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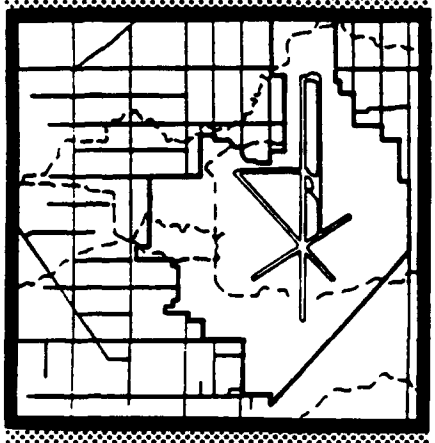
INSTALLATION RESTORATION PROGRAM (IRP)
STAGE 3

# QUALITY ASSURANCE PROJECT PLAN



for McCLELLAN AFB, CALIFORNIA

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AUGUST 1992

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INSTALLATION RESTORATION PROGRAM (IRP)
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# QUALITY ASSURANCE PROJECT PLAN

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This document is the McClellan AFB RI/FS Quality Assurance Project Plan (QAPP), and contains procedures for the collection, analysis, and documentation for all anticipated sampling and analysis tasks. These include groundwater monitoring well installation, geophysical techniques, and sampling and analysis for groundwater, surface water, sediment, soil and soil vapor.

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# PREFACE

This document was prepared by Radian Corporation for the Air Force Center for Environmental Excellence, Environmental Services Office, Environmental Restoration Division, under Air Force Contract No. F33615-90-D-4013, Delivery Order 0004.

This document is a basewide Quality Assurance Project Plan (QAPP) for the McClellan Air Force Base Installation Restoration Program. It has been prepared according to Air Force and U.S. EPA guidelines. This QAPP is issued for use by contractors conducting RI/FS activities at McClellan AFB under the Interagency Agreement between the Air Force, U.S. EPA, and State of California, dated May, 1990. All currently used sampling, analytical, and quality assurance/quality control procedures and specifications are presented. These procedures have been established to promote consistency and comparability, and to assure the defensibility of the data produced by multiple contractors and analytical laboratories. Implementation of these procedures will assure that consistent, technically sound decisions about needs for further action at a site, or between Operable Units will be made.

While this document provides a reference and guidance to promote technical consistency and comparability for basewide RI/FS activities, compliance with these procedures should be assured through protocols established within each contractor's organization, and specified in the Statements of Work for the activities to be performed.

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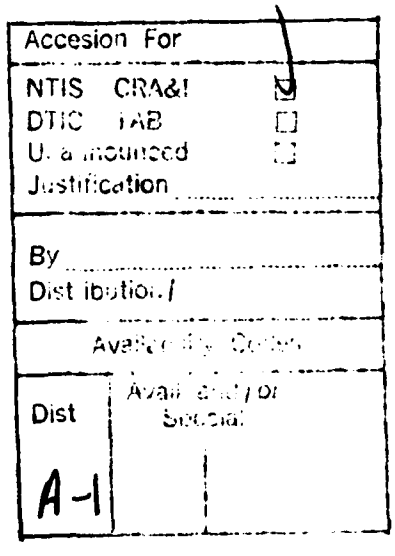


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# 1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) presents in specific terms, the policies, organization, functions, and specific quality assurance (QA) and quality control (QC) activities designed to achieve the data quality goals for the McClellan Air Force Base (AFB) Remedial Investigation/Feasibility Study (RI/FS). The U.S. Environmental Protection Agency (U.S. EPA) quality assurance policy requires a written and approved QAPP for every monitoring and measurement project, mandated or supported by the U.S. EPA through regulations, contracts, or other formalized means, not currently covered by regulation. Guidelines followed in the preparation of this plan are set out in U.S. EPA's Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (December 1980), and U.S. EPA Region 9 QAPP: Guidance for Preparing QAPPs for Superfund Remedial Projects (September 1989). Other documents that have been referenced for this plan include U.S. EPA Region IX Guidance for Preparing Quality Assurance Project Plans for Superfund Remedial Projects (September 1989), U.S. EPA's Guidance on Remedial Investigations Under CERCLA (June 1985), and the Guidance on Feasibility Studies Under CERCLA (June 1985), Compendium of Superfund Field Operations Methods (September 1987), Data Quality Objectives for Remedial Response Activities (March 1987), Guidelines for Assessing and Reporting Data Quality for Environmental Measurements (January 1983) and the Air Force Handbook to Support the Installation Restoration Program Statements of Work for Remedial Investigation/Feasibility Studies (May 1991).

This QAPP supports the ongoing efforts of the Installation Restoration Program (IRP). The IRP was developed by the U.S. Department of Defense (DOD) in 1981 to investigate hazardous material disposal sites on DOD facilities, such as McClellan AFB. McClellan AFB integrated the ongoing IRP with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980 as amended by Superfund Amendments and Reauthorization Act of 1985 (SARA); National Oil and Hazardous Substances Contingency Plan (NCP); pertinent provisions of the Resource Conservation and Recovery Act (RCRA) statutes; Executive Order 12580; the Defense Environmental Restoration Program (DERP); and all applicable or relevant and appropriate state laws and regulations.

The Air Force, the U.S. EPA and the California Environmental Protection Agency - Department of Toxic Substances Control (formerly DHS) signed an Interagency Agreement (IAG) in May 1990, which establishes the process for involving federal and state

regulatory agencies, and the public in the McClellan AFB response action process. The IAG sets schedules for completing specific program tasks including:

- Remedial Investigations;
- Feasibility Studies;
- Response actions; and
- Operation and maintenance activities related to response actions.

This detailed QAPP has been prepared for use by contractors who perform environmental services at McClellan AFB to ensure that the RI/FS produces data that are scientifically valid and defensible. The establishment and documentation of these procedures will also ensure that the data are collected, reviewed, and analyzed in a consistent manner. This is especially important because there are 171 identified potential sites on the base, with a variety of environmental media to be sampled during discrete sampling and analysis tasks during the RI. This QAPP is a comprehensive plan that will be used to guide data collection and reporting efforts for current and future field tasks performed by various contractors. Although specific sampling strategies and detailed Sampling and Analysis Plans (SAPs) for many of the tasks have not been developed at this time, the QAPP includes all currently anticipated hydrogeologic studies, field sampling procedures, analytical methods, and QA documentation procedures related to sampling surface water, groundwater, soil, sediment, and soil gas. As specific SAPs are developed for various field activities, the QAPP will be referenced or excerpted as necessary. An applicability checklist (Figure 1-1) developed from the comprehensive QAPP outline, and an applicability statement (Figure 1-2) will be completed, signed, and submitted along with each SAP to indicate that all appropriate sampling, analytical, and QA procedures are included and will be followed. This procedure will be required for subcontractors as well. This format is necessary because much of the work has not been detailed, SAPs have not been developed, and subcontractors are not known at this time. In this manner, we will reduce redundancy and maintain better control over all aspects of the RI/FS.

This QAPP is intended to guide RI/FS activities for a variety of sampling and analytical efforts, and therefore, does not contain specific Data Quality Objectives (DQOs). The DQOs will be developed for each site and field effort by considering the results of the Preliminary Assessment for each site and the objectives of the current effort, and will be presented in the Operable Unit- (OU-) or site-specific SAP. The QAPP is intended as a

## APPLICABILITY CHECKLIST

| | | |
|---|---|---|
| 1.0 | INTRODUCTION . . . . . | ☐ |
| 2.0 | PROJECT DESCRIPTION . . . . . | ☐ |
| | 2.1 Site History . . . . . | ☐ |
| | 2.2 Sampling and Remediation Activities . . . . . | ☐ |
| | 2.3 Data Collection and Use . . . . . | ☐ |
| 3.0 | PROJECT ORGANIZATION AND RESPONSIBILITY . . . . . | ☐ |
| | 3.1 Project Management . . . . . | ☐ |
| | 3.2 Quality Assurance . . . . . | ☐ |
| | 3.3 Health and Safety . . . . . | ☐ |
| | 3.4 Analytical Services . . . . . | ☐ |
| | 3.5 Primary Task Leaders . . . . . | ☐ |
| 4.0 | QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT AND GEOLOGIC DATA . . . . . | ☐ |
| | 4.1 Precision . . . . . | ☐ |
| | 4.2 Accuracy . . . . . | ☐ |
| | 4.3 Completeness . . . . . | ☐ |
| | 4.4 Representativeness . . . . . | ☐ |
| | 4.5 Comparability . . . . . | ☐ |
| | 4.6 Geologic Data Quality Objectives . . . . . | ☐ |
| 5.0 | FIELD PROCEDURES . . . . . | ☐ |
| | 5.1 Geophysical Techniques . . . . . | ☐ |
| | 5.2 Drilling . . . . . | ☐ |

Figure 1-1. Applicability Checklist

## APPLICABILITY CHECKLIST
(Continued)

| | | | |
|---|---|---|---|
| | 5.3 | Well Installation . . . . . | ☐ |
| | 5.4 | Aquifer Test Methods . . . . . | ☐ |
| | 5.5 | Sample Storage and Preservation, and Handling . . . . . | ☐ |
| | 5.6 | Groundwater Sampling . . . . . | ☐ |
| | 5.7 | Surface Water Sampling . . . . . | ☐ |
| | 5.8 | Soil Sampling . . . . . | ☐ |
| | 5.9 | Sediment Sampling . . . . . | ☐ |
| | 5.10 | Soil Gas Sampling . . . . . | ☐ |
| 6.0 | | SAMPLE CUSTODY . . . . . | ☐ |
| | 6.1 | Documentation Procedures . . . . . | ☐ |
| | 6.2 | Electronically Generated Chain-of-Custody and Master Log . . . . . | ☐ |
| | 6.3 | Sample Handling Procedures . . . . . | ☐ |
| 7.0 | | CALIBRATION PROCEDURES AND FREQUENCY . . . . . | ☐ |
| | 7.1 | Laboratory Instrument Calibration . . . . . | ☐ |
| | 7.2 | Field Instrument Calibration . . . . . | ☐ |
| 8.0 | | ANALYTICAL PROCEDURES AND CALIBRATION . . . . . | ☐ |
| | 8.1 | Laboratory Standards and Reagents . . . . . | ☐ |
| | 8.2 | Extraction Methods . . . . . | ☐ |
| | 8.3 | Organic and Inorganic Analytical Methods for Water and Soil Samples . . . . . | ☐ |

Figure 1-1. (Continued)

## APPLICABILITY CHECKLIST
(Continued)

| | | |
|---|---|---|
| 8.4 | Real-Time Portable Organic Vapor Analyzers . . . . . | ☐ |
| 8.5 | Air Analysis . . . . . | ☐ |
| 8.6 | Analytical Methods for Physical Properties . . . . . | ☐ |
| 8.7 | Analytical Methods for Field Analyses . . . . . | ☐ |
| 9.0 | DATA REDUCTION, VALIDATION, AND REPORTING . . . . | ☐ |
| 9.1 | Data Reduction . . . . . | ☐ |
| 9.2 | Data Transfer and Verification . . . . . | ☐ |
| 9.3 | Data Validation . . . . . | ☐ |
| 9.4 | Reporting . . . . . | ☐ |
| 10.0 | INTERNAL QUALITY CONTROL . . . . . | ☐ |
| 10.1 | Analytical Laboratory Quality Control Samples . . . . . | ☐ |
| 10.2 | Field Quality Control Samples . . . . . | ☐ |
| 11.0 | PERFORMANCE AND SYSTEMS AUDITS . . . . . | ☐ |
| 11.1 | Technical Systems Audit . . . . . | ☐ |
| 11.2 | Performance Audits and Data Quality Audits . . . . . | ☐ |
| 12.0 | PREVENTIVE MAINTENANCE . . . . . | ☐ |
| 12.1 | Maintenance Responsibilities . . . . . | ☐ |
| 12.2 | Maintenance Schedules . . . . . | ☐ |
| 12.3 | Spare Parts . . . . . | ☐ |

Figure 1-1. (Continued)

## APPLICABILITY CHECKLIST
(Continued)

| | | |
|---|---|---|
| 13.0 | DATA ASSESSMENT PROCEDURES . . . . . | ☐ |
| | 13.2 Accuracy . . . . . | ☐ |
| | 13.3 Precision . . . . . | ☐ |
| | 13.4 Completeness . . . . . | ☐ |
| 14.0 | CORRECTIVE ACTION . . . . . | ☐ |
| | 14.1 Corrective Action Report (CAR) . . . . . | ☐ |
| | 14.2 Recommendation for Corrective Action (RCA) . . . . . | ☐ |
| 15.0 | QUALITY ASSURANCE REPORTS . . . . . | ☐ |
| | 15.1 Quality Assurance and Quality Control . . . . . | ☐ |
| | Reporting Formats . . . . . | ☐ |
| 16.0 | SITE MANAGEMENT . . . . . | ☐ |
| | 16.1 Base Point of Contact (BPOC) . . . . . | ☐ |
| | 16.2 Coordination of Field Activities . . . . . | ☐ |
| | 16.3 Location of Field Office Facilities . . . . . | ☐ |
| | 16.4 Site and Equipment Security . . . . . | ☐ |
| | 16.5 Sources of Potable Water . . . . . | ☐ |
| | 16.6 Location of Decontamination Area . . . . . | ☐ |
| | 16.7 Site Access . . . . . | ☐ |
| | 16.8 Cuttings and Groundwater Disposal . . . . . | ☐ |
| | 16.9 Site Health and Safety . . . . . | ☐ |

Figure 1-1. (Continued)

QUALITY ASSURANCE PROJECT PLAN
APPLICABILITY STATEMENT

The McClellan AFB Quality Assurance Project Plan (QAPP) dated _____ (Revision Date) is sufficient for Project Quality Control during performance of the work specification in attached Work Plan, which is:

Titled: _____

Dated: _____ _____

The attached QAPP Applicability Checklist designates the applicable sections of the QAPP.

