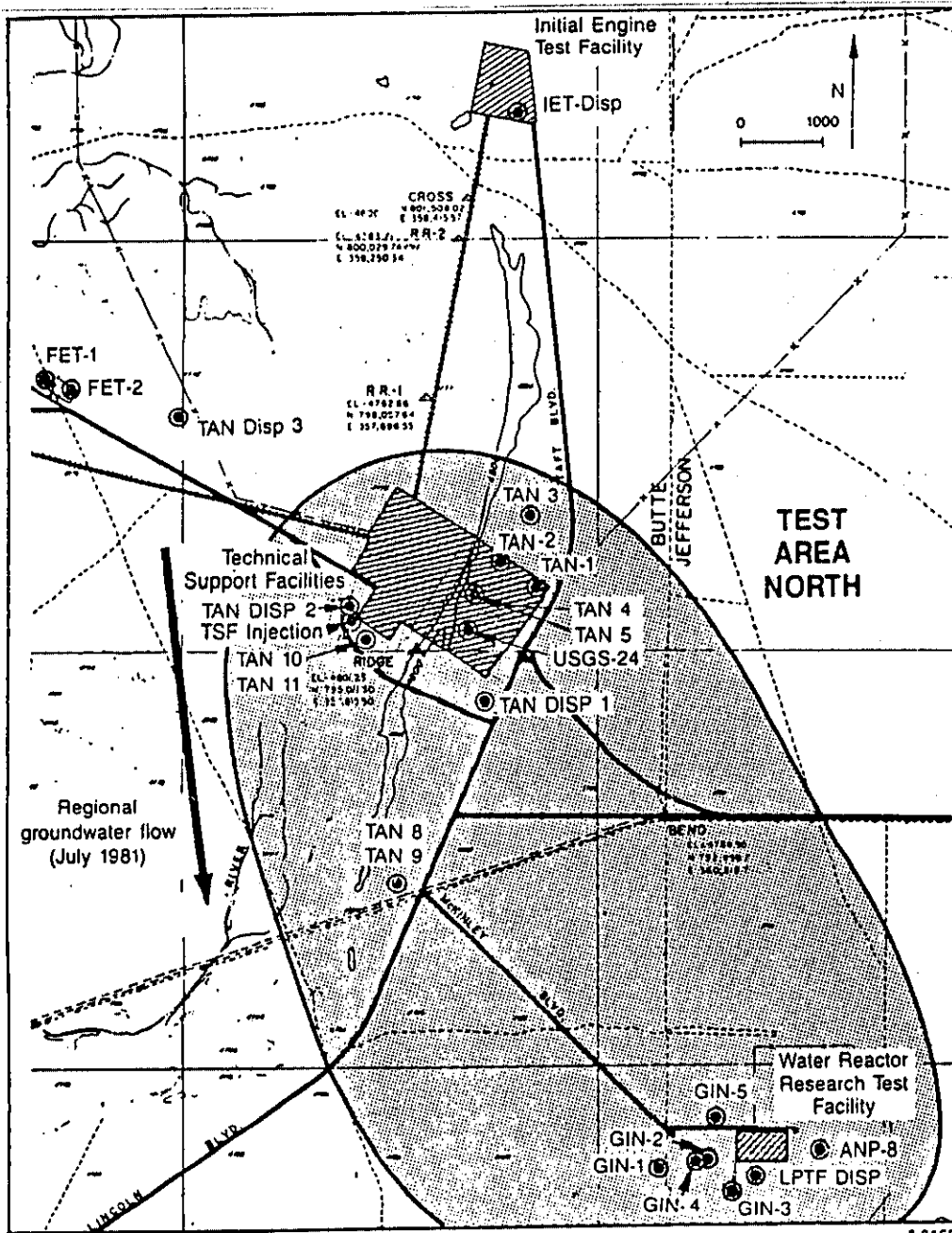
- Locate the source(s) of the release(s) of contaminants [e.g., regulated transportation, shipment, and disposal (T/S/D) units, solid waste management units, and other source areas]
- Characterize the nature and extent of contamination. Define pathways and methods of TCE and PCE migration, including the media, extent, direction, speed, complicating factors influencing movement, concentration profiles, etc.
- Identify areas and populations threatened by releases from the facility

- Determine short- and long-term, present and potential threats to human health and the environment
- Identify and implement interim measures to abate further spread of contaminants
- Identify, develop, and implement corrective measures to prevent and remedy releases of TCE from TAN
- Design a program to monitor the implementation, maintenance, and performance of any interim or final corrective measures to ensure that human health and the environment are protected.

Figure 2-1 shows the sampling locations (i.e., TAN-3, TAN-4, TAN-5, TAN-8, TAN-9, TAN-10, and TAN-11) projected for the RFI, and Table 2-1 summarizes the historical data and the planned activities for the RFI.

The RFI activities that will be conducted to achieve these objectives include:

- Install seven monitoring wells and collect groundwater samples
- Collect 13 groundwater samples from existing wells
- Conduct pumping tests at the TAN-1 and TAN-2 production wells and the ANP-8 well if necessary
- Drill six auger holes in surficial sediments above the basalt and collect samples
- Continue to monitor effluent from TSF to the TSF Disposal Pond on a monthly basis for chlorinated hydrocarbons



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Figure 2-1. Projected sampling locations for the RFI. Shaded area indicates assumed extent of TCE groundwater contamination above 5 µg/L.

TABLE 2-1. SUMMARY OF HISTORICAL DATA AND PROPOSED ACTIVITIES		
Well No.	Historical Data	Proposed Activities
TSF-Injection Well	<u>TCE</u> • 930 - 35000 µg/L  <u>PCE</u> • 50 - 170 µg/L	<ul style="list-style-type: none"> <li>• Collect samples at wells TSF Injection and TAN Disp-1, -2, and -3</li> </ul> <u>Initial resampling:</u> - Volatile organics - Major anions - Trace metals - Tritium  <u>Periodic monitoring</u> - Volatile Organics - Trace Metals
USGS-24	<u>TCE</u> • 840 µg/L  <u>PCE</u> • 38 µg/L	<ul style="list-style-type: none"> <li>• Collect groundwater samples at USGS-21, -24, and -26</li> </ul> <ul style="list-style-type: none"> <li>• <u>Initial resampling</u> <ul style="list-style-type: none"> <li>- Volatile organics</li> <li>- Major anions</li> <li>- Trace metals</li> <li>- Tritium</li> </ul> </li> <li>• <u>Periodic monitoring</u> <ul style="list-style-type: none"> <li>- Volatile organics</li> <li>- Trace metals</li> </ul> </li> </ul>
ANP-8	<u>TCE</u> • 4 - 7 µg/L  <u>PCE</u> • 2 - 4 µg/L	<ul style="list-style-type: none"> <li>• Collect groundwater samples from Wells ANP-9 and -10</li> </ul> <ul style="list-style-type: none"> <li>• <u>Initial resampling</u> <ul style="list-style-type: none"> <li>- Volatile organics</li> <li>- Major anions</li> <li>- Trace metals</li> <li>- Tritium</li> </ul> </li> <li>• <u>Periodic monitoring</u> <ul style="list-style-type: none"> <li>- Volatile organics</li> <li>- Trace metals</li> </ul> </li> </ul>

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TABLE 2-1. (continued)		
Well No.	Historical Data	Proposed Activities
TAN-1	<u>TCE</u> • 3.2 - 7.7 µg/L  <u>PCE</u> • 0.5 - 2.0 µg/L	<ul style="list-style-type: none"> <li>• Drill and complete as monitoring wells TAN Wells -3, -4, -5, -8, -9, -10, -11</li> </ul> <u>Initial resampling</u> - Volatile organics - Major anions - Trace metals - Tritium  <u>Periodic monitoring</u> - Volatile Organics - Trace Metals
TAN-2	<u>TCE</u> • 1.6 - 5.4 µg/L  <u>PCE</u> • 0.4 - 2 µg/L	<ul style="list-style-type: none"> <li>• Drill and complete as monitoring wells TAN Wells -3, -4, -5, -8, -9, -10, -11</li> <li>• <u>Initial resampling</u>                - Volatile organics                - Major anions                - Trace metals                - Tritium</li> <li>• <u>Periodic monitoring</u>                - Volatile organics                - Trace metals</li> </ul>
FET-1	<u>TCE</u> • 0.2 µg/L  <u>PCE</u> • 0.2 µg/L	<ul style="list-style-type: none"> <li>• No further activities</li> </ul>
FET-2	<u>TCE</u> • 0.2 µg/L  <u>PCE</u> • 0.2 µg/L	<ul style="list-style-type: none"> <li>• No further activities</li> </ul>

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TABLE 2-1. (continued)		
Well No.	Historical Data	Proposed Activities
IET-DISP	<u>TCE</u> • 1.3 µg/L  <u>PCE</u> • 1.2 µg/L	<ul style="list-style-type: none"> <li>• Collect groundwater samples at IET-DISP</li> <li>• <u>Initial sampling</u> <ul style="list-style-type: none"> <li>- Volatile organics</li> <li>- Major anions</li> <li>- Trace metals</li> <li>- Tritium</li> </ul> </li> <li>• <u>Periodic monitoring</u> <ul style="list-style-type: none"> <li>- Volatile Organics</li> <li>- Trace Metals</li> </ul> </li> <li>• No further activities at IET-DISP under this program</li> </ul>

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- Compile existing data obtained from the samples collected at the four solid waste management units in 1988
- Install a treatment system at the TSF Injection Well
- Monitor the efficiency of the treatment unit for the TSF Injection Well
- Install a sparging system at the TAN-1 and TAN-2 production wells.

### 3. ORGANIZATION AND RESPONSIBILITIES

EG&G Idaho is the site contractor responsible for operations at the sampling unit. DOE-ID has primary responsibility and authority for the RCRA/CERCLA (Comprehensive Environmental Response, Compensation & Liability Act) United States Environmental Protection Agency (EPA) Regulatory Compliance activities at TAN. Figures 3-1 and 3-2 identify the responsible organizations within EG&G Idaho and the appropriate contacts for various aspects of the sampling plan.

The Data Collection Quality Assurance (QA) Plan will be reviewed and approved by EPA Region X. A copy of the approved QA Project Plan will be distributed by the Project Manager to each person who has a major responsibility for the measurement data quality.

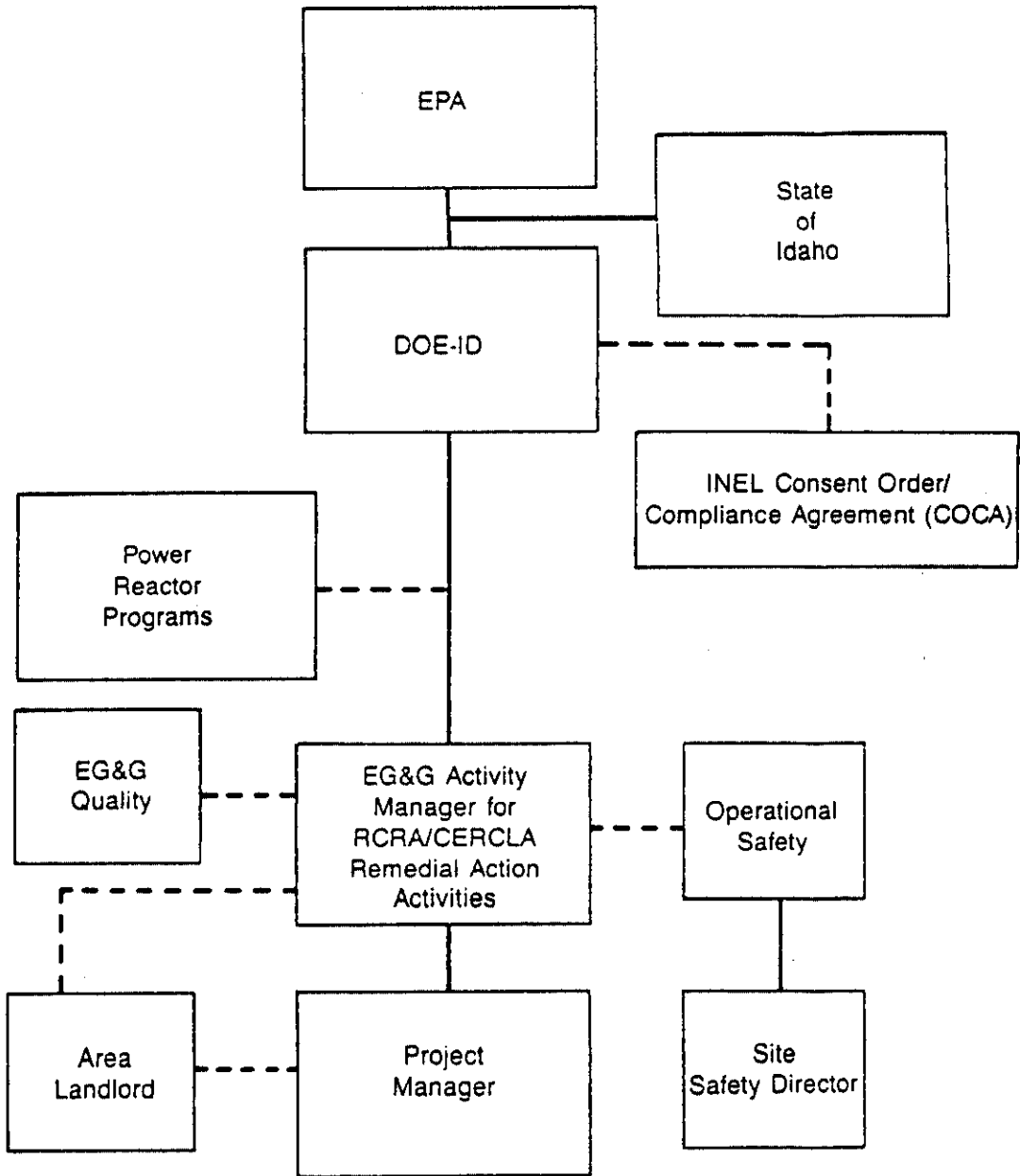
#### 3.1 Activity Manager

The Activity Manager will have overall responsibility for direction of the project and reporting to DOE-ID or to the EPA.

#### 3.2 Project Manager

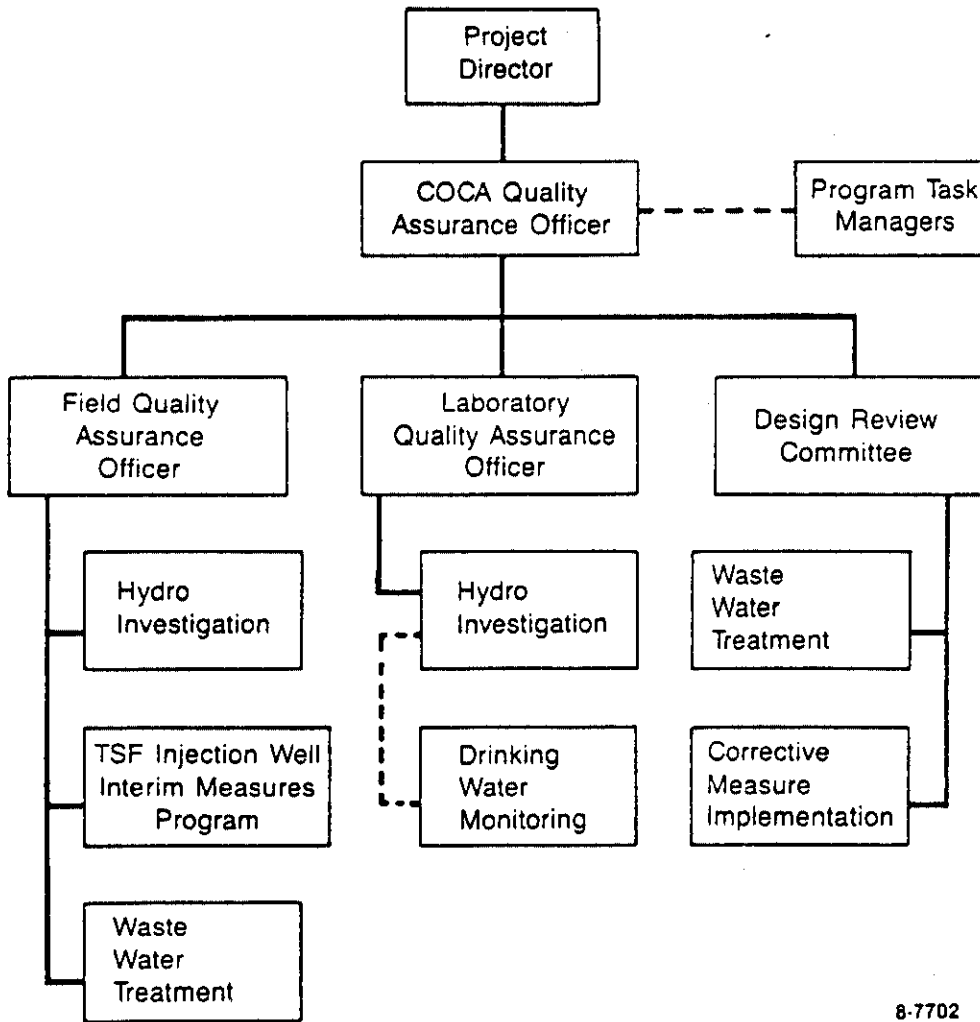
The Project Manager is responsible for coordination, technical consistency, and review of the project, which includes the following:

- Guidance in solving problems
- Site team integrity throughout the period of performance
- Coordination among site teams and support personnel to ensure consistency of performance



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Figure 3-1. Organization chart for Sampling Plan activities.