_____ Name of Task Leader/Contractor

_____ Contractor Project Manager Signature

_____
Date

Figure 1-2. Applicability Statement for McClellan AFB RI/FS Tasks

procedural document developed to ensure consistency in field and analytical procedures, and provide project quality objectives for the analytical levels referenced directly or indirectly in the SAPs (e.g., field screening laboratory procedures, field monitoring instruments, off-site laboratory analyses). Together, the QAPP and SAP complete the documentation needed to define all aspects of field activities at a particular site. The Health and Safety Plan prepared for each OU RI is an additional document that establishes the safety procedures, level of personal protective equipment required, and monitoring requirements and criteria to ensure that field activities will be conducted in a manner that protects personnel who perform the work and others in the vicinity of the activity.

The QAPP is required reading for all staff participating in RI/FS work and will be in the possession of field sampling teams for all sampling efforts. Subcontractors will be required to comply with procedures documented in this QAPP to ensure comparability and representativeness of the data produced. Subcontractor compliance with the QAPP will be included as a necessary requirement in the subcontractor Statement of Work, and audits will be conducted during the course of subcontractor performance to ensure compliance.

This QAPP will be revised as necessary when guidelines and regulatory documents are revised, or when additional sampling or analysis methods are required for the McClellan AFB RI/FS. As revisions are required, they will be prepared as part of the preparation activities for the specific task and incorporated into the comprehensive RI/FS QAPP. This includes revisions to add information regarding subcontractor qualifications for sampling, well drilling, and analytical tasks. A document control system has been established for this QAPP to ensure that updates or revisions will be completely distributed to project staff, subcontractors, and agency representatives. (See the distribution list at the beginning of this document.) As revisions are approved, the revised material will be provided with instructions to replace sections or add inserts to the current document.

# 2.0 PROJECT DESCRIPTION

The United States Air Force is conducting a Remedial Investigation/Feasibility Study (RI/FS) at McClellan Air Force Base (AFB) to assess the extent and magnitude of contamination from past waste disposal and spill sites. This process includes the development of a remediation plan(s) for sites determined to pose a threat to human health or welfare, or the environment. The remedial investigation (RI) phase of the program includes multi-media sampling tasks to complete site characterization studies, and to assess potential exposure pathways.

## 2.1 Site History

McClellan AFB is located near Sacramento, California (Figure 2-1) and is an active aircraft maintenance facility associated with the Air Force Logistics Air Command. As part of past routine maintenance procedures at the base, a variety of hazardous materials were handled and disposed of, including: chlorinated solvents and caustic cleaners; electroplating wastes containing metals; polychlorinated biphenyl (PCB)-contaminated oils; contaminated jet fuels; low-level radioactive wastes; and a variety of oils and lubricants. These wastes were disposed of in several types of facilities, including burial pits, burn pits, and sludge/oil pits. At the time, these disposal methods were accepted industrial practices, and the base obtained permits from appropriate regulatory agencies as required. During the late 1970s, the potential for air pollution and groundwater contamination resulting from these practices was recognized, and land disposal methods were discontinued by 1981.

Initial investigations to identify and characterize contamination on and near the base have identified 171 individual sites, most of which have been grouped into four geographical areas of the base, designated Operable Units (OUs) A, B, C, and D, which are shown in Figure 2-2, and are described below.

### Operable Unit A

Operable Unit A encompasses approximately 530 acres located in the southeast portion of McClellan AFB in an area of heavy industrial activity (e.g., aircraft engine repair and testing) involving trichloroethene (TCE) usage. Operable Unit A contains 25 sites with

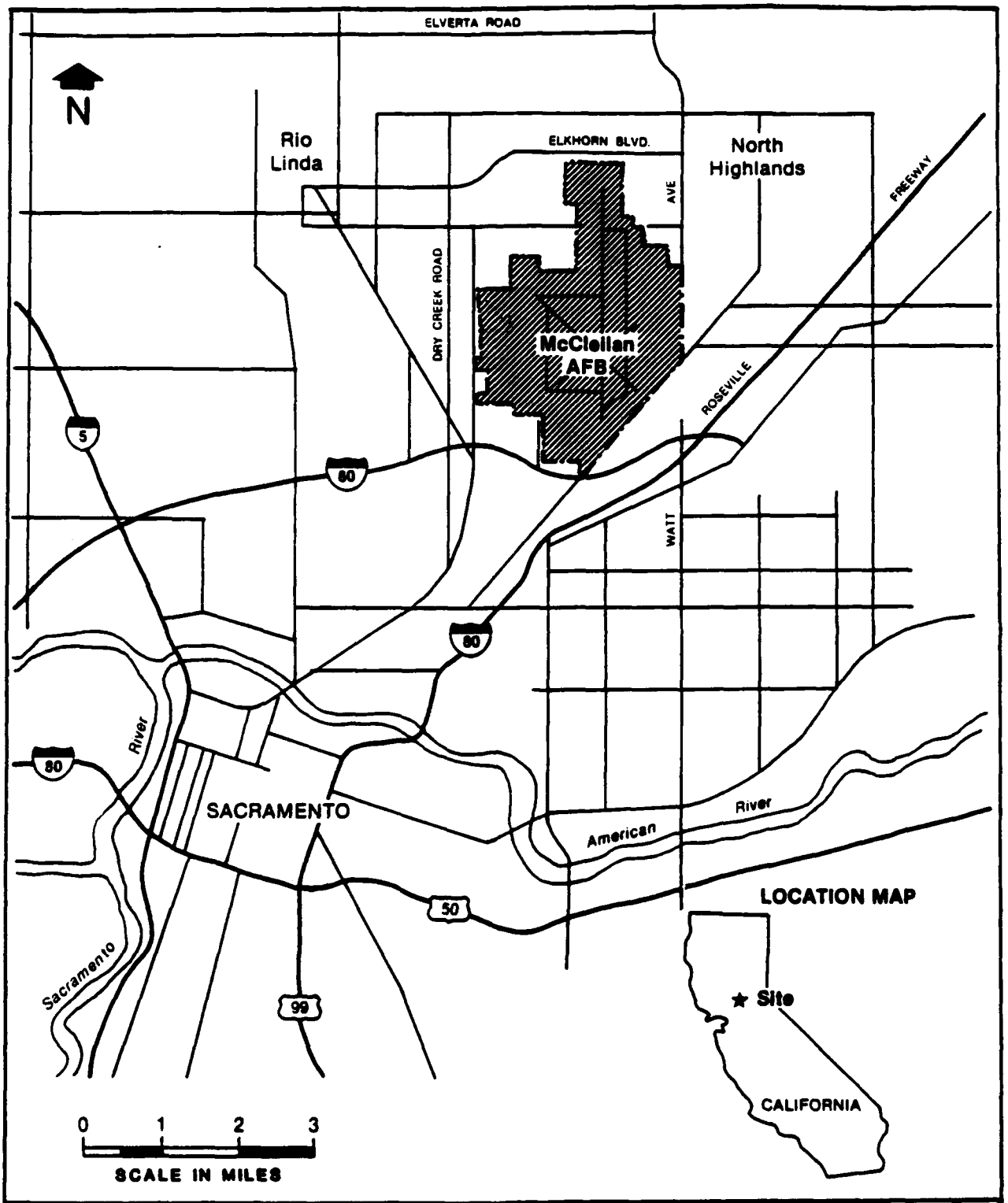


**Figure 2-1. Location of McClellan AFB**

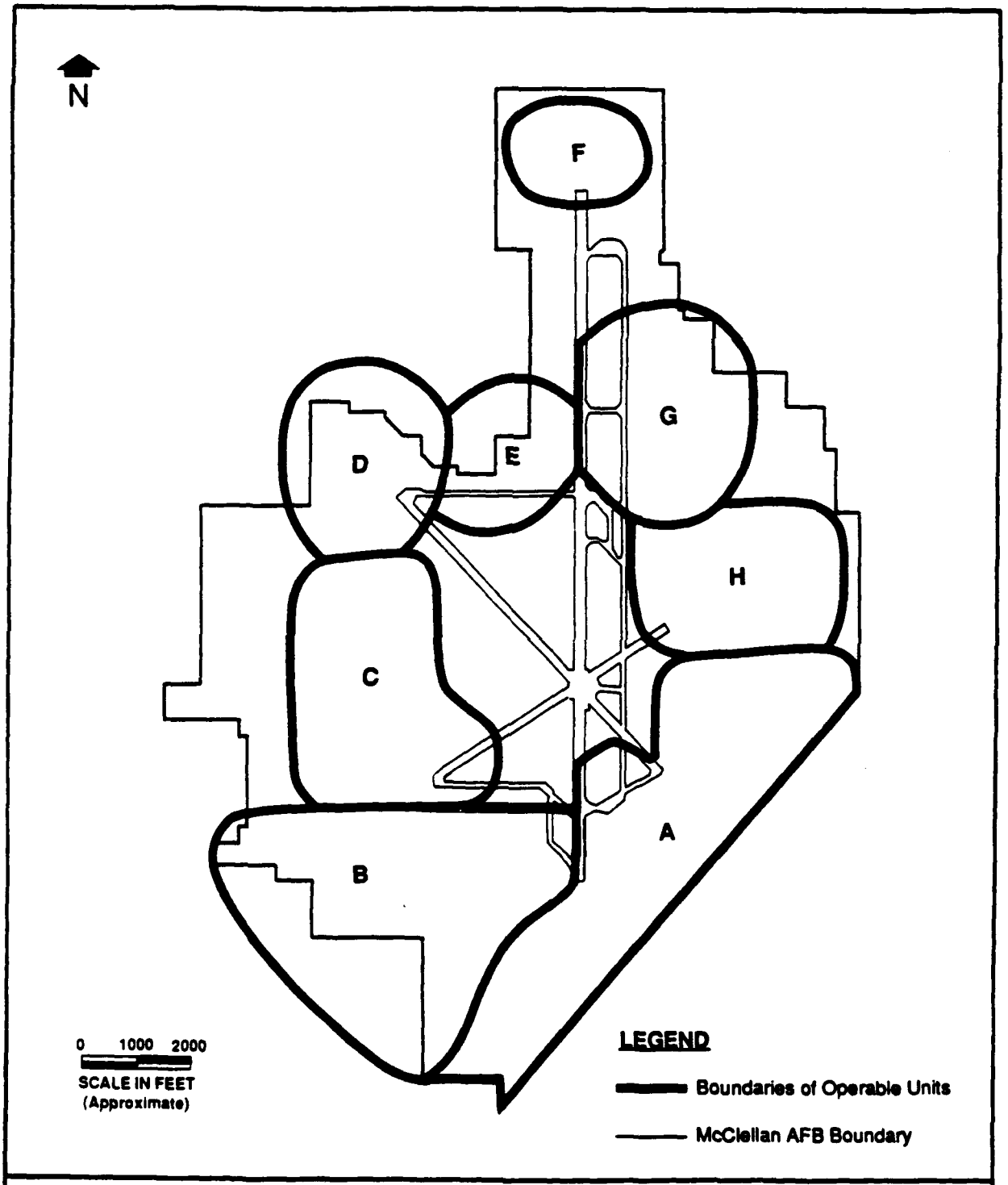


**Figure 2-2. Approximate Boundaries of Operable Units at McClellan AFB.**

0491-078-1

confirmed soil contamination and 35 locations and 61 study areas that are potential sources of hazardous substances to the soils and groundwater. Materials disposed of in OU A include refuse and refuse ash, 1,2-dichloroethane (1,2-DCA), cresylic acid, skimming pond sludges, and industrial wastewater sludge. Groundwater samples collected from monitoring wells in the vicinity of OU A have contained a variety of volatile organic compounds (VOCs) including:

- TCE;
- 1,1-Dichloroethene (1,1-DCE);
- 1,2-DCA;
- Acetone;
- Toluene;
- Carbon tetrachloride;
- 1,1,1-Trichloroethane (1,1,1-TCA);
- Chloroform;
- Methylene chloride;
- Benzene;
- Ethyl benzene; and
- Trichlorofluoromethane (TCFM).