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Figure 3-2. Flow chart of sampling organization.

- Final approval and review of work plans, project deliverables, schedules, contract changes, and manpower allocations
- Completion approval for each work element of each task
- Management work plan preparation, including approval of monitoring site locations, chemical analysis parameters, schedules, and manpower allocation.

### 3.3 COCA Quality Assurance Manager

The Consent Order/Compliance Agreement (COCA) Quality Assurance (QA) Manager has the following responsibilities:

- Ensure that adequate QA documentation is provided
- Ensure that all QA problems are resolved expeditiously and brought to the attention of the Project Manager
- Provide guidance to the contractors during field activities and for submitting blind control samples
- Review all project analysis data for compliance with QA objectives and for technical accuracy
- Validate data for inclusion in the final report
- Conduct field and laboratory audits.

### 3.4 Program Task Manager

The Program Task Manager is primarily an administrator when not participating in the field investigation as Field Team Leader. The Program Task Manager is responsible for:

- Evaluating field programs from a QA standpoint
- Ensuring that all field activities are conducted in accordance with the RFI Plan for TAN Groundwater
- Ensuring proper custody and documentation of samples
- Ensuring that all field activities meet or exceed the requirements of applicable drilling and sampling methodologies, including well installation and borings, soil/formation sampling, logging, well development, and groundwater sampling
- Ensuring that the environmental samples are representative of the formation investigated and ensuring that sample integrity is maintained.

### 3.5 Field Quality Assurance Officer

The Field QA Officer will monitor the performance of field operations. The primary responsibility of the Field QA Officer will be to detect and report any deviation from QA protocol at an early stage so that the validity of the sampling program will not be jeopardized.

### 3.6 Laboratory Quality Assurance Officer

The Laboratory QA Officer determines if the analytical results are within the range of acceptability, if the laboratory has performed control sample analyses properly, and if instrument calibration is being maintained.

Additionally, the Laboratory QA Officer informs the Laboratory Manager and the Project Manager of any nonconformance and confers with the Project Manager regarding decisions to discard and reanalyze sample lots.



#### 4. QUALITY ASSURANCE OBJECTIVES

Comprehensive QA objectives have been developed to provide guidelines for all field and laboratory operations. The goal is to produce data of known and sufficient quality to fully assess the impact of past and present hazardous waste practices; to identify, quantify, and delineate the extent of any contamination; and to develop preliminary alternative action plans. During the course of this investigation, all activities and analyses will be conducted using standard procedures so that known and acceptable levels of accuracy, precision, completeness, representativeness, and comparability are documented. The results produced using established methodology and standard operating procedures will be reproducible at all levels.

A QA program requires the identification and quantification of all sources of error associated with each step in a monitoring program so that the resulting data will be of known quality. Factors considered as sources of error or variance include those associated with sample collection, sample preparation, extraction, and analysis. Thus, QA requires systematic controls of all phases of the measurement process. This includes both field and laboratory procedures to address sampling design and methods as well as accuracy, precision, representativeness, comparability, and completeness.

##### 4.1 Accuracy

Accuracy is a measure of the closeness of an individual measurement to the true value. Accuracy includes both precision and recovery and can be expressed as a percent recovery or percent bias interval.

Accuracy refers to the degree of difference between measured or calculated values and the true value. The closer the numerical value of the measurement comes to the true value or actual concentration, the more accurate the measurement. Analytical accuracy is expressed as the percent

of recovery of an analyte that has been added to the environmental sample at a known concentration before analysis. The equation used to calculate percent recovery is presented below:

$$\text{Accuracy} = \text{Percent recovery} = \frac{A_r - A_o}{A_f} \times 100\%$$

where:

$A_r$  = total amount found in spike sample

$A_o$  = amount found in unspiked sample

$A_f$  = amount added to sample.

The accuracy of simple yet fundamental field analyses is difficult to assess quantitatively. However, sampling accuracy can be maximized by the adoption and adherence of a strict quality assurance program. Specifically, all procedures must be documented as standard protocol, and all equipment and instrumentation must be calibrated properly and well maintained. Trip blanks and field blanks will be included in all sample batches to ensure that all samples represent the particular site from which they were taken; these blanks will also be used to assess any cross-contamination that may have occurred. In addition to equipment operations and standard operating procedures, a high level of accuracy can be maintained by frequent review of field procedures. In this manner, deficiencies can be quickly documented and corrected. Specific QA objectives for the RCRA Facility Investigation at TAN are presented in Sections 1.10 and 1.13.

#### 4.2 Precision

Precision refers to the reproducibility, or degree of agreement, among replicate measurements of the same analyte or parameter. Specifically, precision is a quantitative measure of the variability of a group of measurements compared to their average value. The closer the numerical values of the measurements come to each other, the more precise the measurement. This allows immediate comparison of precision of different results under the same method. Analytical precision is expressed as a percentage of the difference between results of duplicate samples for a given analyte. Relative percent difference is calculated as follows:

Precision = Relative Percent Difference

$$= \frac{C_1 - C_2}{(C_1 + C_2)/2} \times 100\%$$

where:

$C_1$  = concentration of the analyte in the sample

$C_2$  = concentration of the analyte in the duplicate/replicate.

NOTE: Collection of multiple samples will permit an estimation of contaminant variability and a statistical estimation of precision confidence limits.

#### 4.3 Representativeness

Representativeness is expressed as the degree to which the data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness involves the reliability of the mean and the standard deviation as measures of the amount of a parameter

present in a particular area. The precision of a representative set of samples reflects the degree of heterogeneity in the medium collected and reflects the effectiveness of the sampling program.

Representativeness is a qualitative parameter that is essential in properly designed sampling programs. The need for sample representativeness is a function of the use of the data, the matrix being sampled, the analysis conducted, and the defined population. Representativeness is maximized by ensuring that sampling locations are selected properly and that a sufficient number of investigative samples are collected. Individual grab samples at cribs, wells, and other sites may meet program objectives, but multiple samples will be collected at most locations to provide some idea of variability. Collection of multiple samples will permit an estimation of contaminant variability and statistical estimation of precision confidence limits.

#### 4.4 Completeness

Completeness is expressed as the percentage of valid data obtained from a measurement system. For data to be considered valid, they must meet all the acceptance criteria, including accuracy, precision, and any other criteria specified by the analytical method used.

Field sampling conditions are unpredictable and non-uniform. The objective of the field sampling program at TAN is to obtain samples for all analyses required at each sampling location, provide sufficient quantities of sample material to complete those analyses, and produce QC samples that represent all possible contamination situations (e.g., contamination during sample collection, storage, and transport).

The overall data quality objective for completeness at TAN is 85%. Project completeness will be calculated by the following equation:

$$\text{Completeness} = DP_v \frac{DP_t - DP_i}{DP_t} \times 100\%$$

where:

$DP_v$  = valid data points

$DP_t$  = total quality control data points

$DP_i$  = invalid data points (i.e., sum of the percent recovery or RFD values outside laboratory or project control limits and the number of contaminants detected in blank samples).

where:

- Each VOC analysis is equal to 37 data points
- Each surrogate recovery is equal to one data point
- Each metals analysis is equal to 27 data points
- Each field and inorganic parameter is equal to one data point.

For data points considered critical to the investigation, the data quality objective for completeness is 100%. Critical data points (those sites that represent a potential or immediate risk to human health or well-being) for the RFI at TAN will be determined in the field by the Project Manager prior to each applicable field activity and will be documented to the COCA Quality Assurance Manager. Samples for which the critical data points fail accuracy, precision, interference, or

contamination data quality objectives will be reanalyzed until the quality objectives are met. Sufficient sample volume will be collected to ensure that reanalysis can occur as needed.

#### 4.5 Comparability

Comparability is defined by the confidence with which one data set can be compared to another. Field and laboratory procedures greatly affect comparability. To optimize comparability, only the specific methods and protocols that have been selected or specified will be used to collect and analyze samples. By using specific sampling and analysis procedures, all data sets will be comparable through the duration of the project and at other DOE facilities, to ensure that remedial action decisions and priorities are based on a consistent database.

Use of consistent units will ensure that data are compatible. Both field and laboratory data will be expressed in Standard International Units (SIU), usually  $\mu\text{g/L}$ ,  $\text{mg/L}$  or  $\mu\text{g/Kg}$ . Radionuclides will be reported in  $\text{pCi/L}$  or comparable units, as opposed to SIUs.

## 5. SAMPLING

The sampling design for the RCRA Facility Investigation (RFI) at TAN is described in great detail in Task IV. It describes the methods that will be used to obtain additional data for developing a corrective measure alternative for TAN groundwater contaminated by volatile chlorinated hydrocarbons. Data needs were divided into four categories: environmental setting, source characterization, contamination characterization, and potential receptors. This Data Collection Quality Assurance Plan only addresses general RFI activities for Phase I of the hydrogeologic investigation. This Plan will be revised prior to any scheduled field activities to include the specific details that will be addressed in a Technical Work Plan (TWP) and accompanying Standard Operating Procedures (SOP). The TWP and SOPs will describe in detail what field activities are to be performed; what, where, and when data are to be collected; and how to perform field activities, including descriptions of the field equipment to be used. Guidelines for minimal QA/QC samples for field sampling programs are listed in Table 5-1.

### 5.1 Hydrogeologic Investigations

#### 5.1.1 Soil Sampling Methods

The Field Geologist is responsible for on-site supervision of drilling and soil sampling operations and for recording (logging) all pertinent information regarding the geologic materials penetrated during the operations.

A number of sampling techniques are available for collection of soil samples. These techniques include, but are not limited to, collecting auger cuttings; split-spoon sampling; Shelby tube sampling; Pitcher barrel, piston, or Dennison sampling; and single, double, and triple tube core

TABLE 5-1. GUIDELINES FOR MINIMAL QA/QC SAMPLES FOR FIELD SAMPLING PROGRAMS

<u>Media</u>	<u>Field Duplicates</u>	<u>Field Blank</u>	<u>Trip Blank</u>	<u>Equipment Blank</u>	<u>Background Sample</u>	<u>Inter-Lab Split Sample</u>
Aqueous	One in ten	One per day of VOC sampling	One per day of VOC sampling	As needed	Min. of two per sampling event-media	When required to meet objectives
Soil, sediment	One in ten	None	One per day of VOC sampling	As needed	Min. of two per sampling event-media	When required to meet objectives
Air	One in ten	Not available	One per day of sampling	None	Min. of two per sampling event-media	When required to meet objectives
Source material	One in ten	Not usually required				When required to meet objectives

NOTE: These numbers are intended as guidelines only; QA/QC requirements must be developed on a site-specific basis. Laboratory blanks and spikes are method specific and are not included in this table.

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barrel sampling. The most commonly used technique on hazardous waste sites is split-spoon sampling and it is discussed below.

Boreholes for sampling soils above bedrock will be advanced using a truck- or track-mounted drill rig equipped with minimum nominal 4-in. inside diameter hollow-stem augers. The drill rig will be equipped with a cathead-operated, 340-lb hammer with a 30-in. drop for drive sampling and other ancillary equipment required to install required instrumentation, surface casings, and complete the boreholes.

Augers will be advanced into the ground in 5-ft sections. The augers will be either threaded and pinned together or slot-fitted together using a keyway for alignment and then are linked by two high-strength bolts. Rotation of the augers cuts a minimum 7-in.-diameter borehole and advances the bit, allowing cuttings to ascend up the auger flights and accumulate at the ground surface. Samplers, drill and drive rods, plugs, instrumentation, well pipe, and screen, etc., are lowered to the bottom of the borehole through the hollow stem of the augers.

Soils are sampled by lowering the split-spoon sampler attached to the end of a string of drill rods, which extends from the top of the augers to the bottom of the borehole through the hollow-stem of the augers. A 340-lb hammer is attached to the top of the drill rod, and the sampler is driven into the soil at the bottom of the borehole by alternately raising the hammer on a rope, which passes around a rotating cathead, and allowing the hammer to free-fall 30 in. by suddenly releasing the tension on the rope. The sampler is then pulled up from the bottom of the borehole on the drive rods and removed from the bottom of the drive rod string.

### 5.1.2 Soil, Sediment, and Cuttings

The following is a listing of the procedures that will be followed for decontaminating soil, sediment, and cuttings samples at TAN:

- All sampling equipment, including internal components, will be decontaminated prior to use and between samples with laboratory-grade detergent and water, potable water and pesticide-grade methanol and deionized water rinses in that order
- Drilling rig(s) and tools will be thoroughly steam-cleaned before initial use and after each borehole completion
- Decontaminated equipment (i.e., sampling tools) will be allowed to air-dry, will be wrapped in a non-plastic material (aluminum foil or Teflon), and will be stored in a fashion that reduces the potential for accidental contamination
- Each sample cooler will contain a calibrated thermometer. The temperature of the cooler will be recorded each time the cooler is opened
- Containers necessary for soil samples are described in Appendix C.

5.1.2.1 Volatiles. For SW-846 method 8240 analyses, a small squat jar will be filled as full as possible. The Teflon-lined lid will be replaced, and the jar will be put in the cooler. All samples will be kept at 4°C. All volatile analysis samples will be collected independently of the extractable and inorganic portions. Sample portions for all volatile analyses from one discrete sampling location may be combined in one jar if collected at the same time.

5.1.2.2 Metals. A large squat jar will be filled as full as possible. The Teflon-lined lid will be replaced, and the jar will be put in the cooler. All samples will be kept at 4°C. Sample portions for all metals and general chemical analyses (e.g., anions) from one discrete sampling point may be combined in one container if collected at the same time.

### 5.1.3 Groundwater Sampling Methods

Upon completion, development, and hydraulic testing of the monitoring wells, groundwater samples will be collected for laboratory analyses. Prior to sampling, all wells will be developed and purged of at least five times the borehole volume. Typically, a support rig will be provided by the drilling subcontractor to develop and purge wells as required. In some cases, wells can be developed and purged using hand-operated equipment. Extracted water will be put into containers or, if permitted, will be discharged to the ground surface away from the well in such a fashion as to minimize erosion and impact of recharge to the well. Each well will be purged until the deviation between samples of pH, temperature, specific conductance, and color of the discharge have stabilized. The following criteria will be used to determine if the discharge is stabilized:

- pH: +/- 0.1 Standard Units
- Temperature: +/- 0.5°C
- Specific conductance: +/- 10 umho/cm
- Color: no visually discernable difference
- Odor: no nasally discernable difference

NOTE: Odor will not be utilized in sample locations where aromatics may pose a health and safety threat to workers.

Observations during purging operations will be recorded in the field logbook.

Groundwater samples will generally be collected using a bailer or a suitable pumping system. A bailer of sufficient size to provide a large sample, while having a small enough diameter to fit down the well, will be used. Generally, a 3-ft-long, bottom-discharge bailer constructed of Teflon will be used.

#### 5.1.4 Water

The following is a listing of the procedures that will be followed when collecting water samples:

- The procedures listed in Subsection 5.1.2 for the transfer of soil will also be observed for water, except that filtration may be required prior to filling sample containers with water that will undergo certain types of analyses
- Monofilament line or steel wire for lowering sampling equipment into the well will be dedicated for each well
- Water-level probes will be decontaminated before use in each well
- Precautions for decontaminated tool storage are also adopted from Subsection 5.1.2
- All prescribed preservation techniques will be implemented at this stage
- Containers necessary for water samples are described in Appendix B.

5.1.4.1 Purgeables (SW846, Method 8240). The following is a description of the procedure that will be followed when sampling water for gas chromatograph/mass spectrometer (GC/MS) volatiles analyses at TAN:

Sample containers will not be prerinsed with sample nor overfilled. Sufficient 1:1 hydrochloric acid will be added to two of the VOA vials to lower the pH of the water sample to less than 2 (about 4 drops). This preservation technique will be so noted on the sample label and chain-of-custody documentation. For each discrete sampling location, two 40-mL VOA vials will be filled with sample until the meniscus bulges over the rim of the vial. The cap liner will be slid across the rim of the vial (Teflon side down), and the plastic cap will be replaced. To ensure the vials contain no headspace, the vials will be inverted to check for bubbles. The vials will be placed in the cooler and kept at 4°C. If there is a headspace observed, the sample shall be discarded and a new sample obtained.

5.1.4.2 Total Recoverable Metals. The following is a description of the procedure for sampling water for total recoverable metals at TAN:

Sample containers will be prerinsed with sample media. Before sampling, approximately 2 mL of 1:1 nitric acid will be added to each sample container. This preservation technique will be so noted on the sample label and chain-of-custody. For each discrete sampling location, a 250-mL plastic bottle will be filled with sample before replacing the plastic cap. The turbidity of each sample will be noted at this time. The bottle will be placed in the cooler and kept at 4°C.

5.1.4.3 Common Anions. The following is a description of the procedure that will be followed when sampling water for common ions at TAN:

Sample containers will not be pre-rinsed with sample media nor overfilled. For each discrete sampling location, one 250-mL plastic bottle will be filled with sample media before replacing the plastic top. The bottle will be placed in the cooler and kept at 4°C.

5.1.4.4 Nitrate. The following is a description of the procedure that will be followed when sampling water for nitrate analyses at TAN:

Water samples for nitrate analysis will be collected in a separate 250-mL plastic bottle and preserved with a sufficient volume of sulfuric acid to lower the pH of the sample to 2 or less. After collection and preservation, the sample will be placed in the cooler and kept at 4°C.

5.1.4.5 Cyanide. The following is a description of the procedure that will be followed when sampling water for cyanide at TAN:

Sample containers will not be prerinsed nor overfilled. For each discrete sampling location, one 500-mL plastic bottle will be filled with sample. Two sodium hydroxide pellets (NaOH) will be dropped into the sample. The sample will be agitated to dissolve the NaOH and raise the pH of the water to 12. The bottle will be placed in the cooler and kept at 4°C.

#### 5.1.5 Replicate Soil Sampling Procedures

Replicate soil samples will be collected as a means of assessing the precision of the sampling effort at TAN. One replicate sample will be taken every eight sampling locations, or one per site or zone if that site or zone contains less than eight sampling locations. A significant problem that arises during soil sampling from a borehole is accurately duplicating the sample interval. To alleviate this problem, samples will be collected using a 2-in. split spoon sampler. This sampler generally will provide adequate sample volume so that replicate samples can be collected. The procedures for collecting and containerizing the samples are detailed below.

The volatile sample portion will be collected first to minimize compound volatilization. The soil will be split lengthwise using a Teflon

or stainless-steel spatula, and then the sample portion for volatile organic analysis will be immediately collected. Spatulas will be used to transfer the sample from the exposed core directly into the vials or jars. Replicate samples will be collected at the same time, employing the same techniques.

After the volatile organic samples have been collected, the Project Geologist will describe the sample. Once the description has been recorded, the remaining sample will be mixed on a Teflon board and evenly subdivided between the remaining sample containers. This mixing will minimize the heterogeneity of the soil samples and provide a representative replicate sample. This mixing will be used only for inorganic and nonvolatile samples, since agitation may allow escape of the volatile contaminant from the soil.

After collection, all sample bottles will be properly labeled and stored onsite in an ice chest at 4°C until they are packed and shipped via overnight delivery to the laboratory for analysis.

#### 5.1.6 Duplicate Water Sampling Procedures

Duplicate water samples will be collected as a means of assessing the precision of the sampling effort at TAN. One duplicate will be taken for every eight wells, or one per site or zone if that site or zone contains less than eight wells. Duplicates will be coded in a manner such that laboratory personnel will not recognize them as QC samples. For the purposes of this project, water samples will be designated as duplicates, even though several bailer volumes may be needed to fill the sample containers.

#### 5.1.7 Decontamination Procedures

All sampling equipment will be decontaminated before sampling activities begin, before moving sampling to another well, and after sampling

activities have been completed. The bailer will be scrubbed with laboratory-grade detergent (e.g., Liqui-Nox, Alconox) and rinsed with potable water, deionized water, and pesticide-grade methanol and deionized water, in that order. The bailer will be allowed to air dry prior to lowering it into the well. The line used to lower the bailer into the well will be replaced between wells. Water level monitoring devices and measuring tapes will be scrubbed with laboratory-grade detergent and rinsed with ASTM Type II reagent water.

Split spoon samplers will be decontaminated between sample intervals with a laboratory-grade detergent (e.g., Liqui-Nox, Alconox) and rinsed with potable water, deionized water, and pesticide-grade methanol and deionized water, in that order. These samplers will then be allowed to air dry. The procedure will prevent cross-contamination between samples. Field personnel are strongly cautioned to allow ample time for the hexane to evaporate completely. The hydrocarbon mixture that makes up pesticide-grade hexane will interfere with the analysis of GC/MS volatiles if field equipment is not dried completely between uses.

Drilling equipment (including rods, bits, tools) will be decontaminated at the decontamination area with a steam cleaner and laboratory-grade detergent (e.g., Liqui-Nox, Alconox) prior to the initiation of field activities between drilling locations.

Equipment used to dispense ASTM Type II reagent water, methanol, and hexane shall be constructed of glass and Teflon.

Plastic dispensing devices are prohibited. All sample containers provided by the subcontractor will be obtained from I-Chem of Hayward, California, a bottle contractor to the U.S.



Environmental Protection Agency (EPA) Contract Laboratory Program (CLP). The containers provided are those described in 40 CFR 136. These containers are cleaned by I-Chem in accordance with EPA protocols. Each lot of containers is analyzed in accordance with I-Chem QC requirements and is not shipped by I-Chem unless the QC requirements are met. The types of containers provided for the analytes of interest are listed in Appendices B and C along with the holding times and preservatives required for each analysis.

## 5.2 In Situ or Field Analysis

Measurements of selected physical and chemical parameters in air, soil, and water at the investigation sites will be made during multimedia sampling activities. The following subsections describe typical measurements made for field analysis of chemical properties of soil and water samples.

### 5.2.1 Air Measurements

Ambient air conditions will be measured during drilling both up- and down-wind of the drilling location to provide information on the characteristics of any volatile organic vapors at the site. In addition, conditions at the top of the borehole will be measured throughout the sampling using organic vapor analyzers or photoionization detectors.

### 5.2.2 pH Measurements

pH is an important chemical property of water and is used in conjunction with other chemical measurements in the analysis of conditions on hazardous waste sites. In addition, pH measurements will be made during monitoring well purging operations to ensure that the water to be sampled is representative of the formation. pH measurements will be taken on surface and groundwater samples but not on soil samples.

### 5.2.3 Specific Conductance

Specific conductance of water samples is used to adjust certain other chemical parameters to a base value. Specific conductance measurements will be made during monitoring well purging operations to ensure that the water to be sampled is representative of the formation.

## 5.3 Drinking Water Monitoring

### 5.3.1 Drinking Water Sampling Methods

The requirements for the monitoring of drinking water at the INEL are contained in several documents: Federal Safe Drinking Water Act; 40 CFR 141-143; Idaho Regulations for Public Drinking Water Systems; IDAPA 16.01.8000-8900; and various federal and state documents that establish new regulations. The regulations are divided into categories: (a) Primary Drinking Water Standards, which discuss inorganic, organic, radiological, and bacteriological contamination, and (b) Secondary Drinking Water Standards, which discuss additional measurements. The most important new regulation now affecting the INEL is found in the Federal Register, published July 8, 1987, which covers additional requirements of the Safe Drinking Water Act for Volatile Organic Compounds (VOC).

Personnel will collect samples from drinking water systems utilizing written sampling plans. These personnel will receive formal training in sampling procedures and personal protection. The samples will be properly preserved, retained, and analyzed according to written procedures published by the EPA. Analysis will be performed by outside contractors. The USGS periodically monitors the wells and drinking water for some of the secondary drinking water contaminants. These samples are drawn and analyzed utilizing USGS personnel and procedures.

When sampling water from production and monitoring wells, the well pump must first be turned on and the pressure surge must be observed, if possible, to determine when the water has reached the wellhead. At this time, a grab sample will be collected for field measurements (pH, conductivity, temperature, and any other measurement requested for specific sampling). Personnel must continue to collect samples for field measurements at regular intervals until at least five borehole volumes of water have been pumped and the field measurements of pH, specific conductance, and temperature have stabilized, indicating that the well has been adequately purged.

After the well has been purged and field measurements have stabilized, samples will be obtained, starting with volatile organic analysis (VOA) samples. If samples for other constituents are desired, those samples will be collected after the VOA collection. After the last sample is collected, a grab sample will be collected to use for field measurements, and the ending flow meter reading will be recorded. Samples will be preserved and sealed in containers and transported to the analytical laboratory as quickly as possible. Table 5-2 lists the required containers and preservation techniques for water samples.

Records of all samples taken for potable water analysis shall be retained. Specifically, the following information will be kept:

- Records of coliform analysis for five years
- Records of chemical analysis for 10 years
- Date, place, time of sampling, and name of the person collecting the sample

TABLE 5-2. REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Name	Container <sup>a</sup>	Preservation	Maximum Holding Time
<u>Bacterial Tests:</u>			
Coliform, fecal and total	P, G	Cool, 4°C, 0.008% $\text{Na}_2\text{S}_2\text{O}_3$	6 hours
Fecal streptococci	P, G	Cool, 4°C, 0.008% $\text{Na}_2\text{S}_2\text{O}_3$	6 hours
<u>Inorganic Tests:</u>			
Alkalinity	P, G	Cool, 4°C	Analyze onsite
Chloride	P, G	None required	28 days
Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH >12, 0.6 g ascorbic acid	14 days
Fluoride	P	None required	28 days
Hardness	P, G	$\text{HNO}_3$ to pH <2, $\text{H}_2\text{SO}_4$ to <2	6 months
Hydrogen ion (pH)	P, G	None required	Analyze immediately
<u>Metals:</u>			
Chromium VI	P, G	Cool, 4°C	24 hours
Mercury	P, G	$\text{HNO}_3$ to pH <2	28 days
Metals, except chromium VI and mercury	P, G	$\text{HNO}_3$ to pH <2	6 months
Nitrate	P, G	Cool, 4°C	48 hours
Nitrate-nitrite	P, G	Cool, 4°C, $\text{H}_2\text{SO}_4$ to pH <2	28 days
Nitrite	P, G	Cool, 4°C	48 hours
Orthophosphate	P, G	Filter immediately, cool, 4°C	48 hours
Specific conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days
<u>Organic Tests:</u>			
Purgeables	G, PTFE-lined cap	Cool, 4°C, HCl to pH<2	12 days
<u>Radiological Tests:</u>			
Tritium	P, G	$\text{HNO}_3$ to pH<2	6 months

a. Polyethylene (P) or Glass (G)

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- Identification of the sample as to whether it was a routine monitoring sample, check sample, or special purpose sample
- Date of analysis
- Laboratory and person responsible for performing the analysis
- Analytical method used
- Results of the analysis
- Records of actions taken to correct violations of maximum contamination limits (MCL) shall be maintained for not less than three years after the last action taken with respect to the particular violation
- Copies of all written reports, summaries, or communications relating to sanitary survey shall be kept for at least 10 years
- Records concerning a variance or exemption granted to the system shall be kept for at least five years following the expiration of the variance or exemption.

#### 5.4 Effluent Investigation

##### 5.4.1 Effluent Sampling Methods

The best place to collect a representative effluent sample is from a point where the water body is homogenous. Flowing effluent streams usually have enough flow and turbulence to maintain some degree of homogeneity and prevent the floating and sinking of immiscible organics; sumps, pits, and tanks do not. Still waters allow the more dense compounds to sink (e.g.,

PCBs and polyaromatic hydrocarbons) and less dense compounds to float (e.g., benzene and oils). For this reason, individual sampling locations in still waters must be targeted toward obtaining the parameters of concern when sampling for organics. Generally, composite wastewater samples should be used in these still waters. In small bodies of water where turbulent mixing is occurring, this is a relatively straightforward task. In small streams where mixing distances are very short, the sample should be collected from moving water near the middle of the stream at a point approximately six-tenths of the depth. The best method to manually collect a sample is to use the sample container that will be used to transport the sample to the laboratory. This eliminates the possibility of contaminating the sample with an intermediate collection container.

If the effluent cannot be reached by the sampling personnel, an intermediate collection container or device may be used. The container or device will be properly cleaned for the parameters to be sampled. It will also be made of a material that meets the requirements of the parameters being sampled. Collection of discrete samples can be performed with very simple tools. The sample is collected by lowering a properly cleaned Teflon, plastic, glass, or stainless-steel collection vessel into the stream to be sampled. The collection vessel may be lowered by hand or attached to a pole or rope. In some cases, it may be best or necessary to use a peristaltic pump. If a pump is used, it is imperative that all components of the pump that come in contact with the liquid are properly cleaned to ensure the integrity of the sample. Peristaltic pumps collect samples by creating a vacuum and, therefore, are not suitable for sampling of volatile parameters. The selection of which method to use depends primarily on the need to prevent aeration or turbulent mixing during sampling and the accessibility to the sampling point.

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Samples are collected by tipping the collection container in the effluent stream so the mouth of the container faces upstream. The sample container should be rinsed with water from the effluent stream prior to filling for all samples except those for organic parameters.

## 6. SAMPLE CUSTODY

A critical aspect of sound sample collection and analysis protocols is the maintenance of strict chain-of-custody procedures. Chain-of-custody procedures include inventorying and documentation during sample collection, shipment, and laboratory processing. A sample is considered to be in an individual's custody if the sample is (a) in the physical possession or view of the responsible party, (b) secured to prevent tampering, or (c) placed in a restricted area by the responsible party. The Project Geologist is responsible for the custody of the samples collected until they have been properly transferred to a courier or directly to the laboratory.

### 6.1 Chain-of-Custody

#### 6.1.1 Sample Labels

A label will be attached to each sample container at the time of collection. An example of the sample label is presented in Figure 6-1. The label will be completed in indelible ink and contain the following information:

- Date and time collected
- Purpose of the sample (analyte and sample group)
- Sample number
- Source and location of the sample
- Project number and title



(NAME OF SAMPLING ORGANIZATION)

SAMPLE DESCRIPTION \_\_\_\_\_  
\_\_\_\_\_

DATE: \_\_\_\_\_ LOCATION: \_\_\_\_\_  
TIME: \_\_\_\_\_  
MEDIA: \_\_\_\_\_ STATION: \_\_\_\_\_  
SAMPLE TYPE: \_\_\_\_\_ PRESERVATIVE: \_\_\_\_\_

SAMPLED BY: \_\_\_\_\_

SAMPLE ID NO.: \_\_\_\_\_ - - - -

LAB NO. \_\_\_\_\_

REMARKS

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

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Figure 6-1. Sample label.

- Method of collection (i.e., bailer, suction pumps, air-lift pump, etc.)
- Volume removed before sample taken (well samples only)
- Preservative used (if any)
- Name or initials of collector.

#### 6.1.2 Chain-of-Custody Record

Chain-of-custody forms will be used to document the integrity of all samples (Figure 6-2). To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a chain-of-custody form will be filled out for each sample set at each sampling location. The chain-of-custody form will contain the following information:

- Sample number (for each sample in shipment)
- Collection date (for each sample in shipment)
- Time shipment was packed
- Number of containers of each sample
- Sample description (environmental matrix)
- Analyses required for each sample
- Shipment number



- Shipping address of the laboratory
- Date, time, and method of shipment
- Spaces to be signed as custody is transferred from one individual to another.

The individual placed in charge of shipping samples to the laboratory is also responsible for completing the chain-of-custody form, including referencing all applicable blank samples, signing the form and noting the date and time of shipment. This individual will also inspect the form for completeness and accuracy. Any changes made to the chain of-custody form shall be initialed by the person making the change.

#### 6.1.3 Sample Tags

All necessary serialized sample tags will be distributed to field personnel by the Project Manager or a designated project participant. After the tag has been filled out, it is attached to a sample and transferred to another individual by use of a chain-of-custody record. Sample tags contaminated with a hazardous substance are disposed of properly along with any other hazardous wastes. The serial number of the contaminated tag is noted in the appropriate logbook, and the Project Manager is notified. At the completion of the field investigation activities, all unused tags (including those damaged or voided) will be returned to the Project Manager for disposal.

#### 6.1.4 Sample Traffic Report

To provide a permanent record for each sample collected, the sampler completes a Sample Traffic Report, recording the site name, analysis laboratory, sampling office, dates of sample collection and shipment, and

the sample concentration and matrix. The sampler will enter the numbers of sample containers and volumes beside the analytical parameter(s) requested for particular portions.

#### 6.1.5 Receipt-of-Samples Form

Section 300f(a)(2) of the RCRA states "If the officer, employee, or representative obtains any samples prior to leaving the premises, he shall give to the owner, operator, or agent-in-charge, a receipt describing the samples obtained and, if requested, a portion of each such sample equal in volume or weight to the portion retained." Section 104 of CERCLA, as amended by the Superfund Amendments and Reauthorization Act (SARA), contains identical requirements.

Completing a receipt-of-samples form complies with these requirements; such forms should be used whenever splits are offered or provided to the site owner, operator, or agent-in-charge. The form is completed and a copy given to the owner, operator, or agent-in-charge even if the offer for split samples is declined. The original is given to the Project Manager and is retained in the project files.