Contaminants reported in soil samples collected from sites in OU A include:

- Acetone,
- Benzene;
- Chlorobenzene;
- Chloroform;
- Ethyl benzene;
- 1,1-DCA;
- 1,2-DCE;
- TCE;
- 1,1,1-TCA;
- Total xylenes;
- Bis(2-ethylhexyl)phthalate;
- Di-n-butylphthalate;
- Oil and grease;

- Pentachlorophenol;
- Toluene;
- Tetrachlorodibenzofuran;
- 4,4'-DDE;
- Heptachlorodibenzodioxin;
- Hexachlorodibenzodioxin;
- Octachlorodibenzodioxin;
- Methyl ethyl ketone (MEK);
- Methyl isobutyl ketone;
- Tetrachloroethene (PCE);
- Vinyl chloride
- Pyrene; and
- TCFM.

Three base production wells located in OU A (BW-1, BW-2, and BW-12) have been closed because elevated concentrations of TCE have been reported. A preliminary assessment was conducted in 1989 to research known sites and locations and to identify other possible sources of contamination. Sixty-one new study areas were identified as possible sources of contamination. A Draft Sampling and Analysis Plan (SAP) has been developed, and the OU A RI field sampling will begin in mid-1992.

**Operable Unit B**

Operable Unit B encompasses approximately 325 acres in the southwest portion of McClellan AFB, and includes six sites with known contamination and 23 locations and 19 study areas that are potential sources of hazardous substances to the soils and groundwater. Operable Unit B has historically been an area of varied uses, including: storage yards, warehouses, a chemical laboratory, a aircraft fueling area, a woodshop, an instrument repair facility, a paint facility, two industrial waste treatment plants (IWTP), and a plating shop. Materials handled at various locations within the OU include: solvents, diethyl ether, low-level radioactive wastewater, and waste chemicals generated during plating activities. In OU B, TCE has been found in Base Production Well 18, which currently operates with a filtering system. City Well 150 located south of the area was closed because of TCE contamination. Volatile organic compounds reported in groundwater collected from OU B are:

- 1,1-DCE;
- TCE;
- PCE;
- trans-1,2-Dichloroethene (trans-1,2-DCE);
- MEK;
- Carbon disulfide;
- Dichlorobenzene;
- Chloroform; and
- Methylene chloride.

Contaminants reported in soil samples collected from sites in OU B include:

- Acetone;
- Toluene;
- Chloroform;
- Methylene chloride;
- Ethylbenzene;
- Total xylenes;
- PCE;
- trans-1,2-DCE;
- Dichloromethane;
- Oil and grease;
- 2-Butanone;
- TCE;
- Cyanide;
- 1,1,1-TCA;
- PCB-1260;
- Pyrene;
- PCB-1254;
- Phenol;
- Ketones; and
- Phthalates.

Contaminants reported in soil gas samples collected from sites in OU B include:

- Chloroform;
- 1,1,1-TCA;
- TCE;
- Tetrachloroethene;
- Total 1,2-DCE;
- Benzene;
- Toluene;
- Xylenes;
- Acetone;
- Chlorotrifluoroethene;
- Dichlorotrifluoroethane;
- Freon® 113;
- Vinyl chloride;
- Carbon disulfide;
- Ethylhexanone;
- Propylene;
- Dimethylhexane;
- 1,3-Dichlorobenzene;
- Dimethylbutane;
- Dimethylpentane; and
- Pentane.

A preliminary assessment was conducted in 1988 and 1989 to research known sites and locations and to identify other possible sources of contamination. Nineteen new study areas were identified as possible sources of contamination.

In 1989, a groundwater remedial investigation of OU B (ABGOURI) was conducted to determine if any threat of contamination to active municipal water supply wells existed in the OU and southwest of the base. The OU B Engineering Evaluation/Cost Analysis (EE/CA) was conducted in 1989 to further characterize contaminant concentrations and aquifer characteristics. Data collected from the ABGOURI and EE/CA were used to identify and evaluate different expedited response alternatives to address groundwater

contamination in OU B. In early 1991, a groundwater treatment and extraction system, as part of the Expedited Response Action (ERA), was completed in the Building 666 area of OU B to capture contaminated groundwater before it reaches Base Well 18 and off-base wells.

In 1990, the OU B Soil Gas Investigation was conducted to locate areas of VOC contamination in the soil gas. Areas of elevated soil gas concentration (targets) will be investigated during the OU B RI in 1991 through 1993. The OU B RI will include surface water, stream sediment, soil, and soil gas sampling and analysis.

**Operable Unit C**

Operable Unit C encompasses approximately 300 acres in the west-central portion of McClellan AFB. Forty-seven sites in OU C are potential sources of hazardous substances to the soils and groundwater; 40 sites have been partially studied. Currently, OU C includes a Civil Engineering materials storage yard, the IWTP, the Groundwater Treatment Plant (GWTP), an aircraft maintenance hangar, a paint shop, a contaminated soil holding area, aeration lagoons, and a fire training area. These sites include refuse sludge pits, chemical waste pits, burn pits, waste oil and solvent storage ponds (used from the 1940s to 1971), surface storage areas, settling ponds, wastewater ponds, and unlined drainage ditches. The area was used from the 1940s to 1972 for waste disposal. Waste was disposed into excavated trenches, which were sometimes burned, and were subsequently covered with fill material. These wastes were from a variety of sources, and included ash, industrial wastes, burned debris, and general refuse. A wide variety of VOCs have been detected in groundwater samples collected from this area. The VOCs reported in groundwater collected from OU C are:

- TCE;
- 1,2-DCA;
- 1,1-DCE;
- PCE;
- trans-1,2-DCE;
- Toluene; and
- Benzene.

Contaminants reported in soil samples collected from sites in OU C include:

- Chlorinated VOCs;
- Ketones;
- Benzene;
- Toluene;
- Total xylenes; and
- Phthalates.

A remediation measure consisting of the groundwater extraction system in OU C, began operation in August 1988. A preliminary assessment is scheduled to begin in 1992 to research known sites and locations and to identify other possible sources of contamination (study areas). A separate OU (C1) has been identified within OU C for immediate action because of identified soil contamination that may be a source of groundwater contamination. Operable Unit C1 contains the IWTP, portions of the IWL, and several other sites. The RI field sampling in this area is beginning in mid-1992, and soil treatability studies are currently underway.

**Operable Unit D**

Operable Unit D encompasses approximately 175 acres in the northwest corner of McClellan AFB. Thirteen sites in this OU are potential sources of hazardous substances to the soils and groundwater; twelve sites have been studied. These include waste burn and debris pits, fuel and sludge disposal pits, a wastewater sludge disposal and burn pit, a skimmed oil burn area, a sodium valve disposal pit, and an industrial wastewater sludge landfarm. The VOCs reported in groundwater collected from OU D are:

- TCE;
- 1,1-DCE;
- 1,2-DCA;
- Total 1,2-DCE; and
- Vinyl chloride.

Contaminants reported in soil samples collected from sites in OU D include:

- Vinyl chloride;
- 1,1-DCA;
- trans-1,2-DCE;
- Freon® 113;
- TCA;
- TCE;
- Toluene;
- Total xylenes;
- Dichlorobenzene;
- Acetone;
- 4-Methyl-2-pentanone;
- 1,1,1-TCA;
- Ethyl benzene;
- PCE;
- Phthalates;
- Chrysene;
- Pyrene; and
- Phenol.

Two remediation measures have been implemented in OU D: a clay cap was constructed over former sludge/oil disposal sites; and a groundwater extraction and treatment system was installed and has been in operation since early 1987. In 1990, a soil gas testing program began to define the nature and extent of soil gas contamination and to validate the soil gas sampling technique in OU D. A pilot-scale soil gas extraction and treatment system is currently being designed to remediate soil gas contamination in OU D.

**Operable Units E, F, G, and H**

In addition to the sites in OUs A, B, C, and D, historical record searches have identified another 21 sites located in OUs E, F, G, and H that may be potential sources of contamination; none of these sites have been studied.

## 2.2 Sampling and Remediation Activities

Other sampling and remediation measures have been implemented. From 1979 through 1986, residential drinking water wells near the base were sampled and analyzed for organic compounds and priority pollutant metals. Volatile organic compounds were consistently detected in some of these wells above state action levels. As an immediate remediation measure, city water hookups were provided for homes near the base. A monitoring well network has been installed and includes on- and off-base wells that are located upgradient and downgradient of known disposal areas. Currently, between 160 and 180 monitoring wells are sampled quarterly for organic compounds and priority pollutant metals. The quarterly sample data are being analyzed to determine the magnitude and extent of contamination, and to monitor changes in groundwater quality that may be attributed to the extraction and treatment systems.

The analytical results for the residential and monitoring wells to date indicate the presence of shallow to deep groundwater contamination in and near known disposal areas on base, and contamination of the shallow groundwater zone in areas southwest, west, northwest, and east of the base. Contamination in some of these areas appears to be localized. The OU D extraction system appears to be decreasing contaminant concentrations in middle and deep groundwater zones in the immediate area, and is affecting groundwater flow directions in the area by creating a pumping depression.

In 1989, a basewide Preliminary Surface Water Pathway Assessment was conducted to determine the potential for migration of hazardous constituents via surface water.

Field work for the Preliminary Groundwater Operable Unit Remedial Investigation (PGOURI) was conducted from 1989 through 1990. Eighty-four monitoring wells and 36 piezometers were installed basewide to provide basewide data on groundwater flow and contaminant concentrations. Selected monitoring wells has been included as part of the quarterly sampling program.

## 2.3 Data Collection and Use

During the individual OU RIs, separate tasks will be implemented to characterize unstudied sites, expand the monitoring well network to allow more complete characterization of the extent of groundwater contamination, and to sample surface water, groundwater, sediment, soil, and soil gas at designated sites. The samples will be analyzed for volatile and semivolatile organic compounds, priority pollutant metals, cyanide, pesticides, PCBs, dioxins and furans, and total petroleum hydrocarbons as appropriate, determined from historical records and data; samples from one site will be analyzed for low-level radioactivity. Specific sampling and analytical procedures will be selected and Data Quality Objectives (DQOs) developed for each site and phase of work. The DQOs and selected procedures will be presented in the SAP.

In addition to completing the site characterization, the data collected in the various sampling tasks will be used to:

- Identify potential human exposure pathways and environmental hazards;
- Provide input for risk assessment of the exposed human population; and
- Provide information in determining appropriate cleanup levels for hazardous sites.

The planning and implementation of various RI/FS activities in OUs A, B, C, and D is ongoing; activities in the remaining OUs will begin as these higher priority OUs move toward remediation and Records of Decision (RODs). It is not possible to set a detailed schedule for completion of all RI/FS activities because of the number of unstudied locations, and the amount of additional information that may be needed to support feasibility studies and selection of remedies. However, an estimate of 10 years to complete sampling and analysis and other RI/FS activities for all OUs is made, based on the rate of progress to date and comparison with other RI/FS programs.