#### 6.1.6 Custody Seals (Sample Seals)

The Field Sample Custodian will be responsible for the proper recovery, preservation, and storage (locked with limited access) of the samples until they are delivered to the laboratory. Samples will be maintained according to EPA requirements. Sample seals (see Figure 6-3) or evidence tape will be used to detect unauthorized tampering of samples from the sample collection to the time of analysis. The seal or tape will be attached in such a way that it is necessary to break it to open the sample container. The seal will contain the following information:

NAME AND ADDRESS OF ORGANIZATION COLLECTING SAMPLES	
Person Collecting Sample _____ (signature)	Sample No. _____
Date Collected _____	Time Collected _____
Place Collected _____	

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Figure 6-3. Sample seal.

- Unit facility
- Sampling person
- Sample number
- Sample date/time
- Location description
- Radiation Level (if applicable).

## 6.2 Documentation

### 6.2.1 Sample Identification Numbers

A unique sample number will be assigned to each sample collected in the field.

### 6.2.2 Equipment Logbooks

Details on equipment used will be recorded in the field logbook. Information logged may include the following items:

- Identification of well
- Samples taken
- Calibrations done
- Maintenance

- Repairs
- Anomalies, fluctuations in readings, and strange or abnormal behavior
- Climatic conditions.

#### 6.2.3 Sample Analysis Request Sheet

The sample analysis request sheet (see Figure 6-4) will accompany the sample on delivery to the laboratory. The field section of this form will be completed by the person collecting the sample and should include most of the pertinent information noted in the field logbook. The laboratory section of this form is intended to be completed by laboratory personnel.

#### 6.2.4 Transfer of Custody and Shipment

Samples are to be accompanied by an approved chain-of-custody record. When the possession of samples is transferred, both the individual relinquishing the samples and the individual receiving the samples shall sign, date, and time the chain-of-custody document. This record shall represent the official documentation for all transfers of the sample custody until the samples have arrived at the laboratory.

Samples will be wrapped with plastic packing material and packed with Blue Ice into sturdy, thermally insulated coolers. Packaging and shipping procedures are discussed in detail in Subsection 6.4. Sealed coolers will be transported to an air shipping company for overnight delivery to the laboratory. The chain-of-custody record shall be available for the courier when required to maintain chain-of-custody protocols within their own organizations during their time of possession. A copy of the chain-of-custody form will be transported on the outside of the shipping containers in this situation. Other copies of the chain-of-custody form will be placed inside the containers.



### Sampling Analysis Request

Part I: Field section

Collector \_\_\_\_\_ Date sampled \_\_\_\_\_ Time \_\_\_\_\_ Hours \_\_\_\_\_

Affiliation of sampler \_\_\_\_\_

Address \_\_\_\_\_  
                    Number                    Street                    City                    State                    Zip

Telephone (\_\_\_\_) \_\_\_\_\_ Company contact \_\_\_\_\_

<u>Laboratory sample</u>	<u>Collector's sample no.</u>	<u>Type of sample*</u>	<u>Analysis requested</u>
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

Field Information\*\* \_\_\_\_\_

Special handling and/or storage \_\_\_\_\_

Part II: Laboratory section\*\*

Received by \_\_\_\_\_ Title \_\_\_\_\_ Date \_\_\_\_\_

Analysis required \_\_\_\_\_

\*Indicate whether sample is soil, sludge, etc.  
\*\*Use back of page for additional information relative to sample location.

Figure 6-4. Sample analysis request sheet.

If samples are to be split with another laboratory facility or government agency, a separate chain-of-custody record shall be prepared for those samples. This chain-of-custody record shall indicate with whom the sample has been split and shall be appropriately signed and dated with the time of transfer of splits.

#### 6.2.5 Shipment of Radioactive Samples

All samples will be surveyed by a Radiation Protection Technologist (RPT). If the samples fall below EG&G Idaho limits, a green tag will be issued and the samples may leave the facility. If the samples are above 2000 dpm, DOE Form IDF 5480.1A (Rev. 07-83) must be filled out, and the sample must be sent as a radioactive shipment (labeled and shipped as per 40 CFR 170 through 179).

#### 6.2.6 Laboratory Custody Procedures

The following subsections describe laboratory custody procedures associated with sample receipt, storage, preparation, analysis, and general security.

6.2.6.1 Sample Receipt. All samples will be received by the sample custodian, or, in the absence of the sample custodian, by the document control coordinator. All samples received shall be considered hazardous; therefore, all shipping containers will be opened in an approved exhaust hood or an approved, well-ventilated area. All personnel associated with sample receipt are familiar with safety procedures for the handling of hazardous samples. The objective of the sample receipt procedure is to

ensure that all pertinent information about the condition of the sample is recorded for eventual use in reporting and communicated to laboratory personnel, when appropriate, for proper handling, preparation, and analysis.

The sample custodian will compare the information on the chain-of-custody form with the information on the sample tags and record the final cooler temperature on the chain-of-custody form. The sample custodian shall document both agreement and discrepancies among the forms. If all samples recorded on the chain-of-custody form were received by the laboratory and no problems are observed with the shipment, the custodian will sign the form in the "received for laboratory by" box. If problems are noted, the custodian will sign the form and note problems in the "remarks" box. If discrepancies are found, they shall be reported immediately to the Project Manager for clarification.

6.2.6.2 Sample Storage. While within the laboratory, sample integrity is maintained through the use of locked storage areas. Samples remain in locked storage areas except when actively involved in the analytical process. A sample request form is used to inform the sample custodian of which samples are needed for either sample preparation or analysis. Removal of samples will be documented on the internal chain-of-custody form.

Samples to be analyzed for VOCs are stored, with a storage blank, separately from other types of samples. Standards are not stored with any samples.

6.2.6.3 Sample Tracking. Samples will remain in locked storage areas except when actively involved in the analytical process. A sample request form is used to inform the sample custodian of which samples are needed for sample preparation and/or analysis. The sample custodian will distribute the requested samples to the appropriate analysts. An internal

chain-of-custody form will be signed by the individual to whom the samples are to be relinquished. The internal chain-of-custody form shall be secured in the project file and maintained under lock by the sample custodian. All laboratory personnel who receive samples for analytical purposes are responsible for the care and custody of the samples from the time each sample or fraction thereof is received until the samples are returned to the sample custodian or disposal is documented. All subsets (extraction, digestates, etc.) of the sample shall be kept in locked storage controlled by the appropriate laboratory manager.

6.2.6.4 Record Keeping. Documentation in the laboratory is initiated by the sample custodian, who receives samples, assigns laboratory numbers, and generates chain-of-custody forms. Each shipment of samples received is given a unique batch number. A batch consists of a number of samples carried through the entire analytical procedure, along with samples and standards. All work performed on a sample batch is documented in the Sample Receiving Logbook, which is a compilation of computer-generated sample summary forms entered into the Laboratory Information Management System (LIMS) on a sample receipt basis. It is compiled on a monthly basis to document sample receipt information.

6.2.6.5 Laboratory and Sample Security. Samples received at the laboratory are considered to be physical evidence and are handled according to procedural safeguards established by EPA. Because of the legal nature of the work, the laboratory provides complete security for samples, analysis, and data.

To ensure complete security for samples and analytical procedures during sample analyses, analytical samples will always be in the custody of the individual technician assigned to do the task. The following general security measures are employed:

- Doors to the laboratory will be kept closed and secured at all times
- Only authorized personnel and visitors under escort shall have access to the laboratory
- Outside exit doors (with the emergency bar) are kept closed and locked at all times
- All laboratory personnel will be aware of the need to question and determine the legitimacy of a stranger's presence in the laboratory
- Deliveries will be escorted to the laboratory from the main reception area or from the loading dock
- All personnel are to display identification badges at all times.

### 6.3 Documentation

#### 6.3.1 Sample Identification

Most methods of analysis of environmental samples must be accomplished within a specific amount of time after the collection of the sample. Where required, these timeframes ("holding times") are given in the method. Careful tracking of the analytical status of samples is required to ensure that these holding times are met.

Sample tracking will be accomplished in the field by assigning each sample a unique number as it is collected. This number will be traceable back to the day, time, site, and depth (where appropriate) of collection.

This number will be recorded on the sample label and chain-of-custody form as well as in the field logbook. A master log of the identification numbers used will be maintained by the on-site Project Geologist. All containers will be labeled prior to actual sampling. A general identification number will be used to identify such items as monitoring well location and sample interval. Each number or letter provides vital information pertaining to site identification, sample location, and sampling interval.

Sample tracking will be accomplished in the laboratory by assigning each sample a unique laboratory sample number when it is logged into the laboratory. This number must be traceable back to the field sample identification number. This unique sample number will be used to identify the sample during storage, analysis and data reduction, data validation, and reporting.

The following sample information is recorded into the Laboratory Information Management System (LIMS):

- Customer and project information
- Date of receipt
- Client identification
- Date sampled
- Matrix type
- Number of containers

- Analytical requirements
- Other pertinent comments.

After the sample is logged-in, an internal chain-of-custody/tracking form is generated. This internal chain-of-custody document requires that the sample be formally relinquished by one party and accepted by the other at each step of the analytical process. This document accompanies the sample through each step of the analytical process. When the analytical activities are completed, internal and external chain-of-custody documents are completed and filed in the appropriate project file. Based on specific contract requirements, any remaining samples are either archived in locked storage areas or disposed of properly.

In addition to the external and internal chain-of-custody documents, a computer-generated listing of the different parameters for sample analysis is used to further control sample flow and facilitate tracking within the laboratory. Each concerned functional laboratory unit is given the list of parameters and is responsible for maintaining sample integrity, fulfilling chain-of-custody requirements, scheduling sample flow, and tracking sample status.

All sample data will be entered into the LIMS in accordance with the established protocols for sample control chain-of-custody requirements.

### 6.3.2 Daily Logs

Daily logs will be kept during field activities by the Project Geologist at each site. These daily logs will be kept in a bound field notebook of water-resistant paper. All entries will be made legibly in indelible ink, signed, and dated. Information that will be recorded in the field notebook includes:

- Date, time, and place of sampling
- Field QC samples, as applicable
- Weather conditions at time of sampling, including ambient temperature and approximate wind direction and speed
- Data from field analyses (e.g., temperature, specific conductance, pH, and alkalinity of water samples)
- Turbidity of water samples
- Data from physical tests (slug tests, etc.)
- Observations about site and samples (odors, appearance, etc.)
- Information about any activities, extraneous to sampling activities, that may affect the integrity of the samples (such as low-flying aircraft nearby, fossil-fueled motors being used nearby, painting operations being carried out upwind of sampling sites)
- Analyses and required preservation techniques
- Sample cooler temperature readings.

### 6.3.3 Corrections to Documentation

When it becomes necessary to make corrections to any form of documentation (e.g., sample tags, chain-of-custody forms, daily logbooks), the obsolete information will be crossed out with a single line, and the changes will be made, initialed, and dated using indelible ink by the person making the change.



#### 6.3.4 Photographs

A photographic record will be made during the field program, documenting most field activities (i.e., drilling, soil boring, well installation, and sample collection). This photographic record may be used to highlight and enhance appropriate sections in the report.

#### 6.3.5 Disposition of Documentation

Upon conclusion of the field effort at TAN, all field documentation (i.e., maps, well logs, logbooks, photographs) will be clearly labeled and placed in the project files.

### 6.4 Sample Packaging and Shipping

#### 6.4.1 Sample Packaging

Samples will be packaged according to the following procedures:

- Custody tape will be wrapped around the neck and cap of each container
- Glass sample containers will be wrapped with plastic insulating material to prevent contact with other sample containers or the inner walls of the cooler
- Samples will be classified according to the Department of Transportation (DOT) regulations pursuant to Title 49 CFR.

#### 6.4.2 Shipping Containers

Samples will be packaged in thermally insulated, rigid coolers, according to DOT specifications 173.510 and 172 Subparts B, C, and D, and Subparts A and B of Part 173. Sample containers will be placed in a cooler that contains Blue Ice and absorbent packing for liquids or styrofoam packing for solids.

The completed chain-of-custody form will be placed inside the shipping container, unless otherwise noted.

#### 6.4.3 Marking and Labeling

Each cooler will be wrapped twice with custody tape, in a single strip, around the outside. Each cooler will then be marked as follows:

- Proper shipping name: hazardous substance, liquid, or solid
- Hazardous class: to be determined (label placed in upper left corner of outer container)
- Labels: "This Side Up" or arrows placed on the opposite side of the outer container if a liquid is to be shipped.

Figure 6-5 is a sample label.

A hazardous material shippers certification will be filled out and will accompany the shipment. The container will be secured with strapping tape to prevent leakage.

(NAME OF SAMPLING ORGANIZATION)	
SAMPLE DESCRIPTION _____ _____	
PLANT: _____	LOCATION: _____
DATE: _____	_____
TIME: _____	_____
MEDIA: _____	STATION: _____
SAMPLE TYPE: _____	PRESERVATIVE: _____
SAMPLED BY: _____	
SAMPLE ID NO.: _____ - _____ - _____ - _____	
LAB NO. _____	REMARKS       

7-0223

Figure 6-5. Sample label.

## 7. CALIBRATION PROCEDURES AND FREQUENCIES

This section describes the calibration procedures and frequencies for the following types of equipment and instrumentation:

- Field equipment, including water level indicators, and field water quality and field air quality screening equipment
- Laboratory instrumentation, including inorganic and organic analytical instrumentation
- Field soil gas analytical instrumentation.

### 7.1 Field Equipment

In general, all equipment will be calibrated daily prior to use and will be immediately recalibrated if field personnel suspect that the calibration may have been altered. Reasons for such alteration include change of batteries, equipment being dropped or knocked around, or significant changes in temperature since the last calibration.

The MSA combustible gas indicator and HNU photoionization analyzer are used for field personnel safety purposes and for screening potentially hazardous soil samples. The remaining field equipment (i.e., pH meters, conductivity meters, field alkalinity analyzers, and thermometers) is used to evaluate water well stability during purging. Because the instrumentation and procedures are continually being updated, field personnel are required to consult the appropriate instruction manual for calibration instructions.

### 7.1.1 HNU Photoionization Analyzer

A rapid procedure for checking responsiveness involves bringing the probe and readout in proximity to the calibration gas, cracking the valve on the tank, and checking the instrument reading. This provides a useful spot check for the instrument.

The recommended and most accurate procedure for calibration of the instrument from a pressurized container is to connect one side of the 'T' to the pressurized container of calibration gas, another side of the 'T' to a rotameter, and the third side of the 'T' directly to the 8-in. extension to the photoionization probe. The valve of the pressurized container should be cracked until a slight flow is indicated on the rotameter. The instrument draws in the volume of sample required for detection, and the flow in the rotameter indicates an excess of sample. The span pot should be adjusted so that the instrument is reading the exact value of the calibration gas. (If the instrument span setting is changed, the instrument should be turned back to the standby position and the electronic zero should be readjusted, if necessary.)

The calibration gas is prepared in the same matrix (air, nitrogen, hydrogen, etc.) in which it is to be measured; otherwise an inaccurate reading may be obtained. Each calibration gas is an approved instrument part and certified by HNU. Calibration with toxic gases should be performed in a hood or outdoors, since the photoionization detector (PID) is a nondestructive analyzer. The increased response that is seen in oxygen-free gases can be attributed to a reduction in the quenching of ions by oxygen (actually O<sub>2</sub>) and is typical of any ionization detector. The quenching effect of oxygen is constant from about 10% O<sub>2</sub> to very high levels.

If a gas standard prepared in nitrogen is to be used for measurements in air, a 0.5-or 1-L bag should be filled with the standard and 50 or 100 mL of pure oxygen should be added to bring the level to 10 to 12%. Any error between this value and 20% oxygen is quite small.

#### 7.1.2 MSA Combustible Gas Indicator

Gases used to calibrate the MSA Combustible Gas Indicator are approved instrument parts and certified by the manufacturer. The following steps describe how to calibrate an MSA combustible gas indicator:

- Check and zero the instrument
- Attach the flow control to the recommended calibration gas tank
- Connect the adapter-hose to the flow control
- Open the flow control valve
- Connect the adapter-hose fitting to the inlet of the instrument. After approximately 15 seconds, the LEL meter pointer should be stable and within the range specified on the calibration sheet accompanying the calibration equipment. If the meter pointer is not in the correct range, stop the flow and remove the right hand side (speaker) panel. Turn on the flow and adjust the "S" control with a small screwdriver to obtain the reading specified on the calibration sheet
- Disconnect the adapter-hose fitting from the instrument
- Close the flow control valve

- Remove the adapter-hose from the flow control
- Remove the flow control from the calibration gas tank
- Replace the side panel on the instrument
- Make an oxygen system calibration check each time a combustible gas check is made.

### 7.1.3 Specific Conductance Meter

The following steps describe how to calibrate the specific conductance meter:

- With the instrument turned off, check the meter's mechanical zero setting. Adjust the screwdriver adjustment control on the meter face if necessary to obtain a zero reading
- Press the power switch to on, and press the battery check switch. Verify the meter needle deflects to the battery check area
- Connect a clean, dry probe to the instrument. Remove the instrument from its carrying case, and place the instrument on a padded surface
- Press the 0 to 2 range switch, and verify that the meter reads zero. If it does not, adjust the null adjust potentiometer RB9 on the amplifier circuit board to obtain a reading as near zero as possible
- Press the 0 to 2000 range switch

- Immerse the probe in the 1000 mg/L sodium chloride solution. The meter should read 1990  $\mu$ hos/cm. If it does not, adjust the standardization potentiometer R32.

#### 7.1.4 pH Meter

The following steps describe how to calibrate the pH meter:

- Connect the two probes to the appropriate jacks on the instrument panel. Be sure the fill hole in the pH electrode is uncovered. The fill hole is to remain covered at all times except for calibration and pH measurement. When the electrode is not in use, slide the rubber sleeve over the fill hole.
- Prepare pH 4 and pH 9 buffer solutions by dissolving the contents of one powder pillow of each in separate beakers containing 50 mL demineralized water.
- Select the T(c) mode and measure the temperature of each buffer solution. Referring to the temperature coefficients table, determine the actual pH values of the buffer solutions for those temperatures. Calculate the difference between the two pH values.
- Immerse the probes in the pH 4 buffer solution and select the pH mode. Allow approximately 30 seconds to reach equilibrium and adjust the CAL control to obtain 0.00 reading
- Remove the probes from the pH 4 solution and rinse thoroughly with demineralized water



- Immerse the probes in the pH 9 buffer solution. Allow approximately 30 s for the probes to reach equilibrium. Adjust the SPAN control for a reading equal to the difference value calculated above
- Adjust the CAL control for a reading equal to the actual pH value of the pH 9 buffer as adjusted for temperature
- Rinse the probes thoroughly with demineralized water.

#### 7.1.5 Water Level Indicator

This instrument arrives calibrated by the manufacturer for water level measurement.

#### 7.1.6 Digital Thermometer

This instrument arrives calibrated by the manufacturer for temperature measurements.

#### 7.1.7 Field Alkalinity Analyzer

The following calibration check should be performed when interferences are suspected or to verify analytical technique:

- Snap the neck off an Alkalinity Voluette Ampule Standard (0.500N)
- Use a Ten Sette Pipet to add 0.1 mL of standard to the sample already titrated. Resume titration back to the same end point. Record the number of digits needed

- Repeat, using two or more additions of 0.1 mL standard. Titrate to the end point after each addition
- Each 0.1 mL addition of standard should require 25 additional digits of 1.600N titrant or 250 digits of 0.1600N titrant.

## 7.2 Laboratory Instrumentation

Calibration is the process for determining the correctness relative to physical or chemical standards used or assigned values in scales of measuring instruments. Calibration establishes a reproducible reference point to which all sample measurements can be correlated.

### 7.2.1 Instrument Calibration

A step-by-step calibration procedure is kept with each instrument. The methods and frequency of calibration for any particular instrument are in accordance with the manufacturer's recommendation.

To ensure calibration of instruments with daily variances, a series of calibration samples are analyzed before any samples are run. At the end of the instrument working day, or once in 24 h, a calibration check standard is analyzed. As a participant of EPA CLP for organics, the laboratory follows the Invitation for Bid (IFB) Statement of Work GC/MS analyses before initiating any ongoing data collection. This is accomplished, as well as a sensitivity check, with the use of 4-bromofluorobenzene (BFB) for Method 8240 injected at concentration near the instrument detection limit. These standards are run at least daily or every 12 h to validate that the GC/MS system is still tuned correctly. The results of all tuning analyses will be attached to the applicable samples and placed in the pertinent project file.

Calibration of the GC/MS, like GC calibration, is established and validated by the injection of EPA-traceable standards at three concentration levels over the range of likely sample concentrations. During the initial calibration, system performance check compounds and calibration check compounds are analyzed to verify the validity of the calibration. The initial calibration curve is used as long as it can be demonstrated to be valid. For VOA analysis, a method blank is analyzed after the calibration verification. Surrogate standards will be added to each sample prior to preparing the sample for extraction or instrumental analysis. Surrogate standard recovery shall be used as a method for determining the presence of matrix interferences and as an indication of extraction efficiencies. In addition, internal standards will be used, added to standards, extracts or samples just prior to instrumental analysis, as an indicator of variations in instrumental conditions and for use in quantitation. An internal calibration procedure is used; that is, in addition to surrogate recovery compounds, sample extracts are spiked with internal calibration standards that span the retention time range of the analytes of interest. The concentration of the analytes are calculated with reference to the response factor (RF) of the internal standard for each sample.

7.2.1.1 Spectrophotometers (Infrared). A polystyrene sample will be used to check instrument wavelength. Acceptance criteria will be as per manufacturer's specifications. Spectrophotometers are calibrated daily before any samples are analyzed. The calibration standards are prepared from reference materials at five concentrations to cover the anticipated range of measurements and include a calibration blank. The requirement for an acceptable initial calibration is a correlation coefficient equal to or greater than 0.996. Before sample analysis, an initial calibration verification standard is analyzed. The response of this standard must be within  $\pm 15\%$  of the initial calibration or the instrument must be recalibrated.

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7.2.1.2 General Laboratory Equipment. Balances will be calibrated before every use with standard Class-S calibration weights and semi-annually by a licensed specialist. The pH/specific-ion meters will be calibrated before each use with a minimum of three standard buffer solutions (pH 4, 7, and 10). The micro-automatic pipettes will be checked and calibrated before each use by using a combination of precalibrated (marked) pipette tips and by weighing the volume of deionized water dispensed from the pipettes. A minimum of 100 mL ( $\pm 5\%$ ) of water will be collected for this determination, and the mass of water will be collected from the density of water adjusted for temperature. Results shall be documented and maintained with sample results generated.

### 7.3 Field Soil Gas Analytical Instrumentation

Soil samples will be analyzed using a Varian 3300 GC and a SpectraPhysics SP 270 computer integrator or comparable equipment. Quantification of the target parameter concentrations in the soil gases will be accomplished using liquid standards that will be used to calibrate the GC on a daily basis or more frequently if needed. A detailed description of analytical calibration as well as QA/QC procedures is described in Appendix A.

## 8. ANALYTICAL PROCEDURES

### 8.1 Field Testing and Screening

During multimedia sampling activities performed during the IRP Stage 2 field effort at TAN, selected physical and chemical parameters in the air, water, and soil at the site will be measured. Equipment and general procedures for analysis of field samples are listed below. Because field instrumentation and analytical methodology is continually being updated, field personnel are required to consult each manufacturer's instruction manual for operating procedures.

#### 8.1.1 Measurements for pH

Measurements for pH will be taken on water purged from wells prior to sampling. Groundwater samples will be collected after a stable pH is achieved to ensure that conditions are representative of the formation. Measurements for pH also will be taken on surface water samples. A Hach Model 19000 Digital pH meter or equivalent will be used for field analyses using the procedure described below.

The probes will be rinsed thoroughly with ASTM Type II reagent water to prevent any carryover. The pH electrode fill hole must not be covered, and the mode switch will be set to pH. The probes will be immersed in the test sample and the reading taken. The reading will be allowed to stabilize for 30 seconds. After pH measurements are conducted, the sampler will slide the rubber sleeve over the fill hole.

#### 8.1.2 Specific Conductance

Conductivity measurements will be taken on purge water and all groundwater and surface water samples using a Hach Model 16300 portable conductivity meter according to the procedure described below.

The probe will be connected to the PROBE INPUT connector on the front panel. The sampler will press the POWER switch on and perform a battery check. The switch will be placed on the highest range, and the probe will be immersed in a beaker containing the sample solution. The probe will be shaken or tapped on the bottom of the beaker to ensure that no air bubbles are trapped near the electrode. About 10 seconds will be allowed for the probe to stabilize before taking the reading. If the meter indicator falls in the lowest 10% of the range, the next lower range will be used. This will be repeated until the proper range is selected. Dilute if necessary. The probe will be rinsed with ASTM Type II water between each use.

#### 8.1.3 Temperature

Temperature data will be used in conjunction with the chemical data for groundwater and surface water characterization. Temperature measurements will be taken on purge water and all groundwater and surface water samples using a mercury thermometer or the conductivity meter, which is equipped to measure temperature, according to the procedure described below.

The temperature probe will be connected and the T(c) mode selected. The probe will be immersed in the test sample for 30 seconds before taking the reading. The probe will be rinsed with ASTM Type II reagent water after each use.

#### 8.1.4 Air Monitoring Measurements

A portable photoionization analyzer will be used to continuously monitor ambient air conditions during drilling. Measurements will be made both upwind and downwind of the site, in the breathing zone above the borehole, and over fresh, returned cuttings from the borehole. In addition, soil and formation samples will be selected for air testing as an early

warning device in case contaminants are present. A soil sample will be placed in a clean glass jar, and the air sampling probe will be inserted to withdraw an air sample to be analyzed according to the procedures described below.

8.1.4.1 HNU Photoionization Analyzer. The function switch will be turned to the battery check position. The needle on the meter should read within or above the green battery arc on the scale plate. If the needle is in the lower position of the battery arc, the instrument should be recharged prior to making any measurements. If the red LED comes on, the battery should be recharged.

The function switch will be turned to the ON position. In this position, the UV light source should be on. (This can be checked by looking at the end of the probe to see the purple glow of the lamp.)

To zero the instrument, the function switch should be turned to the standby position, and the zero potentiometer should be rotated until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection, while counterclockwise rotation yields a downscale deflection. No zero gas is needed, since this is an electronic adjustment. If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted, if necessary. Wait 15 or 20 seconds to ensure that the zero reading is stable. If necessary, the zero should be readjusted.

The instrument will then be ready for measurement when the function switch is turned to the proper measurement range. The instrument is supplied calibrated to read directly in parts per million (ppm) (v/v) 0 to 20, 0 to 200, 0 to 2000 of benzene with the span position set at 9.8. For additional sensitivity, the span potentiometer is turned counterclockwise (smaller numbers) to increase the gain. By changing the

span setting from 10.0 to 1.0, the sensitivity is increased approximately ten-fold. Then, the 0 to 20, 0 to 200, and 0 to 2000 ppm scales become 0 to 2, 0 to 20, and 0 to 200 ppm full-scale, respectively. This span control also is used to make the instrument scale read directly in ppm of the compound being measured. That is, it is adjusted to match the value of a calibration gas to that same reading on the instrument scale. The span control can be used to calibrate nearly any compound, measured by photoionization, to the direct reading on the 0 to 20 ppm range. For example, gain settings of 4.5 or 8.9 will provide direct reading capability (0 to 20, 0 to 200 ppm) for vinyl chloride and trichloroethylene, respectively.

A small DC-operated fan is used to pull air through the photoionization sensor at a flow rate of 3 to 700 centimeters per minute (i.e, 0.5 liters per minute). The fan provides nearly instantaneous response times while consuming little power. The characteristics of the fan are such that it cannot tolerate a significant pressure drop without affecting the flow rate and, therefore, either the instrument reading or response time. Since photoionization is essentially a nondestructive technique, changes in flow rate do not affect the signal, but if a large pressure drop is imposed at the inlet of the probe, the sample may not reach the sensor.

8.1.4.2 MSA Combustible Gas Indicator. The instrument lid will be opened, and the center ON-OFF control should be turned to the HORN OFF position. Both meter pointers will move, and one or both alarms may light.

The percent LEL meter pointer should be set to zero by adjusting the zero LEL control. Adjustment should be made within 10 seconds after the instrument is turned on; this is to prevent accidental activation of the meter latch circuit.



If the percent oxygen meter pointer stabilizes at a value other than 20.8%, the pointer should be set to 20.8% by using the CALIBRATE O<sub>2</sub> control.

The operator will then press the ALARM RESET button; the alarm(s) should reset, and the green pilot light should flash.

The operator will place a finger over the sample inlet fitting or the end of the sample line probe and should observe that the flow indicator float drops out of sight, indicating no flow.

He or she will then press the CHECK button and observe the percent LEL meter. The pointer must read 80% LEL or higher, as marked by the BATTERY zone on the meter. If the pointer reading is less, the battery pack must be recharged; no tests should be attempted as the instrument will malfunction.

The ON-OFF control will be turned to the ON position. The pilot lamp should light continuously.

Accessory equipment such as sampling lines, probes, carrying harness, filters, or line traps should be attached as required. All connections should be tightened, and flow indication should be tested by blocking flow at the far end of the sampling system. If the flow indicator float does not drop, all connections will be checked, and the test will be repeated. The instrument will then be ready for atmospheric sampling.

#### 8.1.5 Alkalinity Measurements

When measuring alkalinity, a water sample will be titrated with sulfuric acid to a colorimetric endpoint corresponding to a specific pH. Phenolphthalein alkalinity is determined by titration to pH 8.3, as

evidenced by the color change of the phenolphthalein indicator, and indicates the total hydroxide and one half the carbonate present. Total alkalinity is determined by titration to a pH between 3.7 and 5.1, and includes all carbonate, bicarbonate, and hydroxide. The procedure for determining alkalinity is described below:

- Collect samples in clean polyethylene or glass bottles. Fill completely and cap tightly. Avoid excess agitation or prolonged exposure to air. Samples should be analyzed as soon as possible after collection but can be stored 24 h by cooling to 4°C. Warm to room temperature before analyzing.
- Select the sample volume and sulfuric acid titration cartridge corresponding to the expected alkalinity concentrations as mg/L calcium carbonate from Table 8-1.
- Insert a clean delivery tube into the titrator cartridge. Attach the cartridge to the titrator body.
- Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.
- Use a graduated cylinder or pipet to measure the sample volume from Table 8-1. Transfer the sample into a 150-mL Erlenmeyer flask. Dilute to 100 mL with deionized water if necessary.
- Add the contents of one phenolphthalein indicator powder pillow and swirl to mix. A solution of one pH 8.3 buffer powder pillow and one phenolphthalein powder pillow in 50 mL of deionized water is recommended as a comparison for determining the proper endpoint color.

TABLE 8-1. SAMPLE VOLUME SELECTION GUIDE

Range (mg/L as CaCO <sub>3</sub> )	Sample Volume (mL)	Titration Cartridge (H <sub>2</sub> SO <sub>4</sub> )	Digit Multiplier
10-40	100	0.1600N	0.1
40-160	25	0.1600N	0.4
100-400	100	1.600N	1.0
200-800	50	1.600N	2.0
500-2000	20	1.600N	5.0
1000-4000	10	1.600N	10.0

- Titrate to a colorless endpoint if the color turns pink. Place the delivery tube tip into the solution and swirl the flask while titrating with sulfuric acid. Record the number of digits required. Phenolphthalein alkalinity calculation: Digits required x Digit Multiplier = mg/L Phenolphthalein alkalinity (as CaCO<sub>3</sub>).
- Add the contents of one Bromocresol Green-Methyl Red indicator powder pillow to the flask and swirl to mix. A solution of one Bromocresol Green-Methyl Red powder pillow and one pillow of the appropriate pH buffer in 50 mL deionized water is recommended as a comparison for judging the proper endpoint color.
- Continue the titration with sulfuric acid to a light greenish blue-gray (pH 5.1), a light violet-gray (pH 4.8), or a light pink (pH 4.5) color, as required by the sample composition (see Table 8-2). Record the number of digits required. Total alkalinity calculation: Digits required x Digit Multiplier = Total Alkalinity (as mg/L CaCO<sub>3</sub>).
- Carbonate, bicarbonate, and hydroxide concentrations may be expressed individually using the relationships in Table 8-3.

TABLE 8-2. SAMPLE COMPOSITION

Sample Composition	Endpoint
Alkalinity about 30 mg/L	pH 5.1
Alkalinity about 150 mg/L	pH 4.8
Alkalinity about 500 mg/L	pH 4.5
Silicates or phosphates present	pH 4.5
Industrial waste or complex system	pH 3.7

TABLE 8-3. ALKALINITY RELATIONSHIP TABLE

Result of Titration	Hydroxide Alkalinity	Carbonate Alkalinity	Biocarbonate Alkalinity
Phenolphthalein Alkalinity = 0	0	0	Equal to total
Phenolphthalein alkalinity less than one half of total alkalinity	0	2 times the phenolphthalein alkalinity	Total alkalinity minus 2 times phenolphthalein alkalinity
Phenolphthalein alkalinity equal to one half of total alkalinity	0	2 times the phenolphthalein alkalinity	0
Phenolphthalein alkalinity greater than one half of total alkalinity	2 times the phenolphthalein alkalinity minus the total alkalinity	2 times the difference between the total and phenolphthalein alkalinity	0
Phenolphthalein alkalinity equal to the total alkalinity	Equal to the total alkalinity	0	0

## 8.2 Laboratory Methods

The laboratory employs analytical procedures based on the EPA-approved methods for both inorganic and organic analyses of multi-media environmental samples. Methods to be used for the inorganic and organic analysis of routine samples are based on 40 CFR 136 as published in the Federal Register dated Friday, October 26, 1984; EPA's Test Methods for Evaluating Solid Waste (SW-846 3rd Edition); EPA's Methods for Chemical Analyses of Water and Wastes, and EPA's Standards Methods for the Examination of Water and Wastewater.