# 3.0 PROGRAM ORGANIZATION AND RESPONSIBILITIES

Overall program management responsibilities on the McClellan Air Force Base (AFB) Installation Restoration Program (IRP) are defined by the terms of an Interagency Agreement (IAG) dated May 1990. The IAG specifies that the Air Force, the United States Environmental Protection Agency (U.S. EPA) (Region IX), and the California Department of Health Services and the Central Valley Regional Water Quality Control Board (RWQCB) (now consolidated under the California Environmental Protection Agency [Cal/EPA]), will work cooperatively in directing the course of the McClellan AFB IRP. This direction includes all decisions related to priorities for site investigation and cleanup, procedures to be followed in accomplishing all program activities, and establishing program milestones.

The efforts required to implement the IRP at McClellan AFB are directed and funded by the Air Force through the McClellan AFB Environmental Management Directorate (EM). Assisting the Air Force in this mission are a number of base contractors, subcontractors, and consultants. In this section, responsibilities are defined for each of the key personnel or functional areas in the organizations listed above. The general IRP organizational structure is presented in Figure 3-1.

## 3.1 U.S. Air Force, U.S. EPA, and Cal/EPA

The signatories to the IAG have each assigned a Remedial Program Manager (RPM) who serves as spokesman for his/her organization in directing the course of the McClellan AFB IRP. The RPMs as of August 1992 are Fran Slavich (McClellan AFB EM), Kathi Moore (U.S. EPA), Mark Malinowski (Cal/EPA DTSC), and Alex MacDonald (Cal/EPA RWQCB).

Within the McClellan AFB EM organization, all project activities are directed by a team leader who has responsibility for implementing the projects that have been agreed to by the RPMs, and directing the efforts of base personnel and contractors who perform the work. For purposes of executing the IRP at McClellan AFB, the base has been divided into eight Operable Units (OUs), labelled A through H; OUs currently under active investigation and remediation are OUs A through D. Operable unit team leaders as of August 1992 are Jerry Styles (OUs A and D) and Bud Hoda (OUs B and C). These OU team leaders are assisted in their efforts by an Operations and Maintenance Team, led by Kirk Schmalz, and a Program Execution Team, led by Pat Christman. These team leaders are responsible for

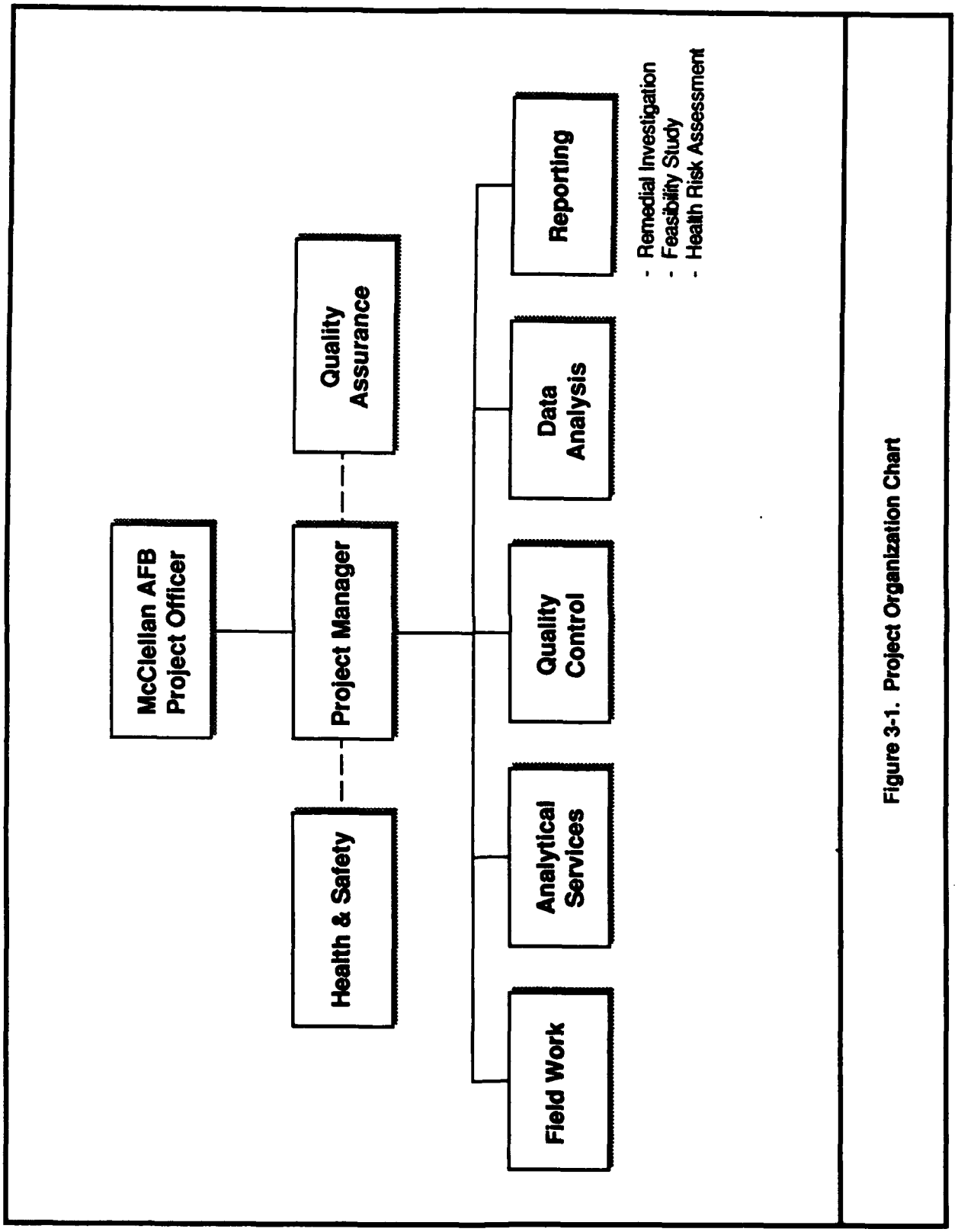


Figure 3-1. Project Organization Chart

implementing basewide programs such as the management of underground storage tanks, which are not specific to an OU.

The primary mechanism for implementing the IRP at McClellan AFB is through projects. Project objectives, scopes, and schedules are established by the RPMs who work in concert with the team leaders. The team leaders are then responsible for assigning the project to internal staff, to a contractor, or to a combination of these two. A Project Manager (Contractor) and/or Officer (Air Force) is assigned and is ultimately responsible for implementing the project and meeting the objectives. This includes developing detailed project plans and obtaining appropriate review and concurrence with the procedures before proceeding with implementation of the plan. In the case of remedial investigation (RI) projects, the planning documentation includes this Quality Assurance Project Plan (QAPP) and a Sampling and Analysis Plan (SAP). The SAP specifies in detail the data quality objectives and requirements, the locations of samples to be collected, the sampling and analytical procedures to be followed, and project quality control and quality assurance procedures. A project management plan that specifies the activities to be conducted, the procedures to be followed, critical milestones for tracking project progress, project team member responsibilities, and required resources to accomplish project objectives is also developed.

This project-oriented approach is necessary accommodate the range of IRP activities being conducted at McClellan AFB. The projects have widely differing data quality objectives, documentation, and review requirements, and the flexibility of this organizational structure allows involvement and representation of the appropriate combination of Air Force, agency, and technical experts.

## 3.2 Contractor Project Organization

The organization for a contractor's Remedial Investigation/Feasibility Study (RI/FS) project team will generally include technical professionals with experience in project management, quality assurance, analytical chemistry, field investigations, data management, and other technical/engineering skills. Contractors may use different organizational structures and titles for individual positions; however, the responsibilities for program areas described here must be assigned to personnel within the chosen structure. The specific individual responsible for project implementation in each of these areas will be described in the project management plans.

- Project Management personnel are responsible for the contractual aspects of the project work, ensuring that appropriate staff are assigned to the project, and that there are adequate field equipment and other resources allocated to fulfill the needs of the project. They are also responsible for ensuring that the technical activities have appropriate planning and oversight to assure that the quality and timeliness of data and reports produced meet or exceed project requirements. The project manager has the primary responsibility for decision making within the contractor's organization, and communication with the Air Force.

- The Quality Assurance (QA) Coordinator has overall responsibility to independently assure that the planning, implementation, and reporting for RI/FS activities fulfills the objectives for data use. The QA Coordinator must have reporting responsibility outside of the project organization to assure independence in decision making and recommendation for corrective actions. The QA Coordinator is responsible for assuring that the established data validation procedures are followed, that audits of field and laboratory activities are conducted, and that needed corrective actions are implemented. Preparing QAPP revisions or addenda, and communication with Air Force and other contractor QA representatives are also the responsibility of the QA Coordinator.

- The Data Management task leader is responsible for maintaining the project-specific database, reporting results in required formats to the Air Force and data users, and assuring the integrity of the project-specific database.

- The Field Activity task leader is responsible for directing field work, assuring that the field staff has adequate training in both the field sampling and measurement procedures and Health and Safety procedures, reporting the status of the field activity to project management and the Air Force, and directing subcontractors. The Field Activity task leader has the overall responsibility for implementing the field sampling plan and communicating any difficulties encountered.

- The other primary project staff members include technical professionals with expertise in risk assessment, chemistry, geology, hydrology, treatability studies, and remedial engineering, as appropriate for the scope of activities performed by the contractor. These individuals have responsibility for internal communication and task planning to assure that all data obtained can be used for its intended purpose, and to provide the direction and oversight needed to assure that technically sound decisions are made within their area of expertise.

# 4.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENTS AND GEOLOGIC DATA

The purpose of a quality assurance/quality control (QA/QC) program is to produce data of known quality that satisfy the project objectives and that meet or exceed the standard requirements for the analytical methods. The QA/QC program shall:

- Provide a mechanism for ongoing control and evaluation of measurement data quality; and

- Provide measures of data quality in terms of precision, accuracy, representativeness, completeness, and comparability (PARCC) and assess whether the data meet the Data Quality Objectives (DQOs) and can be used for their intended purpose.

The PARCC or Quality Assurance Objectives (QAOs) for each field effort will vary based on the end use of the data. To allow flexibility in conducting different phases of field work, the QAOs are specified by analytical level to allow the most efficient and cost-effective methods to be selected and the data to be evaluated appropriately. General analytical levels are described in the U.S. Environmental Protection Agency (U.S. EPA) Guidance for Data Quality Objectives (DQOs) for Remedial Response Activities (1987). For purposes of the current McClellan Air Force Base (AFB) Remedial Investigation/Feasibility Study (RI/FS) field projects, Levels 1, 2, and 3 are defined as follows:

- Level 1 data are produced from field screening instruments such as organic vapor analyzers (OVAs), HNu®s, and organic vapor monitors (OVMs) used for health and safety monitoring and soil gas screening, and pH and conductivity meters used for groundwater monitoring during well purging.

- Level 2 data are produced from on-site laboratory analyses for soil and soil gas screening. These analyses are conducted to determine the presence or absence of selected analytes, or classes of analytes, at concentrations greater than specified limits. The operating requirements and QC acceptance criteria for on-site analytical methods are less

stringent than reference methods (e.g., SW846) to allow rapid screening of a large number of samples.

- Level 3 data are produced from off-site laboratory analyses using reference methods (e.g., SW846). These analyses must follow specific method and project requirements for instrument calibration and QC acceptance criteria. Level 3 data are required for quantitation and speciation of method analytes with greater precision and accuracy than can be achieved by Level 1 or 2 analyses.

Level 4 and 5 analyses are not currently planned or expected for the RI/FS; however, if higher level analyses are required, the QAOs for those levels will be included in an update of the QAPP.

The analytical levels required for a sampling effort are determined in DQO development and are specified in the Sampling and Analysis Plan (SAP). The QAOs for each analytical level are established to ensure consistency and comparability of data collected during all phases of the RI/FS. Use of one set of QAOs for each analytical level and method is necessary to maintain consistent assessment and interpretation of data produced throughout the duration of the RI/FS and between Operable Unit (OU) RIs. This also provides a common performance specification for all laboratories that analyze samples for large and/or long-term field efforts.

The U.S. EPA DQO guidance conceptualizes the approach for establishing QAOs based on project requirements. In reality, capabilities and routinely achievable performance levels for the analytical methods, knowledge of site conditions and history of use, and any previous analytical results must also be considered when establishing the QAOs. The selection of analytical methods that consider these factors becomes a critical step in developing both DQOs and QAOs.

After selecting the "best" method for the analytes of interest using this information, the method specifications for precision and accuracy are the most appropriate and realistic QAOs to be applied to the project data. The method specifications represent the baseline or minimum performance level for all laboratories performing the method, and data produced will be comparable within those specifications. For these reasons, the project

QAOs are taken largely from the method specifications. Where method specifications have not been established, the QAOs are based on DQOs, available laboratory performance information for the method and matrix, and understanding of the method capabilities. This occurs mostly for soil and waste matrices. These QAOs will be used to assess project data, until the attained precision and accuracy can be calculated. Then, comparison with the DQOs can be made to determine whether the attained performance satisfies the DQOs or whether additional iterations of sampling and analysis are needed.

Tables 4-1 through 4-13 present precision and accuracy QAOs for each analytical level, method, and matrix that have been established for the McClellan AFB monitoring and RI/FS projects. The objectives for Levels 1 and 2 data are based on the DQOs and historical performance for these methods. As discussed above, the objectives for Level 3 data are the method-specified limits for spike recovery and laboratory duplicate sample or spike analyses. If method-specified limits have not been established, the objectives are based on DQOs and historical performance (i.e., laboratory-based limits). These limits will be used to assess project data.

The basis for assessing precision, accuracy, completeness, representativeness, and comparability is discussed in the following subsections. Specific calculations for data quality measurements, and data assessment and validation procedures, are presented in Section 13.0. The validity of individual data points is determined during the data assessment process. Valid data are data that satisfy the QA objectives. The "qualified data" should be considered as estimates based on the results of associated QC samples (blanks, spikes, duplicates, performance evaluation samples, etc.). Qualified data, though estimates, are documented and, therefore, defensible. Accordingly, qualified data are considered to be usable as long as the cited quality flags and any associated limitations are evaluated for appropriateness for data use.

## 4.1 Precision

Precision measures the reproducibility of repetitive measurements. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Analytical precision is a measurement of the variability associated with duplicate (2) or replicate (more than 2) analyses of the same sample in the laboratory and is determined by analysis of matrix

**TABLE 4-1. QUALITY ASSURANCE OBJECTIVES FOR LEVEL 1 ANALYSES**

| | Precision[a] | Accuracy |
|---|---|---|
| Organic vapor analyzer (OVA), HNu® | ±20% | r≥0.9950 |
| pH[b] | -- | 0.1 |

[a] Precision measured as comparison between daily response factor and multipoint calibration response factor.
[b] Accuracy criterion for pre- and post-sampling calibrations.

**TABLE 4-2. QUALITY ASSURANCE OBJECTIVES FOR LEVEL 2 ANALYSES**

| | Precision | Accuracy |
|---|---|---|
| Soil gas analyses | ±30%[a] | ±30%[c] |
| Soil volatile organic compound (VOC) analyses | ±30%[b] | ±50%[d] |
| Soil polychlorinated biphenyl (PCB) analyses | ±30%[b] | ±50%[d] |

[a] Precision measured as the relative percent difference (RPD) between duplicate injections.
[b] Precision measured as the RPD between duplicate spikes or duplicate samples.
[c] Accuracy measured using daily calibration check sample results compared with expected results.
[d] Accuracy measured using matrix or surrogate spike recoveries.

**TABLE 4-3. QUALITY ASSURANCE/QUALITY CONTROL OBJECTIVES FOR LEVEL 3 MEASUREMENTS IN AIR**

| Measurement Parameter | Reference Method | Field Duplicate Analyses (RPD) | Laboratory Duplicate Analyses (RPD) | Control Sample Analyses (% Recovery) |
|---|---|---|---|---|
| Volatile Organic Compounds | TO14; U.S. EPA Compendium Method, GC/FID-PID-HECD, GC/MS stainless steel canister | <50 | <50 | ±30[a] |

[a] Recovery limit for at least 10 of 12 compounds in daily control samples, and recovery of compounds in performance evaluation audit samples.

GC/FID-PID-HECD = Gas chromatography/flame ionization detector-photoionization detector-Hall electroconductivity detector.

OVA-FID = Organic vapor analyzer-flame ionization detector.

NA = Not applicable.

RPD = Relative percent difference.

## TABLE 4-4. QUALITY ASSURANCE/QUALITY CONTROL OBJECTIVES FOR LEVEL 3 MEASUREMENTS IN WATER

| Measurement Parameter | Reference Method | Precision Objectives: Field Duplicate Analyses (RPD) | Precision Objectives: Laboratory Duplicate Analyses (RPD) | Accuracy Objectives: Matrix Spike Analyses (Recovery) | Accuracy Objectives: Laboratory Control SampleAnalyses (Recovery) |
|---|---|---|---|---|---|
| **INORGANICS** | | | | | |
| Metals | SW6010 (ICPES) | <50 | <20 | 80 - 120[b] | 80 - 120[b] |
| Arsenic | SW7060 (Graphite Furnace AA) | <50 | <20 | 80 - 120 | 80 - 120[b] |
| Lead | SW7421 (Graphite Furnace AA) | <50 | <20 | 80 - 120 | 80 - 120[b] |
| Mercury | SW7470 (Cold Vapor AA) | <50 | <20 | 80 - 120 | 80 - 120[b] |
| Selenium | SW7740 (Graphite Furnace AA) | <50 | <20 | 80 - 120 | 80 - 120[b] |
| Anions | E300.0 (IC) | <50 | <10 | 90 - 110 | NS |
| Alkalinity | E310.1 | <50 | <20 | 85 - 115 | NS |
| Total and Amenable Cyanide | SW9010/SW9012 | <50 | <20 | 85 - 115 | NS |
| Nitrates | E353.2 | <50 | <20 | 85 - 115 | NS |
| **ORGANICS** | | | | | |
| Halogenated Volatile Organics | SW8010 | <30 | <30 | See Table 4-6 | See Table 4-6 |
| Total Petroleum Hydrocarbons | SW8015 Modified | <50 | <30 | 50 - 150[d] | 50 - 150[d] |
| Aromatic Volatile Organics | SW8020 | <30 | <30 | See Table 4-7 | See Table 4-7 |
| Organochlorine Pesticides & PCBs | SW8080 | <50 | See Table 4-8 | See Table 4-8 | See Table 4-8 |
| Organophosphorus Pesticides | SW8140 | <50 | <30 | See Table 4-9 | See Table 4-9 |
| Chlorinated Herbicides | SW8150 | <50 | <30 | See Table 4-10 | See Table 4-10 |
| GC/MS Volatile Organics | SW8240 | <50 | See Table 4-11 | See Table 4-11 | See Table 4-11 |
| GC/MS Semivolatile Organics | SW8270 | <50 | See Table 4-12 | See Table 4-12 | See Table 4-12 |
| Dibenzodioxins/Dibenzofurans | SW8280 | <50 | <50 | NS | 40 - 120[c] |
| Polynuclear Aromatic Hydrocarbons | SW8310 | <50 | <30 | See Table 4-13 | See Table 4-13 |
| 1,2-Dibromoethane (EDB) | E504 | <50 | <30 | 50 - 150[d] | 50 - 150[d] |

[a] SW846, 3rd edition
[b] U.S. EPA Contract Laboratory Program ILM02.0 (limits not applicable to silver or antimony by ICPES).
[c] Estimated range of recovery for surrogate spike standards. Recovery data for specific matrices are given in Method SW8280, Table 6 (SW846).
[d] Until laboratory generated limits can be established; also applies to surrogate spikes for SW8015.

RPD = Relative Percent Difference
E = United States Environmental Protection Agency.
PCBs = Polychlorinated biphenyls.
ICPES = Inductively coupled plasma-atomic emission spectroscopy.
GC/MS = Gas chromatograph/mass spectrometer.
LCS = Laboratory control sample.
SW = Methods from SW846, 3rd Edition.
IC = Ion chromatograph.
AA = Atomic absorption.
NA = Not available.
NS = Not spiked.

## TABLE 4-5. QUALITY ASSURANCE/QUALITY CONTROL OBJECTIVES FOR LEVEL 3 MEASUREMENTS IN SOIL AND SEDIMENTS

| Measurement Parameter | Reference Method | Precision Objectives: Field Duplicate Analyses (RPD) | Precision Objectives: Laboratory Duplicate Analyses (RPD) | Accuracy Objectives: Matrix Spike Analyses (Recovery) | Accuracy Objectives: Laboratory Control SampleAnalyses (Recovery) |
|---|---|---|---|---|---|
| **INORGANICS** | | | | | |
| Metals | SW6010 (ICPES) | <50 | <30 | 75 - 125[a] | 80 - 120 |
| Arsenic | SW7060 (Graphite Furnace AA) | <50 | <30 | 75 - 125[a] | 80 - 120 |
| Lead | SW7421 (Graphite Furnace AA) | <50 | <30 | 75 - 125[a] | 80 - 120 |
| Selenium | SW7740 (Graphite Furnace AA) | <50 | <30 | 75 - 125[a] | 80 - 120 |
| Mercury | SW7471 (Cold Vapor AA) | <50 | <30 | 75 - 125 | 80 - 120 |
| Hexavalent/Chromium | SW7196 | <50 | <30 | 75 - 125 | 80 - 120 |
| Total and Amenable Cyanide | SW9010/SW9012 | <50 | <30 | 75 - 125 | 80 - 120 |
| Total Organic Carbon | SW9060 | <50 | <30 | NA | NS |
| **ORGANICS** | | | | | |
| Organic Lead | HML 338 | <50 | <50 | 50 - 150[b] | 50 - 150[b] |
| Halogenated Volatile Organics | SW8010 | <50 | <50 | See Table 4-6 | See Table 4-6 |
| Total Petroleum Hydrocarbons | SW8015 Modified | <50 | <50 | 50 - 150[b] | 50 - 150[b] |
| Aromatic Volatile Organics | SW8020 | <50 | <50 | See Table 4-7 | See Table 4-7 |
| Organochlorine Pesticides & PCBs | SW8080 | <50 | See Table 4-8 | See Table 4-8 | See Table 4-8 |
| Organophosphorus Pesticides | SW8140 | <50 | <50 | See Table 4-9 | See Table 4-9 |
| Chlorinated Herbicides | SW8150 | <50 | <50 | See Table 4-10 | See Table 4-10 |
| GC/MS Volatile Organics | SW8240 | <50 | See Table 4-11 | See Table 4-11 | See Table 4-11 |
| GC/MS Semivolatile Organics | SW8270 | <50 | See Table 4-12 | See Table 4-12 | See Table 4-12 |
| Dibenzodioxins/Dibenzofurans | SW8280 | <50 | <50 | NS | 50 - 150[c] |
| Polynuclear Aromatic Hydrocarbons | SW8310 | <50 | <50 | See Table 4-13 | See Table 4-13 |
| Nitrosamines | E607 | <50 | <50 | 50 - 150[b] | 50 - 150[b] |

[a] U.S. EPA Contract Laboratory Program ILM02.0.
[b] Until laboratory generated limits can be established.
[c] Estimated range of recovery for surrogate spike standards. Recovery data for specific matrices are given in Method SW8280, Table 6.

NS = Not spiked.
E = United States Environmental Protection Agency.
PCBs = Polychlorinated biphenyls.
ICPES = Inductively coupled plasma-atomic emission spectroscopy.
GC/MS = Gas chromatograph/mass spectrometer.
SW = Methods from SW846, Third Edition.
NA = Not available.
AA = Atomic absorption.
LCS = Laboratory control sample.

**TABLE 4-6. QUALITY CONTROL ACCEPTANCE CRITERIA FOR METHOD SW8010**

| | Aqueous Matrix Spike | | Solid Matrix Spike | | |
|---|---|---|---|---|---|
| Analyte[a] | Accuracy (% Recovery) | Precision (RPD) | Accuracy (% Recovery) | Precision (RPD) | Spike Accuracy (% Recovery)[d] |
| Carbon tetrachloride | | <30 | | <50 | 43 - 143 |
| Chlorobenzene | | <30 | | <50 | 38 - 150 |
| Dibromochloromethane | | <30 | | <50 | 24 - 191 |
| 1,2-Dichloroethane | | <30 | | <50 | 51 - 147 |
| 1,1-Dichloroethene | | <30 | | <50 | 28 - 167 |
| trans-1,2-Dichloroethene | | <30 | | <50 | 38 - 155 |
| 1,2-Dichloropropane | | <30 | | <50 | 44 - 156 |
| Methylene chloride | | <30 | | <50 | 25 - 162 |
| 1,1,2,2-Tetrachloroethane | | <30 | | <50 | 8 - 184 |
| Trichloroethene | | <30 | | <50 | 35 - 146 |
| **Surrogate** | | | | | |
| 1-Bromo-4-fluorobenzene | 59 - 143[b] | NA | 76 - 140[b] | NA | NE |
| Bromochloromethane | 50 - 150[c] | NA | 50 - 150[c] | NA | NE |

[a] Subset of analytes spiked; if additional analytes are spiked, SW846 limits will be used to assess accuracy.
[b] Laboratory generated limits; method specifications not established.
[c] Until laboratory generated limits can be established.
[d] SW846 3rd edition, Table 3, Method SW8010; recovery range for laboratory control samples and matrix spikes.