All work completed on a sample set is to be recorded in the following laboratory notebooks:

- Standards Logbook: To record the preparation and use of all standards in the laboratory. The logbook will indicate standard traceability to EPA or NBS standards. It will note date of preparation, expiration, concentration, and name of the preparer.
- Chemist's Bound Notebook: To record the raw data and final data of every batch. This notebook is used to document all activities associated with the analytical process. Laboratory notebooks of each staff member are a functional record and are pre-numbered.
- Instrument Benchsheet Logbook: To record sample run sequence or injections done during a day or shift, noting all instrument working parameters.

## 9. DATA REDUCTION, VALIDATION, AND REPORTING

### 9.1 Field and Technical Data

The collected data will be divided into field data and technical documentation. Technical documentation will be combined field and analytical data and will enable definitive characterization of the extent and magnitude of specific contaminants at each site. Field data will contain data from all measurements performed onsite including well stability measurements, well logging, water level measurements, soil gas readings, and PID measurements. Technical data will include all field and analytical data plus the results of the field and laboratory QC samples. This technical data will be incorporated into the final report.

#### 9.1.1 Field and Technical Data Reduction

All field measurements and observations will be recorded in project logbooks, field data records, or similar types of recordkeeping books. Field measurements include pH, temperature, conductivity, alkalinity, water flow, and certain air quality parameters. All data will be recorded directly and legibly in field logbooks with all entries signed and dated. If entries must be changed, the change should not obscure the original entry. The reason for the change should be stated, and the correction and explanation should be signed and dated or identified at the time the correction is made. Field data records will be organized into standard formats whenever possible and retained in permanent files.

All laboratory data will be cross-referenced to the appropriate trip blank, field blank, equipment blank, method blank, field duplicate or replicate, matrix spike, and matrix spike duplicate. In addition, all pertinent dates (dates collected, received by the laboratory and analyzed)

for each laboratory analysis applicable to the RFI will be referenced against their respective holdtimes.

#### 9.1.2 Field and Technical Data Validation

Validation of field data will be performed on two different levels. First, all data will be validated at the time of collection by following standard procedures and QC checks specified in Section 13. Second, data will be validated by the Project Geologist, who will review the data to ensure the correct codes and units have been included. After data reduction into tables or arrays, the Project Geologist will review data sets for anomalous values. Any inconsistencies discovered will be resolved immediately, if possible, by seeking clarification from the field personnel responsible for data collection. The Project Geologist is also responsible for ensuring that defensible and justifiable data were obtained by following the field objectives described below:

- Adherence to the RFI Plan for TAN Groundwater
- Proper calibration and working order of equipment and instruments
- Sample collection according to standard operating procedures
- Collection of sufficient sample volumes to maintain sample integrity and conduct all required analyses
- Proper preservation of samples
- Providing all applicable blanks and field QC samples with each sample set

- Complete documentation of the chain-of-custody throughout the duration of the TAN field effort and inclusion of copies with each sample shipment
- Arrival of field samples at the laboratory in good condition.

Random checks of sampling and field conditions will be made by the Project Geologist, who will check recorded data at that time to confirm observations. Whenever possible, peer review also will be incorporated into the data validation process to maximize consistency between field personnel.

Once both field and analytical data have been combined, the resulting technical documentation will be validated against the following criteria:

- Stated objectives of the RFI Plan for TAN Groundwater
- Stated QA objectives of the Data Collection Quality Assurance Plan
- Analysis date versus the applicable holdtimes
- Percentage of QA analyses conducted
- Field and laboratory blank contamination
- Laboratory accuracy (percent recovery versus control limits)
- Laboratory and field precision (RPD versus control limits).

Descriptive statistics for completeness will be calculated and reported.



### 9.1.3 Field and Technical Data Reporting

A detailed description of the type and format for technical reports to be produced during this project is presented in Subsection 4.19 of Task IV.

## 9.2 Laboratory Data

All bench chemists will document sample preparation activities in a bound laboratory notebook, which will serve as the primary record for subsequent data reduction. The data for GC/MS analyses will be generated by stand-alone computers. The data for atomic absorption analysis will be conducted using the instrument's recorder to measure absorbance readings and strip chart to record absorbance expressed in peak height units. Results of each analysis will be transcribed manually onto analytical results forms specific to the particular analysis. All data will be checked for accuracy and precision at the bench and instrument operator/analyst level, the laboratory manager's level, and the Laboratory QA Officer's level. The Laboratory QA Officer's review will consist of comparing spike recovery and/or relative percent difference to control limits established for the parameter analyzed. Concentration of the analytes found in the analysis will be expressed according to the required units, depending on the sample matrix (i.e., mg/L for aqueous samples or  $\mu\text{g}/\text{Kg}$  for soil samples).

### 9.2.1 Laboratory Data Reduction

9.2.1.1 Gas Chromatography/Mass Spectrometry Results. Qualitative identification will be determined by obtaining mass spectra for each target analyte and comparing the sample spectrum to a standard spectrum generated on the same instrument. Positive identification will be based on the following criteria:

- The intensity of the three characteristic masses of each analyte must maximize in the same ratio ( $\pm 20\%$ ), within one scan of each other
- The retention time must fall within  $\pm 30$  seconds of the retention time of the authentic compound
- The relative peak heights of the three characteristic masses in the EICPs must fall within  $\pm 20\%$  of the relative intensities of these masses in a reference mass spectrum (standard analysis or reference library).

Structural isomers to be listed as separate analytes must have an acceptable resolution. Acceptable resolution is achieved if, in a standard mix, the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

The calculation for the concentration for the suspect peak will be made using the average response factor (RF) for each analyte.

$$\text{Concentration} = \frac{(A_s)(C_{is})}{(\mu\text{g/L})(A_{is})(\text{RF})}$$

where:

$A_s$  = Area of characteristic  $m/z$  for the analyte to be measured

$A_{is}$  = Area of characteristic  $m/z$  for the internal standard

$C_{is}$  = Concentration of the internal standard, in  $\mu\text{g/L}$

RF = Average response factor as calculated from the area of the peak formed on an intensity plot of the ion of interest.

9.2.1.2 Atomic Absorption Spectrophotometry Results. Photometric absorbance is governed by the relationship:

$$\text{Absorbance} = \log (100/\%T) = 2 - \log\%T$$

where:

$$\%T = 100 - \% \text{ absorption}$$

Percent absorption is based on the amount of light of a particular wavelength absorbed by a specific metal. Its calculation is based on the loss of light after a beam of light of a particular wavelength is passed through a flame into which a solution containing metals of interest has been aspirated.

Calibration curves establishing the absorbance relationship with concentration are generated at various concentrations. From these curves, a comparison is made with absorbance from sample measurement. Since absorbance is directly related to concentration, a plot of the two parameters will be linear in certain operable ranges and will allow for determination of unknown concentrations in solutions (direct samples or extracts) after measurement of absorbances.

Atomic absorption spectrophotometry is based on the principle that if light of a resonance wavelength is passed through a flame containing atoms of an element to be measured, then part of the light will be absorbed and the extent of absorption will be proportional to the number of atoms present in the flame. Because of the sophistication of current instrumentation, the partial application of this technique for the measurement of metal concentrations in liquids relies on a Beer's absorption law approach, comparing absorbance from an unknown against the linear correlation between absorbance and concentration in standards.

In many spectrophotometric measurements, interferences occur in the absorption of light confusing the Beer's Law relationship between absorption and concentration; this is especially true for atomic absorption. To alleviate this problem, a technique known as the "method of standard additions" is used in which sequential known amounts of the component being analyzed for are added to a sample of unknown concentration. By making an initial absorbance measurement and measurement after each addition, the effects of interferences present in the analytic matrix can be accounted for.

#### 9.2.2 Laboratory Data Validation

Data validation will be performed by the specific analytical task leader, the Laboratory QA Officer, and the Laboratory Project Manager. Validation will be accomplished through routine audits of the data collection and flow procedures and monitoring of QC sample results. Data collection and flow audits include:

- Review of sample documents for completeness by the analyst(s) at each step of the analysis scheme
- Daily review of instrument logs, performance test results, and analyst performance by the analytical Program Task Manager
- Unannounced audits of report forms, notebooks, and other data sheets by the Laboratory QA Officer
- Daily review of performance indicators such as blanks, surrogate recoveries, duplicate analyses, matrix spike analyses
- Checks on a random selection of calculations

- Review of all reports prior to and subsequent to computerized data entry
- Review and approval of final report.

Results from the analysis of project and blind audit QC samples will be calculated and evaluated as reported. If these results indicate data quality problems, immediate corrective action will be taken, and all data collected since previous QC audits will be carefully reviewed for validity.

### 9.2.3 Laboratory Data Reporting

Results of laboratory analyses of environmental samples will be reported in an appendix to the project report and will be grouped with the appropriate QC samples. If one of these check samples corresponds to more than one group of samples, it will be reported with each group for ease of data evaluation. All QC data pertinent to the environmental samples taken as part of the RFI will be reported and cross-referenced to each applicable sample including method blanks.

For those samples where confirmation of identification and quantitation is required by second-column chromatography, the confirmation data will be reported side-by-side with the first column data, separate from the environmental data. This is in addition to reporting the first column chromatographic analysis results with the other environmental data.

Laboratory QA/QC data will be reported separately from the environmental data but will be grouped by analysis method. Data necessary for calculation of percent recoveries will be presented along with the analytical results. The section containing QA/QC data also will include upper and lower control limits for percent recovery and relative percent difference for all analyses.

Complete evaluation of the analytical data requires that the data be reported completely and correctly. The following information is required for complete evaluation of the analytical data, and this information will be reported separately:

- Dates that samples were collected in the field
- Dates that samples were received in the laboratory
- Extraction and analysis dates for all samples
- Applicable holding times for each analysis
- Analysis dates for laboratory QC samples.

## 10. INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

### 10.1 Internal Field Quality Control Checks

Several types of internal QC checks will be used during the sampling. These will include, but are not limited to, the following:

- Equipment blanks
- Trip blanks
- Field blanks
- Field splits
- Replicates.

These QC samples will be maintained at a minimum of 10% of the total samples to be taken at the site. For example, for every 10 samples, two samples will be internal QC check samples.

Collection and analysis of field blanks, equipment washes, trip blanks, and field replicates are intended as QC checks on the integrity of sample collection and handling procedures and equipment decontamination procedures.

The success of these QC techniques may be negated by impurity of the water used to prepare check blanks. Field blanks, equipment blanks, and trip blanks will be prepared using ASTM Type II Reagent-grade water and sample containers randomly selected from the containers prepared for environmental samples. ASTM Type II Reagent-grade water is used to prepare the blanks, regardless of the environmental medium being sampled, for the following reasons:

- It mimics the physical characteristics of groundwater and surface water
- It mimics a reproducible fraction (moisture) of soils and sediments
- No reproducible, affordable material is available that mimics the clay and organic portion of soils and sediments
- An organic or aqueous reservoir is necessary for the absorption, dissolution, or solvation of organic and inorganic contaminants.

All sample containers will be provided by the contract laboratory.

#### 10.1.1 Trip Blanks

Trip blanks will be prepared at the beginning of the sampling trip by pouring ASTM Type II Reagent-grade water into prepared sample containers. These sample containers will be randomly selected from the supply of prepared sample containers. Sample containers will be filled to yield an appropriate sample volume for each type of analysis, resulting in a complete trip blank for the sampling event. The trip blanks will be prepared at the laboratory, shipped to the facility to be sampled with the remaining sample containers, stored with the remaining containers, transported to the sampling site with the remaining containers, and then shipped to the testing laboratory for analysis with the samples collected during the sampling event. The trip blanks will remain unopened throughout the transport and storage processes. Data from the analysis of trip blanks are used to assess contamination of sample containers during transport and storage and to assess contamination of samples during transport. One trip blank will be included in each shipping container.



### 10.1.2 Field Blanks

Field blanks (ambient condition samples) will be prepared at the beginning of each sampling event at each discrete sampling site by pouring ASTM Type II Reagent-grade water into prepared sample bottles. Three sample containers will be randomly selected from the supply of prepared sample containers. Sample containers will be filled to yield an appropriate sample volume for each type of analysis, resulting in a complete field blank for the sampling event. The field blanks will be handled and analyzed in the same manner as environmental samples. Because field blanks and environmental samples are collected under the same conditions, field blank analyses are used to indicate the presence of external contaminants that may have been introduced into the samples during collection. Field blanks may also become contaminated during transport, but this will be assessed by the use of trip blanks, as discussed above.

### 10.1.3 Equipment Blanks

Equipment blanks will be prepared for manual and small automated sampling equipment used to collect environmental samples (i.e., equipment blanks will not be collected from the drill rig or drilling equipment). Equipment blanks will be collected during the sampling day by pouring ASTM Type II Reagent-grade water into/through/over a decontaminated piece of sampling equipment, such as a split-spoon auger bailer, mixing bowl, or trowel and then dispensing the water into prepared sample bottles. These sample containers will be filled to yield an appropriate sample volume for each type of analysis, resulting in a complete equipment blank for the sampling event. The equipment blanks will be handled and analyzed in the same manner as environmental samples. Analysis of the equipment blanks will allow assessment of the adequacy of equipment decontamination procedures in preventing cross-contamination between sampling sites and samples.

The following QC samples will be collected for each day of sampling:

- One trip blank per container per sampling team for every batch of VOC samples
- One field blank per field sampling team for every VOC sampling round at a particular size of zone
- One field replicate for every 10 water samples at preselected monitoring points. A duplicate sample will be collected independently at a sampling location during a single act of sampling. Field duplicates will be indistinguishable from other analytical samples so that personnel performing the analyses are not able to determine which samples are duplicates. Field duplicates will be used to evaluate the reproducibility of analytical techniques.

#### 10.2 Internal Laboratory Quality Control Checks

The laboratory analyzes QC check samples equivalent to 10 to 20% of sample load in each project and/or laboratory function. Matrix spikes and matrix spike duplicate analyses are performed once every 20 samples. Method blanks are analyzed with each batch of prepared samples.

Control limits will be established for each parameter in accordance with standard protocols. Typical control limits are maintained at  $\pm 2\sigma$  for warning limits and  $\pm 3\sigma$  for control limits. A spike recovery result is out of control if it falls beyond the control limits or if it is the seventh successive value on the same side of the actual value. Table 10-1 lists the interferent and analyte elemental concentrations used for ICP interference check samples. Laboratory QC limits are presented in Tables 10-2 and 10-3.

TABLE 10-1. INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR ICP INTERFERENCE CHECK SAMPLES

Element	(mg/L)	Interferents	(mg/L)
Silver (Ag)	1.0	Aluminum (Al)	300
Barium (Ba)	0.3	Calcium (Ca)	300
Beryllium (Be)	0.5	Iron (Fe)	200
Cadmium (Cd)	1.0	Magnesium (Mg)	300
Cobalt (Co)	0.3		
Chromium (Cr)	0.3		
Copper (Cu)	0.3		
Manganese (Mn)	0.3		
Nickel (Ni)	1.0		
Iron (Fe)	1.0		
Vanadium (V)	0.5		
Zinc (Zn)	1.0		

TABLE 10-2. LABORATORY QUALITY CONTROL LIMITS GC/MS MATRIX SPIKE  
 COMPOUNDS--VOLATILE ORGANIC ANALYSIS (SW-846 Method 8240)

Parameter	Relative Percent Difference		Percent Recovery	
	Aqueous	Soil/Sediment	Aqueous	Soil/Sediment
1,1-Dichloroethene	14	22	61-145	59-172
Trichloroethene	14	24	71-120	62-137
Chlorobenzene	13	21	75-130	60-133
Toluene	13	21	76-125	59-139
Benzene	11	21	76-127	66-142

TABLE 10-3. LABORATORY QUALITY CONTROL LIMITS GC/MS SURROGATE SPIKING COMPOUNDS--VOLATILE ORGANIC ANALYSIS (SW-846 Method 8240)

Compound	Concentration in Sample/Extract		% Recovery Control Limits	
	Water ( $\mu\text{g/L}$ )	Low/Medium Soil ( $\mu\text{g/Kg}$ )	Water	Low/Medium Soil
Toluene-d <sub>8</sub>	50	50	88-110	81-117
4-Bromofluorobenzene	50	50	86-115	74-121
1,2-Dichloroethane-d <sub>4</sub>	50	50	76-114	70-121

In general, internal laboratory QC checks will consist of the following:

- **Organic Analyses.** Before processing any samples, the analyst will demonstrate, through the analysis of a reagent water blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent water blank will be processed as a safeguard against chronic laboratory contamination. The blank samples will be carried through all stages of the sample preparation and measurement steps.

A matrix spike and matrix spike duplicate will be analyzed for each analytical batch (up to 20 samples). The spiked samples will be carried through all stages of the sample preparation and measurement steps.

Each day that an analysis is performed, a daily calibration sample will be evaluated to determine if the chromatographic system is operating properly. If any changes are required to be made to the system (e.g., column changed), recalibration of the system may be necessary depending upon results of the daily calibration run subsequent to the changes. Surrogate recoveries are discussed below.