RPD = Relative percent difference.
NA = Not applicable.
NE = Not established.

**TABLE 4-7. QUALITY CONTROL ACCEPTANCE CRITERIA FOR METHOD SW8020**

| | Aqueous Matrix Spike | | Solid Matrix Spike | | |
|---|---|---|---|---|---|
| Analyte[a] | Accuracy (% Recovery) | Precision (RPD) | Accuracy (% Recovery) | Precision (RPD) | Spike Accuracy (% Recovery)[d] |
| Benzene | | <30 | | <50 | 39 - 150 |
| Toluene | | <30 | | <50 | 46 - 148 |
| Ethylbenzene | | <30 | | <50 | 32 - 160 |
| Total xylenes | | <30 | | <50 | 61 - 129[b] |
| **Surrogate** | | | | | |
| 1-Bromo-4-fluorobenzene | 59 - 142[b] | NA | 58 - 152[b] | NA | NA |
| Trifluorotoluene | 50 - 150[c] | NA | 50 - 150[c] | NA | NA |

[a] Subset of method analytes spiked; if additional analytes are spiked, SW846 limits will be used to assess accuracy.
[b] Laboratory generated limits; method specifications not established.
[c] Until laboratory generated limits can be established.
[d] SW846, 3rd edition, Method SW8020; recovery range for laboratory control samples and matrix spikes.

RPD = Relative percent difference.
NA = Not applicable.

**TABLE 4-8. QUALITY CONTROL ACCEPTANCE CRITERIA FOR METHOD SW8080**

| | Aqueous Matrix Spike | | Solid Matrix Spike | | |
|---|---|---|---|---|---|
| Analyte[a] | Accuracy[b] (% Recovery) | Precision[b] (RPD) | Accuracy[b] (% Recovery) | Precision[b] (RPD) | LCS Spike Accuracy[c] (% Recovery) |
| Aldrin | 40 - 120 | <22 | 34 - 132 | <43 | 42 - 122 |
| 4,4'-DDT | 38 - 127 | <27 | 23 - 134 | <50 | 25 - 160 |
| Dieldrin | 52 - 126 | <18 | 31 - 134 | <38 | 36 - 146 |
| Endrin | 56 - 121 | <21 | 42 - 139 | <45 | 30 - 147 |
| gamma-BHC (Lindane) | 56 - 123 | <15 | 46 - 127 | <50 | 32 - 127 |
| Heptachlor | 40 - 131 | <20 | 35 - 130 | <31 | 34 - 111 |
| PCB-1242 | | | 32 - 179[d] | <64[d] | 39 - 150 |
| **Surrogate** | | | | | |
| TCMX | 60 - 150 | NA | 60 - 150 | NA | NA |
| Decachlorobiphenyl | 60 - 150 | NA | 60 - 150 | NA | NA |

[a] Subset of analytes spiked; if additional analytes are spiked, SW846 limits will be used to assess accuracy.
[b] U.S. EPA Contract Laboratory Program, OLM01.6.
[c] SW846 3rd edition, Table 3, Method SW8080.
[d] Laboratory generated limits, until project limits can be established for PCB-only samples.

RPD = Relative percent difference.
NA = Not applicable.
LCS = Laboratory control sample.

**TABLE 4-9. QUALITY CONTROL ACCEPTANCE CRITERIA FOR METHOD SW8140**

| | Solid Matrix Spike | | |
|---|---|---|---|
| Analyte[a] | Accuracy[b] (% Recovery) | Precision[b] (RPD) | Spike Accuracy[c] (% Recovery) |
| Azinphos methyl | 48 - 140 | <50 | 9 - 128 |
| Bolstar | 46 - 151 | <50 | 46 - 84 |
| Chlorpyrifos (Dursban) | 23 - 187 | <50 | 82 - 115 |
| Coumaphos | 54 - 177 | <50 | 71 - 147 |
| Demeton | 29 - 144 | <50 | 36 - 99 |
| Diazinon | 43 - 154 | <50 | 49 - 85 |
| Disulfoton | 20 - 166 | <50 | 55 - 109 |
| Fenthion | 56 - 146 | <50 | 9 - 128 |
| Methyl parathion | 37 - 164 | <50 | 80 - 112 |
| Phorate | 29 - 152 | <50 | 36 - 89 |
| Stirophos | 18 - 168 | <50 | 48 - 84 |
| **Surrogate** | | | |
| Triphenylphosphate | 36 - 140 | NA | 39 - 151[b] |

[a] Subset of analytes spiked; if additional analytes are spiked, SW846 limits will be used to assess accuracy.
[b] Laboratory generated limits, until project limits can be established.
[c] SW846 3rd edition, Table 3, Method SW8140; average recovery ± 3 times standard deviation; recovery range for laboratory control samples and aqueous matrix spikes.

RPD = Relative percent difference.
NA = Not applicable.

**TABLE 4-10. QUALITY CONTROL ACCEPTANCE CRITERIA FOR METHOD SW8150**

| Analyte | Solid Matrix Spike | | |
|---|---|---|---|
| | Accuracy[a] (% Recovery) | Precision (RPD) | Spike Accuracy[b] (% Recovery) |
| 2,4-D | 54 - 90 | <50 | 63 - 87 |
| 2,4-DB | 70 - 106 | <50 | 84 - 102 |
| Dalapon | 36 - 126 | <50 | 42 - 90 |
| Dicamba | 50 - 116 | <50 | 58 - 100 |
| Dichloroprop | 81 - 99 | <50 | 91 - 103 |
| Dinoseb | 66 - 99 | <50 | 69 - 93 |
| MCPA | 76 - 102 | <50 | 86 - 110 |
| MCPP | 82 - 108 | <50 | 82 - 106 |
| 2,4,5-T | 60 - 104 | <50 | 67 - 103 |
| 2,4,5-TP (Silvex) | 60 - 106 | <50 | 73 - 103 |
| **Surrogate** | | | |
| 2,4-Dichlorophenylacetic acid | 49 - 127 | NA | 49 - 127[a] |

[a] Laboratory generated limits, until project limits can be established.
[b] SW846 3rd edition, Table 3, Method SW8150; reagent water mean recovery ± 3 times standard deviation (Municipal water used for Dinoseb); recovery range for laboratory control samples and aqueous matrix spikes.

RPD = Relative percent difference.
NA = Not applicable.

**TABLE 4-11. QUALITY CONTROL ACCEPTANCE CRITERIA FOR METHOD SW8240**

| | Aqueous Matrix Spike | | Solid Matrix Spike | | |
|---|---|---|---|---|---|
| Analyte[a] | Accuracy[b] (% Recovery) | Precision[b] (RPD) | Accuracy[b] (% Recovery) | Precision[b] (RPD) | LCS Spike Accuracy[c] (% Recovery) |
| Benzene | 76 - 127 | <11 | 66 - 142 | <21 | 37 - 151 |
| Chlorobenzene | 75 - 130 | <13 | 60 - 133 | <21 | 37 - 160 |
| 1,1-Dichloroethene | 61 - 145 | <14 | 59 - 172 | <22 | 10 - 234 |
| Toluene | 76 - 125 | <13 | 59 - 139 | <21 | 47 - 150 |
| Trichloroethene | 71 - 120 | <14 | 62 - 137 | <24 | 71 - 157 |
| **Surrogate** | | | | | |
| 1,4-Bromofluorobenzene | 86 - 115 | NA | 74 - 121 | NA | NA |
| 1,2-Dichloroethane | 76 - 114 | NA | 70 - 121 | NA | NA |
| Toluene-$d_8$ | 88 - 110 | NA | 81 - 117 | NA | NA |

[a] Subset of analytes spiked; if additional analytes are spiked, SW846 limits will be used to assess accuracy.
[b] U.S. EPA Contract Laboratory Program, OLM01.6.
[c] SW846 3rd edition, Method SW8240.

RPD = Relative percent difference.
NA = Not applicable.
LCS = Laboratory control sample.

**TABLE 4-12. QUALITY CONTROL ACCEPTANCE CRITERIA FOR METHOD SW8270**

| Analyte[a] | Aqueous Matrix Spike | | Solid Matrix Spike | | LCS Spike |
|---|---|---|---|---|---|
| | Accuracy[b] (% Recovery) | Precision[b] (RPD) | Accuracy[b] (% Recovery) | Precision[b] (RPD) | Accuracy[c] (% Recovery) |
| **Base/Neutral Extractables** | | | | | |
| Acenaphthene | 46 - 118 | <31 | 31 - 137 | <19 | 47 - 145 |
| 1,4-Dichlorobenzene | 36 - 97 | <28 | 28 - 104 | <27 | 20 - 124 |
| 2,4-Dinitrotoluene | 24 - 96 | <38 | 28 - 89 | <47 | 39 - 139 |
| n-Nitrosodipropylamine | 41 - 116 | <38 | 41 - 126 | <38 | 0 - 230 |
| Pyrene | 26 - 127 | <31 | 35 - 142 | <33 | 22 - 147 |
| 1,2,4-Trichlorobenzene | 39 - 98 | <28 | 38 - 107 | <23 | 44 - 142 |
| **Acid Extractables** | | | | | |
| 4-Chloro-3-methylphenol | 23 - 97 | <42 | 26 - 103 | <33 | 22 - 147 |
| 2-Chlorophenol | 27 - 123 | <40 | 25 - 102 | <50 | 23 - 134 |
| 4-Nitrophenol | 10 - 80 | <50 | 11 - 114 | <50 | 29 - 182 |
| Pentachlorophenol | 9 - 103 | <50 | 17 - 109 | <47 | 14 - 176 |
| Phenol | 12 - 110 | <42 | 26 - 90 | <35 | 5 - 112 |
| **Surrogates[b]** | | | | | |
| 2-Fluorobiphenyl | 43 - 115 | NA | 30 - 115 | NA | NA |
| 2-Fluorophenol | 21 - 100 | NA | 25 - 121 | NA | NA |
| Nitrobenzene-$d_5$ | 35 - 114 | NA | 23 - 120 | NA | NA |
| Phenol-$d_5$ | 10 - 94 | NA | 24 - 113 | NA | NA |
| p-Terphenyl-$d_{14}$ | 33 - 141 | NA | 18 - 137 | NA | NA |
| 2,4,6-Tribromophenol | 10 - 123 | NA | 19 - 122 | NA | NA |

[a] Subset of analytes spiked; if additional analytes are spiked, SW846 limits will be used to assess accuracy.
[b] U.S. EPA Contract Laboratory Program, OLM01.6.
[c] SW846 3rd edition, Table 6, Method SW8270; recovery range for laboratory control samples.

RPD = Relative percent difference.
NA = Not applicable.
LCS = Laboratory control sample.

## TABLE 4-13. QUALITY CONTROL ACCEPTANCE CRITERIA FOR METHOD SW8310

| Analyte | Spike Accuracy[a] (% Recovery) |
|---|---|
| Acenaphthene | D-124 |
| Acenaphthylene | D-139 |
| Anthracene | D-126 |
| Benzo(a)anthracene | 12-135 |
| Benzo(a)pyrene | D-128 |
| Benzo(b)fluoranthene | 6-150 |
| Benzo(ghi)perylene | D-116 |
| Benzo(k)fluoranthene | D-159 |
| Chrysene | D-199 |
| Dibenzo(a,h)anthracene | D-110 |
| Fluoranthene | 14-123 |
| Fluorene | D-142 |
| Indo(1,2,3-cd)pyrene | D-116 |
| Naphthalene | D-122 |
| Phenanthrene | D-155 |
| Pyrene | D-140 |
| **Surrogate** | |
| Terphenyl-$d_{14}$[b] | aqueous samples 22-157<br>solid samples 20-188 |

[a] These criteria will be used until project limits can be established.
[b] Laboratory-generated limits will be used to assess surrogate spikes until project limits can be established.

D = Detected

spike duplicates or laboratory duplicates. Total precision is a measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate or replicate field samples, and incorporates variability introduced by the analytical procedure, sample collection and handling, and matrix factors. Precision data must be interpreted by taking into consideration these possible sources of variability. Duplicate (2) samples or spiked samples will be analyzed to assess field and analytical precision for McClellan AFB RI/FS tasks and the results will be assessed using the relative percent difference (RPD) between duplicate measurements.

The results from an historical evaluation of QC results for groundwater samples collected and analyzed for volatile organic analytes and metals (Level 3 analyses) between October 1986 and November 1990 for various McClellan AFB projects indicate that total precision, evaluated by the average RPD between field duplicates, was within the initially established QAO of less than 50 percent. For field duplicate results, the average RPDs for the data collected over the four-year period range from 12.1 to 23.4 percent for organic analytes, and from 2.3 to 40.9 percent for metals.

The greatest degree of uncertainty that should be expected from sampling and analysis procedures is represented by the upper limit of the 95 percent confidence interval about the average RPDs. These upper limit values reflect precision estimates for each analyte and have been used to adjust the precision QAOs for analytes in aqueous samples measured by Method SW8010 to an RPD of less than 30 percent for field duplicate results. Because of the similarity in Method SW8010 and SW8020 data, the QAOs for analytes in aqueous samples measured by Method SW8020 have also been adjusted to less than 30 percent for field duplicate RPDs.

The RPD range for metals is influenced by the low concentrations reported because at low concentrations, even a small difference in magnitude between pairs results in a high RPD. Therefore, the QAOs for aqueous inorganic analyses, as well as all other analyses (both aqueous and solid matrices) will be less than 50 percent for field duplicate RPDs.

The results of the historical data assessment also indicate that analytical precision, evaluated by the average RPD between matrix spike duplicates, is within the QAOs of less than 20 percent for inorganic analytes and less than 30 percent for organic analytes. The

average RPD for five representative organic analytes ranged from 6.9 to 16.9 percent. The average RPD for metals ranged from 1.4 to 4.5 percent. Because this evaluation is matrix- and laboratory-specific, no adjustments to the matrix spike duplicate QAOs have been made at this time.

Precision objectives are presented for each analytical level, method, and matrix in Tables 4-1 through 4-13.

## 4.2 Accuracy

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

The results of matrix spike analyses were also included in the previously discussed historical data assessment. The calculated control limits (the mean recovery plus or minus three times the standard deviation) for most analytes showed a very similar, or in some cases a tighter range than those recommended in the methods, indicating that the system is within the accuracy objectives. Exceptions are for analytes such as silver, which may precipitate as silver chloride because of sample preservation, and low molecular weight volatile organic analytes such as chloromethane and vinyl chloride, which are known to be difficult to recover from environmental samples.

Analytical accuracy objectives are presented for each analytical level method and matrix in Tables 4-1 through 4-13.

Both accuracy and precision are calculated for specific sampling or analytical batches, and the associated sample results must be interpreted by considering these specific measures. Application of calculated precision and accuracy to measurement sample results is discussed in Section 13.0. An additional consideration in applying accuracy and precision is the concentration level of the samples; a procedure capable of producing the same value within 50 percent would be considered precise for low-level (near the detection limit) analyses of minor constituents, but would be unacceptable, and possibly useless, for major constituents at high concentrations.

## 4.3 Completeness

Completeness is calculated for the aggregation of data for all methods for any particular sampling event or other defined set of samples. The number of valid, unqualified results, divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness for the data set. The objective for completeness is 95 percent for aqueous samples and 90 percent for soil samples. If there were any instances of samples that could not be analyzed for any reason (holding time violations in which resampling and reanalysis were not possible, samples spilled or broken, etc.), the numerator of this calculation becomes the number of valid, unqualified results minus the number of possible results not reported.

## 4.4 Representativeness

Objectives for representativeness will be defined for each sampling and analysis task and will be a function of the investigative objectives. Representativeness is achieved in part, through use of the standard field, sampling, and analytical procedures described in this QAPP. Representativeness is also determined or influenced by appropriate program design, with consideration of elements such as proper well locations, drilling and installation procedures, or sampling locations. Representativeness is addressed during the planning and DQO development stages where representatives for each technical discipline involved in producing and using the data participate to ensure that appropriate objectives are established and procedures that will satisfy the objectives are selected. Decisions regarding sample/well/boring locations, numbers, and analytical methods are documented in the SAP, which is reviewed and approved before field activities are initiated.

## 4.5 Comparability

Comparability is the confidence with which one data set can be compared to other data. The objective for this QA/QC program is to produce data with the greatest degree of comparability possible. The number of matrices that will be sampled, and the range of field conditions encountered, must be considered in ultimately determining comparability. Comparability will be achieved by using standard methods for sampling and analysis, reporting data in standard units, and using standard and comprehensive reporting formats. Complete field documentation using standardized data collection forms also supports the assessment of comparability. Analysis of performance evaluation samples will be used to provide additional information for assessing the comparability of analytical data produced among subcontracting laboratories.

## 4.6 Geologic Data Quality Assurance Objectives

Geologic data will be obtained from the drilling of numerous soil borings and monitoring wells during an RI/FS. While it is more difficult to measure the precision, accuracy, completeness, representativeness, and comparability of the data collected from these activities, several steps will be undertaken to ensure the data obtained are representative, comparable, and as accurate as possible. Specifically, all geologists who will be performing lithologic logging will review the geologic samples and cores currently available through McClellan AFB Environmental Management (EM). This review will familiarize the geologists with the types of lithologies that may be encountered in drilling and provide the opportunity to standardize sample lithologic description methods. Logged sediments will be examined using a hand lens, and will be identified and described using:

- The Unified Soil Classification System;
- Munsell color charts; and
- Standard geologic descriptions and field tests, including dilatancy, plasticity, grain size and distribution, angularity or roundness percent, mineral content, organic content, and silt-clay-sand percent.

Geologists new to McClellan AFB will be accompanied by an experienced geologist for training to promote comparability and representativeness in procedures and documentation. Standardized lithologic logging, well completion and installation, and materials and sample collection forms will be used to document boring or well drilling activities. This will also promote comparability of the information collected. Additional geologic information that may be required to satisfy the DQOs for a specific site or activity will be documented in the SAP, and standardized forms will be developed and additional training will be conducted prior to the start of the activity.

If cores are to be screened for evidence of contaminants or if samples are to be collected for physical or chemical analysis, sample collection and handling procedures documented in this QAPP will be followed and documented on standard forms described in Section 5.2.6. Specific sample collection criteria and sampling locations will be stated in the site-specific DQOs in the SAP and field task instructions. Field screening instrument calibrations will be conducted according to the procedures presented in Section 7.0 of this QAPP and in Standard Operating Procedures for instrument calibration, use, and documentation of those measurements.

## 4.7 Hydrologic Data Quality Assurance Objectives

The compilation of hydrologic data will be conducted in several parts of the RI/FS to satisfy the DQOs for each phase or activity. The DQOs for each type of hydrologic measurement will dictate the type of equipment and the relative accuracy and precision needed. Tolerances developed by the U.S. Geological Survey measurements related to hydrologic data are presented in Table 4-14, and were compiled from Stallman (Techniques of Water Resources Investigations of the U.S. Geological Survey, Chapter B1: "Aquifer Test Design, Observation, and Data Analysis," U.S. Department of the Interior, 1971). Hydrologic data to be compiled will consist primarily of water-level measurements collected during well installation and development and over specific time intervals. Water levels will be measured in quarterly soundings, during monitoring well purging, and during aquifer pumping tests.

The water levels will be measured with calibrated electronic sounders for quarterly soundings and well purging prior to sampling. Electronic sounders will be checked for accuracy and precision before each use, and each sounder will have a unique identification

**TABLE 4-14. HYDROLOGIC DATA QUALITY ASSURANCE OBJECTIVES**

| |
|---|
| Control-well discharge (±10%) |
| Depth to water measurements with an electric sounder or transducer (±0.01 feet) |
| Distance from control well to each observation well (±0.5%) |
| Synchronous time (±1% of time since pumping started or stopped) |
| Elevations of measuring points (±0.01 feet) |
| Vertical distance between measuring point and land surface (±0.01 feet) |
| Total depths of all wells (±1%) |
| Depth and length of screened intervals of all wells (±1%) |
| Diameter, casing type, screen type, and method of construction of all wells (nominal) |

number that can be used to document instrument use and performance. If more than one sounder is used during a sounding event, sounders will be checked for comparability of measurements and calibrated with respect to one standard sounder prior to use.

When water-level measurements are required in one or more wells during pumping tests, pressure transducers will be suspended in each well to collect the measurements. The transducers will be connected by electronically shielded cable to a multi-channel data logger. The transducers will be calibrated and checked for precision, accuracy, and comparability of pressure readings using hand-held, water-level probes at the beginning and throughout the duration of the test. The required accuracy for transducer measurements will be $\pm 2$ percent of its full scale reading. The data logger will compile and store the measurements electronically.

Additional hydrologic data that may be collected during the RI/FS are pump discharge rates measured during well purging or aquifer testing. The accuracy of the method needed for pump discharge rate measurements will be determined by the intended uses of the discharge data. For monitoring well purging, discharge data are needed to demonstrate that the well to be sampled was adequately purged and that the discharge was sufficient to induce groundwater flow from the formation into the well casing. Discharge measurement to assure the adequacy of purging can be obtained with sufficient accuracy by allowing purged water to fill a bucket calibrated in 1-gallon increments while the time interval to reach a 3- to 5-gallon volume is measured with a stopwatch.

For aquifer testing in which greater discharge rates are needed to adequately stress an aquifer, a calibrated flow meter will be placed in the discharge line near the wellhead to obtain sufficient accuracy, precision, and comparability. A flow meter calibrated to accurately measure 1 gallon per minute of discharge will be used to collect flow data for aquifer tests. The meter should also record total flow from the well over the entire length of the pump test. The use of a calibrated flow meter-totalizer will assure accuracy within $\pm 10$ percent of the discharge rate and total volume discharged. The meters will be calibrated using a calibrated volumetric container and stopwatch at the beginning and end of the test.

Hydrologic measurements will be documented on standardized forms that are developed for the specific activity (e.g., monitoring well sample collection forms, aquifer test forms). Electronically stored data from the data logger will be identified by well number,

dates of testing, and equipment numbers, and downloaded to a computer and stored using magnetic media. Examples of standard forms for these activities are described in Sections 5.2 through 5.4. Completed forms are maintained in project files after the data are checked for quality and entered into electronic databases for analyses and presentation.

# 5.0 FIELD PROCEDURES

This section contains detailed descriptions of field procedures and sampling protocols that will be used during remedial investigations and other field activities at McClellan Air Force Base (AFB). The specific sample locations and field procedures to be used for a sampling activity will be selected during the Data Quality Objective (DQO) process, and documented in the Sampling and Analysis Plan (SAP) or Field Sampling Plan (FSP) for the effort. Task instructions will also be developed for a field or sampling activity to provide field staff with schedules, responsibilities, quality control (QC) requirements, and other logistical information required to conduct the activity.

All field personnel, their supervisors, and project management and administrative personnel will read pertinent sections of the Quality Assurance Project Plan (QAPP). In addition, field personnel will have a copy of the QAPP in their possession.

## 5.1 Geophysical Techniques

Borehole and surface geophysical techniques will be used during field investigations at McClellan AFB. The techniques that may be used for each type of geophysical investigation are described.

Borehole geophysical techniques are used to assist in determining subsurface geologic conditions such as strata thickness, lithology, and permeability. These borehole methods may also be valuable in determining the presence and relative natural quality of groundwater. Geophysical logs are generally used in conjunction with borehole lithology logs to refine geologic descriptions. There are several types of borehole geophysical techniques that may be employed during remedial investigation/feasibility study (RI/FS) site characterization activities at McClellan AFB. These techniques include resistivity logging, spontaneous potential logging (SP), and gamma ray logging, and are discussed below. All geophysical measurements will be collected digitally to allow scale adjustments to be made prior to hard copy reproduction. In addition, any required permits will be obtained before using radioactive sources for borehole geophysical techniques. Much of the text in this section that presents the theory and general procedures for borehole geophysical techniques are from Groundwater and Wells, Fletcher G. Driscoll, Ph.D., 1986.