- **Surrogate Standards.** Surrogate standards will be added to all standards, method blanks, and samples prior analysis. Surrogate recoveries will be evaluated against the applicable control limits. When recovery data do not meet control limits, the analyst will check the surrogate recovery calculations, the integrity of the surrogate spike solution, and the instrument performance. If no indications of laboratory failure are uncovered, sample matrix interference will be assumed, and the data will be flagged as estimated concentrations.

- Inorganic Analyses--Inductively Coupled Argon Plasma (ICAP) Metals Scan. Two types of blanks will be required for the analysis. The calibration blank will be used in establishing the analytical curve, and the reagent blank will be used to correct for possible contamination resulting from varying amounts of the acids used in the sampling processing. A minimum of one laboratory blank will be run per sample batch to determine if contamination or any memory effects are occurring.

The instrument check standard will be prepared by the analyst by combining compatible elements at concentrations equivalent to the midpoint of their respective calibration curves.

The interferences check solution will be prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors.

The QC sample will be prepared in the same acid matrix as the calibration standards at 10 times the instrumental detection limits and in accordance with the instructions provided by the supplier.

For each analytical batch (up to 20 samples), a matrix spike and matrix spike duplicate will be analyzed. The spiked samples will be carried through all stages of the sample preparation and measurement steps.

Matrix standard addition is discussed below under "Inorganic Analysis--Atomic Absorption Spectrophotometric (AA) Analysis for Metals."

- Inorganic Analyses--ICP Interference Check Sample Analysis. To verify interelement and background correction factors, the contractor must analyze and report the results for the ICP Interference Check Samples at the beginning and end of each analysis run or a minimum of twice per 8-h working shift, whichever is more frequent, but not before initial calibration verification. The ICP Interference Check Samples will be obtained from EPA (EMSL/LV) if available and analyzed according to the instructions supplied with the Interference Check Sample.

The Interference Check Samples consist of two solutions: Solution A and Solution AB. Solution A consists of the interferences, and Solution AB consists of the analytes mixed with the interferences. An analysis of an Interference Check Sample consists of analyzing both solutions consecutively (starting with Solution A) for all wavelengths used for each analyte reported by ICP.

Results for the ICP analyses of Solution AB during the analytical runs must fall within the control limit of  $\pm 20\%$  of the true value for the analytes included in the Interference Check Samples. If not, the analysis must stop, the problem must be corrected, the instrument must be recalibrated, and the analytical samples analyzed since the last good run must be reanalyzed.

Sample results unaffected by interferences must be demonstrated for samples analyzed by SW Method 6010. Therefore, all soil digestates will be spiked with the complete list of the elements of interest. Matrix interference will be assumed for those samples for which the element recoveries are outside the 75 to 125% recovery window. Those samples will be analyzed by the method of standard addition.



- Inorganic Analyses--Atomic Absorption Spectrophotometric (AA) Analysis for Metals. A calibration curve will be prepared each day with a minimum of a reagent blank and three standards, verified by use of at least a reagent blank and one standard at or near the mid-range. Checks throughout the day must be within 20% of the original curve.

If 20 or more samples are analyzed per day, the working standard curve must be verified by running an additional standard at or near the mid-range for every 10 samples.

For each analytical batch (up to 20 samples), a method blank, matrix spike, and matrix spike duplicate will be analyzed. The blank and spiked samples will be carried through all stages of the sample preparation and measurement steps.

Where the sample matrix is so complex that viscosity, surface tension, and components cannot be accurately matched with standards, the method of standard addition will be used.

## 11. PERFORMANCE AND SYSTEM AUDITS

### 11.1 Performance Audits

#### 11.1.1 Field Operations

Field performance audits will be performed on an ongoing basis during the project as field data are generated, reduced, and analyzed. All numerical analyses, including manual calculations, will be documented. All records of numerical analyses will be legible, of reproduction-quality, and complete enough to permit logical reconstruction by a qualified individual other than the originator.

The primary objective of a field performance audit is to determine the status of sampling operations. Emphasis will be placed on the following items:

- Verifying that operational aspects and procedures are in accordance with the protocols and the Data Collection Quality Assurance Plan
- Verifying the collection of all samples including duplicates and field plans
- Verifying that documentation is in order and sufficient to establish the collection location of any sample collected
- Determining discrepancies that exist and initiating corrective action as appropriate
- Collecting independent samples.

The on-site field audit will inspect sample records and equipment including the following:

- Chain-of-custody forms
- Sample tags
- Unit description forms
- Logbooks.

Operational procedures inspected should include the following items:

- Sampling procedures
- Equipment
- Techniques
- Decontamination
- Collection of duplicate and field blank samples
- Security
- Sample storage and transportation
- Containers
- Contaminated waste storage and disposal
- Unit description for entries.

### 11.1.2 Laboratory Operations

Laboratory performance audits will be conducted on a routine basis by the subcontractor Laboratory QA Officer and will include the following items:

- Verification of written procedures and analyst understanding
- Verification and documentation of procedures and documents
- Weekly unannounced inspection of the sample handling group
- Weekly unannounced inspection of the analytical process record keeping
- Review of 25% of all analytical data and calculations.

### 11.2 Systems Audits

The purpose of the systems audit is to ensure that the QA system planned for the project is in place and functioning. The system audit is an overall evaluation of the sampling project and will perform the following functions:

- Verify that sampling methodology is being performed in accordance with program requirements
- Check on the use of appropriate QC measures
- Check methods of sample handling, i.e., packaging, labeling, preserving, transporting, and archiving in accordance with program requirements

- Identify any existing quality problems
- Check program documentation, i.e., records (site description, chain-of-custody collection and analytical tags, field and sample blank logbooks, and field work sheets)
- Initiate corrective action if a problem is identified
- Assess personnel experience and qualifications, if required
- Follow-up on any corrective action previously implemented
- Provide debriefings for sampling team and sample bank personnel
- Provide a written evaluation of the sampling and the sample bank program.

#### 11.2.1 Field Operations

Periodically, system audits of site activities will be accomplished by an inspection of major field site activities. This audit is comprised of comparison, by the audit team, of current field practices with standard procedures. After the audit, any deficiencies will be discussed with the field staff, and corrective measures will be identified. If any of these deficiencies might affect the integrity of the samples being collected, the audit team will inform the field staff immediately, so that any corrections can be made. The audit team will consist of a senior chemist and field-supervisory level geologist.

### 11.2.2 Laboratory Operations

Laboratory system audits are qualitative audits of the measurement systems to ensure their proper care and use. These audits will be performed on a regular basis and will include review of the following:

- Analytical and support instrumentation maintenance logs
- Analytical and support instrumentation calibration logs
- Refrigerator and freezer temperature records
- Distilled/deionized water supply records
- Sample tracking system
- Standard tracking system
- Reagent chemical log-in, tracking, and disposal.

Another laboratory system audit is the on-site audit by the COCA QA Office. During this audit, laboratory records and procedures will be inspected for completeness, accuracy, precision, and adherences to prescribed methods. This inspection will include the following:

- Following the sample chain-of-custody from time of sample receipt through all analysis steps to data reduction, validation, and report generation
- Examination of maintenance and calibration logbooks to ensure that maintenance and calibration are performed on a scheduled basis

- Examination of procedures and records for data calculation transfer and validation
- Spot-check of calibration, QC, and sample data from selected instruments for selected days to ensure precision, accuracy, and completeness
- Inspection of storage areas, glassware preparation area, and distilled/deionized water system records and procedures
- Examination of QA procedures and records (standard and spike solution logbooks and storage areas, control charts, QA manuals).

An additional laboratory system audit will be performed by spot-checking analytical data from the laboratory. During the time that analytical activities are under way, the laboratory will be requested to send the COCA QA Office all raw data for the analyses of a fraction of the samples.

## 12. EQUIPMENT MAINTENANCE

### 12.1 Field Equipment

Preventive maintenance will be carried out on all field equipment prior to its use. This preventive maintenance will include the following:

- Checking sampling equipment for proper operation
- Maintaining a sufficient stock of spare sampling equipment, parts, and supplies to minimize down time due to equipment malfunctions
- Preparing sample containers in advance; extra containers will be provided in case of breakage, sample contamination, etc.
- Checking batteries regularly.

Field personnel will be strongly cautioned that these instructions are only for general purposes. If the field crews are unable to repair equipment in the field within a reasonable amount of time, the Project Manager will be notified. For specific preventive maintenance procedures, consult the manual for the appropriate instrument.

### 12.2 Laboratory Equipment

#### 12.2.1 Gas Chromatography/Mass Spectrometry

Thorough preventive maintenance will be performed at quarterly intervals. The preventive maintenance will include the following services:

- Changing the water filter
- Changing the mechanical pump oil



- Cleaning the analyzer as required
- Cleaning the ionizer as required
- Cleaning the mass filter as required
- Cleaning the electron multiplier as required
- Calibrating the system
- Checking the module function
- Analyzing sensitivity
- Changing disc drive filters.

In addition, the GC/MS system resolution and performance will be evaluated and calibrated using BFB to satisfy the EPA-specified criteria. GC/MS system sensitivity will be tested by a 50-ng injection of BFB for the electron impact ionization mode.

#### 12.2.2 Atomic Absorption Spectrophotometry (AAS)

In AAS, a number of instrumental variables will be checked for performance consistency as a part of preventive maintenance: instrument warmup, burner alignment, gas flow, lamp intensity, slit width, wavelength, matrix effects, aspiration time, and aspiration rate. Preventive maintenance procedures include a minimum warmup period of 30 minutes. The hollow cathode tube will be aligned to produce the maximum emitted light to the detector. In flameless AA, the inert gas flow inside the furnace is optimized to ensure maximum sensitivity.

The digital readout values obtained for the standard curve of each element will be checked to ensure that they fall within a specified range. If readings are excessively low, the operator will check gas flows, burner or cell alignment, wavelength, slit width, photomultiplier voltage, and lamp intensity prior to analysis. Burner heads, nebulizers, quartz cells, and reduction flasks will be cleaned according to manufacturer's instructions whenever excessive electronic noise is apparent or whenever indicated by visual inspection. Tygon tubing will be replaced when deterioration is apparent. Optical lenses will be cleaned periodically.

### 12.2.3 General Laboratory Equipment

The most prevalent pieces of equipment in this category are analytical balances of various capacities and operational modes. The balances will be cleaned and adjusted annually by a licensed specialist and officially recorded as a verification of performance. All combination pH electrodes will be stored in a pH 4 buffer solution. A high-purity water system will be used to produce water that is free from particulates and total dissolved solids and has a purity of 18 megohm/cm. The purity will be checked before every use with a resistivity meter. When the water purity falls 12 megohm/cm purity, new deionizing and filtration cartridges will be installed.

13. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA  
PRECISION, ACCURACY, AND COMPLETENESS

A percentage of the samples will be split in the field and sent to the laboratory under different labels. This will provide a routine check of the precision of the laboratory and sample homogeneity. Accuracy measurement will be available by analyzing laboratory QC data and examining blank and spike results. This QC data will include interlaboratory checks and laboratory blanks, standards, and spikes.

The Quality Assurance Plans for the current contract laboratories are attached as Appendix A. Should the contract laboratory change, all interlaboratory checks will be conducted for the new laboratory.

13.1 Data Assessment Procedures for Field Data

13.1.1 Precision

All liquid samples will be tested for temperature, pH, specific conductance, and alkalinity using procedures developed specifically for each measurement. Duplicate and replicate samples analyzed by the laboratory will assess the precision of the sampling effort. Control limits for duplicate/replicate RPDs will be set at 0 to 20% to provide an initial guide. Once a sufficient amount of replicate data becomes available, field precision control charts will be constructed like the laboratory precision charts. For any given concentration, the mean and standard deviation of the replicates will be calculated. The mean is the centerline. Data from each sample set will be pooled with previous sample sets to generate control and warning limits for the next set. Warning and control limits for water samples will be set at  $\pm 2\sigma$  and  $\pm 3\sigma$ , respectively. Control limits for solid samples will be more liberally established due to that matrix's heterogeneity. Data outside any control limit are subject to QA review.

### 13.1.2 Accuracy

Field instruments are calibrated daily or more frequently, if needed, to ensure accuracy of the measurement of field parameters. Specifically, the pH measurement must be accurate to  $\pm 0.1$  unit, temperature must be accurate to  $\pm 0.5^{\circ}\text{C}$ , and specific conductance must be accurate to  $\pm 10$  umhos/cm. Each well will be purged until the above parameters are stable within the specified limits.

All blanks associated with each sample set will be analyzed and evaluated for cross-contamination. Blank contamination and the resulting corrective action will be assessed on an individual basis.

### 3.1.3 Completeness

The Project Geologist will be responsible for ensuring that all field instrumentation and equipment are functioning properly and calibrated according to set procedures and that all data are recorded accurately and legibly. In addition, the Project Geologist must ensure all sites are sampled for all of the specified analyses, sufficient sample volume has been provided to complete those analyses, and all of the QA samples have been included with each sample set. For the purposes of this project, the goal for completeness for each sample set shipped to the laboratory will be 100%. The minimum acceptable completeness limit is 95%.

## 13.2 Data Assessment Procedures for Laboratory Data

### 13.2.1 Accuracy

Accuracy control charts typically will be generated as follows, unless otherwise directed by the requirements of a project:

- Accuracy will be determined from the results of matrix spike and matrix spike duplicates performed at the rate of one set every 20 samples. Control limit for recovery will be defined by historical laboratory data.
- Using historical laboratory data, a control chart will be developed. From this control chart the control limits will be defined as the mean (center line)  $\pm 3$  standard deviations.

#### 13.2.2 Precision

Precision will be based on the results of the relative percent differences, as calculated from the percent recoveries of the matrix spike and matrix spike duplicates associated with this project for each parameter of interest. The control limits for precision will be based on historical laboratory data.

#### 13.2.3 Completeness

Completeness will be expressed as the percentage of the amount of valid data obtained compared to the amount of data expected. For the percent recovery required to ensure data accuracy, unless specified in the method, a minimum 85% completeness will be the goal for the project when sample values are above 10 x minimum detection limit (MDL) and 60% when sample values are below 10 x MDL. For metals, however, a minimum 90% completeness will be the goal for the contract when sample values are above 10 x MDL and 80% when values are below 10 x MDL.

## 14. CORRECTIVE ACTION

### 14.1 Field Corrective Action

The initial responsibility for monitoring the quality of field measurements lies with the field personnel. The Field Team Leader or Project Geologist is responsible for verifying that all QC procedures are followed. This requires that the Project Geologist assess the correctness of the field methods and the ability to meet QA objectives. If a problem occurs that might jeopardize the integrity of the project or cause some QA objective not to be met, the Project Geologist will notify the Project Manager, the Laboratory Manager, and the Program Manager, if appropriate. Corrective action measures will then be decided upon and implemented. Table 14-1 lists corrective action procedures for responding to field measurements problems. Figure 14-1 is the field corrective action form. The Project Geologist will document the problem, the corrective action, and the results. Copies of the documentation will be provided to the Program Manager, the Project Manager, and the COCA QA Office.

### 14.2 Laboratory Corrective Action

The need for corrective action comes from several sources: equipment malfunction; failure of internal QA/QC checks, method blank contamination, failure of performance or system audits; and noncompliance with QA requirements.

When measurement equipment or analytical methods fail QA/QC, the problem will immediately be brought to the attention of the Analytical Task Leader and subcontractor Laboratory QA Officer. If failure is due to equipment malfunction, the equipment will be repaired, precision and accuracy will be reassessed, and the analysis will be rerun. All attempts

TABLE 14-1. CORRECTIVE ACTION PROCEDURES ACCORDING TO SITUATION<sup>a</sup>

Situation	Field Objective Affected	Corrective Action Procedure
Equipment malfunction	Equipment is calibrated and operating properly	<ul style="list-style-type: none"> <li>• Notification of site supervisory personnel</li> <li>• Repair or replace malfunctioning parts</li> <li>• Resample or repeat task if necessary</li> <li>• Document to Project Manager and COCA QA Office</li> </ul>
Incorrect sample collection procedures	Samples are taken according to standard operating procedures	<ul style="list-style-type: none"> <li>• Notification of site supervisory personnel</li> <li>• Review of situation and correct procedures</li> <li>• Notify Laboratory Manager</li> <li>• Re-sample</li> <li>• Document to Project Director and COCA QA Office</li> </ul>
Insufficient sample volume collected	Sufficient sample volume is provided to maintain sample integrity and so that all required analyses can be conducted	<ul style="list-style-type: none"> <li>• Notification of site supervisory personnel by the laboratory manager</li> <li>• Review site affected and impact of samples on site characterization</li> <li>• Resample to provide more sample volume if necessary</li> <li>• Document to Project Director and COCA QA Office</li> </ul>
Incorrective measurement data	Measurements are conducted according to standard operating procedures	<ul style="list-style-type: none"> <li>• Notification of site supervisory personnel</li> <li>• Review situation and correct procedures</li> <li>• Conduct tests again</li> <li>• Document to Project Director and COCA QA Office</li> </ul>

a. Situations requiring corrective action are too numerous to comprehensively list here. This table is provided to illustrate several examples.

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TABLE 14-1. (continued)

Situation	Field Objective Affected	Corrective Action Procedure
Field contamination (equipment blanks only)	Acquisition of defensible data	<ul style="list-style-type: none"> <li>• Notification of Project Director and COCA QA Office by the laboratory manager</li> <li>• Notify site supervisory personnel</li> <li>• Review decontamination procedures and correct deficiencies</li> <li>• Document to file for final report note: no resampling necessary</li> </ul>
Field contamination (field and equipment blanks only - VOC analysis)	Acquisition of defensible data	<ul style="list-style-type: none"> <li>• Notification of Project Director and COCA QA Office by the laboratory manager</li> <li>• Notify site supervisory personnel</li> <li>• Review ASTM Type II reagent water sources, test water, onsite, and replace if necessary</li> <li>• Document to file for final report.</li> </ul>
Field contamination (interfering compounds detected in all blanks, except the laboratory method blank, and corresponding environmental samples) - VOC analysis	Acquisition of defensible, justifiable data	<ul style="list-style-type: none"> <li>• Notification of Project Director and COCA QA Office by the laboratory manager</li> <li>• Notify site supervisory personnel</li> <li>• Review situation, determine source of extent of affected samples</li> <li>• Executive decision by Project Director as to course of action</li> <li>• Resample if necessary</li> <li>• Document to file for final report</li> </ul>

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TABLE 14-1. (continued)

Situation	Field Objective Affected	Corrective Action Procedure
No QC sample support of sample batch	Acquisition of defensible, justifiable data	<ul style="list-style-type: none"> <li>• Notification of site supervisory personnel by the laboratory manager</li> <li>• Mail appropriate samples if available</li> <li>• If samples are not available, the Project Director and COCA QA Office will be notified</li> <li>• Review site affected and impact of samples on site characterization</li> <li>• Resample if necessary</li> <li>• Document to file for final report</li> </ul>
Duplicate or replicate RPDs outside of control limits	Acquisition of defensible, justifiable data	<ul style="list-style-type: none"> <li>• Notification of Project Director and COCA QA Office by laboratory manager</li> <li>• Re-analysis of in-house samples by the laboratory</li> <li>• Notification of site supervisory personnel if RPDs remain outside of control limits</li> <li>• Review site affected and impact of samples on site characterization</li> <li>• Executive decision by Project Director concerning the importance of affected data</li> <li>• Resample if necessary</li> <li>• Document to Project Director and COCA QA Office</li> </ul>
Expired samples	Acquisition of defensible, justifiable data	<ul style="list-style-type: none"> <li>• Notification of site supervisory personnel by laboratory manager</li> <li>• Notification of site supervisory personnel by Project Manager</li> <li>• Review of site affected and impact of samples on site characterization</li> <li>• Executive decision by Project Director concerning the importance of the affected data</li> <li>• Resample if necessary</li> <li>• Document to Project Director and COCA QA Office</li> </ul>

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will be made to reanalyze all affected parts of the analysis so that in the end, the product is not affected by failure of QA requirements. All incidents of QA failure and the respective corrective action task will be documented, and reports on the failure will be placed in the appropriate project file. Also, corrective action will be taken promptly for deficiencies noted during the spot-checks of raw data. Table 14-1 lists corrective action procedures for responding to laboratory problems. Figure 14-2 is the laboratory corrective action form. As soon as sufficient time has elapsed for corrective action to be implemented, evidence of the correction will be presented. Documentation on the corrective action measure will be forwarded to the COCA QA Office and the Project Manager.

Laboratory corrective action will be initiated to ensure data quality meets the criteria outlined in the written laboratory QA plans. The laboratory chosen will use an EPA-approved QA plan and will participate in round-robin EPA contract laboratory-type testing.

EG&G Idaho Project:

LABORATORY CORRECTIVE ACTION

DESCRIPTION OF EVENT:

Date and time recognized: \_\_\_\_\_ By: \_\_\_\_\_

Date of actual occurrence: \_\_\_\_\_ By: \_\_\_\_\_

Date corrected: \_\_\_\_\_ By: \_\_\_\_\_

ANALYTICAL METHOD:

Analyte: \_\_\_\_\_ Method: \_\_\_\_\_

Analyst: \_\_\_\_\_ Section Manager: \_\_\_\_\_

Description of QC problem encountered:

Corrective/Preventative Action Taken:

Contract/Project Number: \_\_\_\_\_

Samples affected (Lab ID):

SIGNATURE:

Analyst: \_\_\_\_\_ Date: \_\_\_\_\_

Section Manager: \_\_\_\_\_ Date: \_\_\_\_\_

QA Officer: \_\_\_\_\_ Date: \_\_\_\_\_

Laboratory Manager: \_\_\_\_\_ Date: \_\_\_\_\_

Figure 14.2. Laboratory Corrective Action Form

15. QUALITY ASSURANCE REPORTS TO MANAGEMENT

15.1 Field Quality Assurance Reports

The Field QA Officer will prepare a Field Audit report.

15.2 Laboratory Quality Assurance Reports

The Laboratory QA Officer will prepare a written report on the analytical data for the Project Manager. The report will review the validity, quality, and completeness of the data and, if necessary, will make recommendations for corrective action, further sampling, or additional analytical data.

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APPENDIX A  
QUALITY ASSURANCE PLANS  
FOR THE CURRENT CONTRACT LABORATORIES

Partial Appendix IX Analyte Listing  
 CLP Target Compound List VOA Analytes

Based on 40 CFR 264, Federal Register 52 (131), p. 25942

Common Name	CAS Number	Fraction	Meth.	PQL μg/l
Chloromethane/Methyl chloride	74-87-3	VOA	8240	10
Bromomethane/Methyl bromide	74-83-9	VOA	8240	10
Vinyl chloride	75-01-4	VOA	8240	5
Chloroethane/Ethyl chloride	75-00-3	VOA	8240	10
Methylene chloride/Dichloromethane	75-09-2	VOA	8240	5
Acetone	67-64-1	VOA	8240	100
Carbon disulfide	75-15-0	VOA	8240	5
1,1-Dichloroethene/1,1-Dichloroethylene	75-35-4	VOA	8240	5
1,1-Dichloroethane	75-34-3	VOA	8240	5
1,1-Dichloroethene (total)	156-60-5	VOA	8240	5
Chloroform	67-66-3	VOA	8240	5
1,2-Dichloroethane/Ethylenedichloride	107-06-2	VOA	8240	5
2-Butanone/Methyl ethyl ketone/MEK	78-93-3	VOA	8240	100
1,1,1-Trichloroethane/Methyl chloroform	71-55-6	VOA	8240	5
Carbon tetrachloride	56-23-5	VOA	8240	5
Vinyl acetate	108-05-4	VOA	8240	5
Bromodichloromethane	75-27-4	VOA	8240	5
1,2-Dichloropropane	78-87-5	VOA	8240	5
cis-1,3-Dichloropropene	10061-01-5	VOA	8240	5
Trichloroethene	79-01-6	VOA	8240	5
Dibromochloromethane/Chlorodibromomethane	124-48-1	VOA	8240	5
1,1,2-Trichloroethane	79-00-5	VOA	8240	5
Benzene	71-43-2	VOA	8240	5
trans-1,3-Dichloropropene	10061-02-6	VOA	8240	5
Bromoform/Tribromomethane	75-25-2	VOA	8240	5
4-Methyl-2-pentanone/MIBK	108-10-1	VOA	8240	50
2-Hexanone	591-78-6	VOA	8240	50
Tetrachloroethene/PCE/Tetrachloroethylene	127-18-4	VOA	8240	5
1,1,2,2-Tetrachloroethane	79-34-5	VOA	8240	5
Toluene	108-88-3	VOA	8240	5
Chlorobenzene	108-90-7	VOA	8240	5
Ethylbenzene	100-41-4	VOA	8240	5
Styrene	100-42-5	VOA	8240	5
Xylene (total)	1330-20-7	VOA	8240	5



Appendix IX Analyte Listing  
Inorganic Analytes

Based on 40 CFR 264, Federal Register 52 (131), p. 25942

<u>Common Name</u>	<u>CAS Number</u>	<u>Fraction</u>	<u>Meth.</u>	<u>PQL</u> <u>µg/l</u>
Antimony	7440-36-0	INORG	6010	300
Arsenic	7440-38-2	INORG	7060	10
Barium	7440-39-3	INORG	6010	20
Beryllium	7440-41-7	INORG	6010	3
Cadmium	7440-43-9	INORG	6010	40
Chromium	7440-47-3	INORG	6010	70
Cobalt	7440-48-4	INORG	6010	70
Copper	7440-50-8	INORG	6010	60
Lead	7439-92-1	INORG	7421	10
Mercury	7439-97-6	INORG	7470	2
Nickel	7440-02-0	INORG	6010	50
Selenium	7782-49-2	INORG	7741	20
Silver	7440-22-4	INORG	6010	70
Thallium	7440-28-0	INORG	7841	10
Vanadium	7440-62-2	INORG	6010	80
Zinc	7440-66-6	INORG	6010	20
Cyanide	57-12-5	INORG	9010	40
Tin	7440-31-5	INORG	6010	1000
Sulfide	18496-25-8	INORG	9030	200

APPENDIX B  
RECOMMENDATION FOR SAMPLING AND PRESERVATION  
OF WATER SAMPLES ACCORDING TO MEASUREMENT



## APPENDIX B

RECOMMENDATION FOR SAMPLING AND PRESERVATION  
OF WATER SAMPLES ACCORDING TO MEASUREMENT<sup>a</sup>

Measurement	Vol. Req. (ml)	Container <sup>b</sup>	Preservative <sup>c,d</sup>	Holding Time <sup>e</sup>
<u>Physical Properties</u>				
Conductance	100	P,G	Cool, 4.	Analyze immediately
pH	25	P,G	None Req.	Analyze immediately
Temperature	1,000	P,G	None Req.	Analyze immediately
<u>Volatile Organics</u>	80(2x40)	G	Cool, 4°C	12 days
<u>Metals</u>				
Total Recoverable	100	P,G	HNO <sub>3</sub> to pH <2	6 mos.
Mercury (Total)	100	P,G	HNO <sub>3</sub> to pH <2	28 days
<u>Inorganics, Non-Metallics</u>				
Alkalinity	100	P,G	Cool, 4°C	Analyze immediately
Chloride	50	P,G	None. Req.	28 days
Fluoride	300	P,G	None. Req.	28 days
<u>Nitrogen</u>				
Nitrate	100	P,G	Cool, 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	14 days
Sulfate	50	P,G	Cool, 4°C	28 days

a. More specific instructions for preservation and sampling are found with procedures as detailed in EPA-600/4-79-020, revised March 1983, and in the Federal Register, Vol. 49, No. 209, Oct. 26, 1984, EPA 40 CFR Part 136, Table II.

## APPENDIX B

### RECOMMENDATION FOR SAMPLING AND PRESERVATION OF WATER SAMPLES ACCORDING TO MEASUREMENT<sup>a</sup> (continued)

b. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.

c. Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4.C until compositing and sample splitting is completed.

d. When any sample is to be shipped by common carrier or sent through the United States mail, it must comply with the Department of Transportation Hazardous Materials Regulations (40 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table 1, the Office of Hazardous Materials, . Materials Transportation Bureau, Department of Transportation, has determined that the Hazardous Materials Regulations do not apply to the following materials: hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

e. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.

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APPENDIX C  
RECOMMENDATION FOR SAMPLING AND PRESERVATION  
OF SOIL SAMPLES ACCORDING TO MEASUREMENT



APPENDIX C

RECOMMENDATION FOR SAMPLING AND PRESERVATION  
OF SOIL SAMPLES ACCORDING TO MEASUREMENT<sup>a</sup>

Measurement	Vol. Req. (oz)	Container <sup>b</sup>	Preservative <sup>c,d</sup>	Holding Time <sup>e</sup>
<u>Metals</u>				
Total Recoverable	32	G	Cool, 4°C	6 mos.
Extraction Procedure Toxicity	32	G	Cool, 4°C	6 mos.
<u>Organics</u>				
VOCs and Xylenes, EPA Method 8240	32	G	Cool, 4°C	14 days

a. More specific instructions for preservation and sampling are found with procedures as detailed in EPA-600/4-79-020, revised March 1983, and in the Federal Register, Vol. 49, No. 209, Oct. 26, 1984, EPA 40 CFR Part 136, Table II.

b. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.

c. Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

d. When any sample is to be shipped by common carrier or sent through the United States mail, it must comply with the Department of Transportation Hazardous Materials Regulations (40 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation, has determined that the Hazardous Materials Regulations do not apply to the following materials: hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).



APPENDIX C

RECOMMENDATION FOR SAMPLING AND PRESERVATION  
OF SOIL SAMPLES ACCORDING TO MEASUREMENT<sup>a</sup>  
(continued)

e. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.

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APPENDIX D  
REFERENCES



## APPENDIX D

### REFERENCES

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APPENDIX E  
40 CFR 264, APPENDIX IX  
ANALYTE LISTING



Appendix IX Analyte Listing  
CLP VOA Analytes

Based on 40 CFR 264, Federal Register 52 (131), p. 25942

Common Name	CAS Number	Fraction	Meth.	PQL µg/l
Chloromethane/Methyl chloride	74-87-3	VOA	8240	10
Bromomethane/Methyl bromide	74-83-9	VOA	8240	10
Vinyl chloride	75-01-4	VOA	8240	5
Chloroethane/Ethyl chloride	75-00-3	VOA	8240	10
Methylene chloride/Dichloromethane	75-09-2	VOA	8240	5
Acetone	67-64-1	VOA	8240	100
Carbon disulfide	75-15-0	VOA	8240	5
1,1-Dichloroethene/1,1-Dichloroethylene	75-35-4	VOA	8240	5
1,1-Dichloroethane	75-34-3	VOA	8240	5
1,1-Dichloroethene (total)	156-60-5	VOA	8240	5
Chloroform	67-66-3	VOA	8240	5
1,2-Dichloroethane/Ethylenedichloride	107-06-2	VOA	8240	5
2-Butanone/Methyl ethyl ketone/MEK	78-93-3	VOA	8240	100
1,1,1-Trichloroethane/Methyl chloroform	71-55-6	VOA	8240	5
Carbon tetrachloride	56-23-5	VOA	8240	5
Vinyl acetate	108-05-4	VOA	8240	5
Bromodichloromethane	75-27-4	VOA	8240	5
1,2-Dichloropropane	78-87-5	VOA	8240	5
cis-1,3-Dichloropropene	10061-01-5	VOA	8240	5
Trichloroethene	79-01-6	VOA	8240	5
Dibromochloromethane/Chlorodibromomethane	124-48-1	VOA	8240	5
1,1,2-Trichloroethane	79-00-5	VOA	8240	5
Benzene	71-43-2	VOA	8240	5
trans-1,3-Dichloropropene	10061-02-6	VOA	8240	5
Bromoform/Tribromomethane	75-25-2	VOA	8240	5
4-Methyl-2-pentanone/MIBK	108-10-1	VOA	8240	50
2-Hexanone	591-78-6	VOA	8240	50
Tetrachloroethene/PCE/Tetrachloroethylene	127-18-4	VOA	8240	5
1,1,2,2-Tetrachloroethane	79-34-5	VOA	8240	5
Toluene	108-88-3	VOA	8240	5
Chlorobenzene	108-90-7	VOA	8240	5
Ethylbenzene	100-41-4	VOA	8240	5
Styrene	100-42-5	VOA	8240	5
Xylene (total)	1330-20-7	VOA	8240	5



Appendix IX Analyte Listing  
Inorganic Analytes

Based on 40 CFR 264, Federal Register 52 (131), p. 25942

<u>Common Name</u>	<u>CAS Number</u>	<u>Fraction</u>	<u>Meth.</u>	<u>PQL</u> <u>µg/l</u>
Antimony	7440-36-0	INORG	6010	300
Arsenic	7440-38-2	INORG	7060	10
Barium	7440-39-3	INORG	6010	20
Beryllium	7440-41-7	INORG	6010	3
Cadmium	7440-43-9	INORG	6010	40
Chromium	7440-47-3	INORG	6010	70
Cobalt	7440-48-4	INORG	6010	70
Copper	7440-50-8	INORG	6010	60
Lead	7439-92-1	INORG	7421	10
Mercury	7439-97-6	INORG	7470	2
Nickel	7440-02-0	INORG	6010	50
Selenium	7782-49-2	INORG	7741	20
Silver	7440-22-4	INORG	6010	70
Thallium	7440-28-0	INORG	7841	10
Vanadium	7440-62-2	INORG	6010	80
Zinc	7440-66-6	INORG	6010	20
Cyanide	57-12-5	INORG	9010	40
Tin	7440-31-5	INORG	6010	1000
Sulfide	18496-25-8	INORG	9030	200