Surface geophysical techniques are used to identify subsurface structures or locate buried objects such as tanks, pipes, or other man-made objects. Techniques that may be used include ground penetrating radar (GPR) and magnetometry. The appropriate technique for use at a site will be dictated by the DQO for the site, which will generally be to locate the boundaries of suspected or known underground objects. The suspected objects and locations will be described and shown on site maps presented in the SAP or FSP. The surface geophysical surveys will be conducted within pre-determined grids along transect lines established for each site or area of interest. The spacing of the grids will be based on information regarding the approximate dimensions of the features to be located. These surveys will be conducted by qualified subcontractors, supervised by a project geologist or engineer. Location and elevation information sufficient to map and assess the survey results will be recorded. This may include northing and easting from a surveyed reference point, measurements in a third order survey, depth below ground surface, and/or professionally surveyed points and transects, depending on the required level of accuracy and detail needed to satisfy the objectives for the survey.

### 5.1.1 Borehole Geophysical Investigation Procedures

Geophysical logging will be completed at a selected number of boreholes. Typically, only the deepest borehole or well in a monitoring well cluster will be logged. Resistivity, SP, and caliper logs will be run in boreholes drilled by the direct mud-rotary method. Natural gamma logging may also be conducted in boreholes along with resistivity and SP methods to augment the correlation of electric and natural gamma logs that have been completed on various wells and boreholes in the past.

Borehole geophysical logging will be conducted by a qualified contractor under subcontract to either the contractor or contractor's drilling subcontractor. A project geologist or engineer will supervise and observe all logging activities. Cell cables, probes, and other equipment will be steam cleaned prior to being lowered into a borehole or monitoring well.

In cases where geophysical logs are being run in an open borehole prior to well construction, the data will be evaluated to optimize the well design. Specifically, the geophysical logs will be used to determine the most appropriate intervals for the screen, sand pack, and bentonite seal.

Geophysical data collected in boreholes will be stored in hard-copy and electronic formats. After logging, a reproducible copy of the field log will be maintained in the project file. Digital measurements and borehole identification data will be stored on floppy disk.

#### 5.1.1.1 Electric Logs

Resistivity logging is typically conducted in conjunction with spontaneous potential logging. These two methods, when used together, are commonly referred to as electric or "E" logs. Electric logging can only be used in uncased boreholes that are filled with water or another drilling fluid; therefore, electric logging is typically used to log boreholes drilled with the mud rotary method.

**Borehole Resistivity Logs**

A resistivity log can provide a detailed representation of the character and thickness of different strata and an indication of natural groundwater quality by measuring the apparent electrical resistivity of the materials surrounding a borehole. Variations in resistivity are caused primarily by differences between the character of the subsurface strata and the mineral content of the associated groundwater. Factors that influence the response of the resistivity log are relative resistivities of borehole fluids and interstitial fluids, the diameter of the borehole and the distance between the electrodes on the geophysical probe. Borehole lithology logs should be used with resistivity logs when attempting to identify formation material because of these influencing factors.

**Spontaneous Potential Logs**

Spontaneous potential logs are usually run in conjunction with resistivity logs. Spontaneous potential logs measure the naturally occurring electrical potentials (voltage) that result from physical and chemical changes at contacts between differing types of subsurface geologic materials. For example, electrical potentials occur spontaneously at the contact surface between a sand stratum and an underlying clay stratum, or between sand formations and igneous rock. In a borehole, electrical potentials also occur between the drilling fluid and the formation, and the drilling fluid and the borehole filter cake.

Resistivity and SP surveys will be logged downhole at a common scale whenever possible. The appropriate scale will be determined in the field by conducting offset logs prior to running the final survey. Offset logs are a quality assurance (QA) and calibration step that involves logging an upper or lower portion of the borehole and adjusting the response of the log to obtain the optimum scale. Scales to be adjusted are the horizontal (millivolts for spontaneous potential and ohm-meters for resistivity) and vertical (feet). The resistivity logs will consist of the short-normal (16-inch) and long-normal (64-inch) configurations.

The following information will be included on the resistivity and SP survey logs:

1) Well number;
2) Date and time;
3) Log recorder;
4) Equipment number;
5) Depth and diameter of borehole;
6) Type of fluid in borehole;
7) Instrument length and diameter;
8) Rate of descent and ascent; and
9) Resistivity of drilling mud.

### 5.1.1.2 Natural Gamma Ray Logs

Natural gamma ray logging is a borehole geophysical method used to estimate lithologic characteristics of geologic formations by recording gamma radiation emission. Gamma logging may be used in conjunction with spontaneous potential, resistivity, and caliper logs in boreholes drilled by mud rotary methods. The gamma ray logging technique will be used in boreholes drilled at McClellan AFB only at locations in which the mineral constituents of the geologic formations indicate the log will provide additional interpretive information.

Gamma radiation is emitted from certain elements present in geologic materials that are unstable and decay spontaneously into more stable elements. Certain radioactive elements, such as potassium-40 and the decay products of thorium and uranium, occur naturally in igneous and metamorphic rock and as depositional particles in sedimentary rock.

Clay minerals, mica, and feldspar normally emit natural gamma radiation due to the presence of potassium-40. Conversely, quartz contains no potassium and does not produce gamma radiation. Therefore, gamma ray logs generally indicate more counts per unit of time for sediments such as clay and silt, and fewer counts per unit of time for quartz-rich sand. Interpretive problems can occur when a sand formation includes a significant proportion of feldspar grains. In such cases, it may not be possible to differentiate between fine-grained materials, such as silt and clay, and feldspar-rich sand. Similarly, interpretative problems can arise where silt and clay-size particles contain little or no potassium and respond with low counts per unit time. These lithologic conditions appear to be the case at McClellan AFB indicating that gamma ray logs may not be clearly or easily interpreted. Other factors, including borehole radius and the nature of gamma radiation emission and detection, must be considered when interpreting gamma logs. On the basis of previous gamma ray logging results, the technique does not provide sufficient additional data to warrant widespread use. Therefore, the technique will be used sparingly in RI field efforts.

If natural gamma ray logs are employed at McClellan AFB during the RI the following information will be provided on the logs:

1) Well number;
2) Date and time;
3) Equipment number;
4) Length and diameter of tool;
5) Location of scintillation probe on tool;
6) Time constant;
7) Velocity of detector; and
8) Casing depth, type, and diameter, and cement type.

#### 5.1.1.3 Caliper Logs

Caliper logs are used to measure the diameter of a borehole. This method is useful itself and in conjunction with other geophysical methods. A caliper log consists of a mechanical device, with one to four adjustable legs, and a probe that can measure the diameter of the borehole. By knowing the diameter of the borehole, such factors as borehole erosion, the presence of swelling clays or resistant strata, and the volume of filter pack of grout needed for well completion can be determined. Caliper logs are operated by lowering

the device into the borehole and recording the measurements as the caliper is withdrawn. The following information will be included on each caliper log:

1) Well number;
2) Date and time;
3) Log recorder's name;
4) Equipment number;
5) Depth of borehole; and
6) Maximum and minimum tool diameters.

### 5.1.2 Surface Geophysical Investigation Procedures

Surface geophysical investigations will be conducted using GPR and/or magnetometry techniques to identify subsurface features or conditions at selected sites. Location data, instrument numbers, calibration information, and geophysical interpretation information and maps for all geophysical surveys will be stored in hard-copy format in the project files. Survey data may entered into electronic files for mapping.

#### 5.1.2.1 Ground Penetrating Radar

Ground penetrating radar is a shallow geophysical survey system that provides a continuous real-time cross section of shallow subsurface conditions. The instrumentation used for this investigation will be an SIR-3 system manufactured by Geophysical Survey Systems, Inc. or equivalent instrumentation. The system consists of a radar control unit, signal processing and conditioning circuitry, and a graphical recorder all housed in a single metal case. This unit is connected by an electrical umbilical cable to a transducer. As the transducer (antenna) is towed along a traverse, it transmits radar impulses downwards into the ground. At interfaces where changes in the electrical properties of the subsurface occur, the radar impulse typically undergoes an abrupt change in velocity causing some of the radar energy to be reflected back to the antenna on the ground surface. The amount of energy that is reflected is dependent on the contrast of the respective radar velocity. The time it takes for the radar signal to travel from the antenna to a reflecting interface and back to the antenna is directly proportional to the depth of the interface. Recording these depth-dependant impulses on a scanning, time-based graphic chart recorder results in a cross-section depicting the longitudinal distribution of subsurface strata and other features over which the radar antenna has passed.

#### 5.1.2.2 Magnetometry

Magnetometer surveys consist of measuring variations in the earth's magnetic field. Measurements of the magnetic gradient can be used to locate buried ferrous objects such as tanks, pipelines, and metallic debris.

Magnetometer surveys will be conducted using a Geometrics model 856AG proton precession magnetometer/gradiometer or its equivalent. The magnetometer has two sensors and an electronics package. The magnetometer can collect both total field data and vertical gradient data. The magnetometer can discriminate to 0.2 gammas in a total field of 40,000 to 60,000 gammas. Magnetic readings are stored in memory with the time of day, station numbers, and line numbers of the readings. A base station for magnetic readings will be established at the start of each day's measurements. Magnetic readings will be collected and recorded in the morning, at noon, and at the end of day to evaluate drift in the instrument.

## 5.2 Drilling

### 5.2.1 Site Selection

The selection of locations and depths for monitoring well and subsurface soil sample sites will be a cooperative effort of the contractor, the United States Air Force (USAF), and involved federal and state regulatory agencies.

Criteria for the selection of monitoring well and subsurface sampling locations will be based on specific objectives of each drilling or sampling effort. However, the locations will generally depend upon the following:

- Suspected presence or absence of contamination in the vadose and saturated zones;
- Current or historic groundwater gradients and flow directions;
- The need to further characterize the geologic conditions;

- Proximity to past waste disposal sites;
- Proximity to receptors such as water supply wells;
- Favorable access for drilling equipment;
- Presence of overhead power or telephone lines; and
- Location of underground structures such as sewer lines, power lines, and water lines.

Final selection of a site will be dependent on securing all necessary rights-of-entry, clearances, and permits. Prior to drilling, electrical cable and pipe-locator instruments are used in conjunction with underground utility maps, magnetometer readings, and ground penetrating radar to determine if underground utilities underlie the drilling location.

Sites selected for monitoring well installation will also be based on review of available hydrogeologic and analytical data. All proposed monitoring well sites and other borehole locations will be evaluated prior to drilling by examining hydrogeologic and contaminant distribution maps.

### 5.2.2 Drilling Preparation

Prior to beginning drilling activities at any site, the contractor's Field Coordinator (FC) will meet with representatives of McClellan AFB to verify that all necessary permits and clearances have been granted for each well. Only then will drilling equipment be mobilized to a well site. The contractor will be responsible for obtaining all off-base permits necessary for monitoring well installation and/or soil borings. United States Air Force protocol requires McClellan AFB Environmental Management (EM) to obtain on-base permits; the contractor is responsible to ensure this is done for all on-base locations.

The FC shall be present each time the drill rig is moved to a well site. Prior to positioning the rig, the entire drill site shall be covered with 6-mL-thick polyethylene film to protect the immediate area from possible contact with contaminated drill cuttings and to facilitate cleanup efforts (see Section 5.2.5). In addition, where containment of fluids is a concern (i.e., drilling fluids, groundwater), lumber (4 inch x 4 inch) or polyvinyl chloride

(PVC) pipe shall be utilized to berm the edges of plastic sheeting for the entire area covered with plastic. This berm must be sufficient to prevent any fluids from running off sheeting during drilling operations. The FC shall also supervise positioning of the rig such that the center of the borehole is within 0.5 feet of the predetermined well location (identified by a stake or paint mark). Such accuracy is necessary to avoid underground utilities, possible violation of property lines, rights-of-entry, or other agreements that have been negotiated with private property owners or McClellan AFB personnel.

Prior to drilling a new monitoring well or a temporary borehole, the drill string (casing, auger, drill pipe, bit, etc.) and rear portions of the drill rig shall be cleaned by a high pressure, hot water wash ($>180°F$ and $>200$ psi) in the designated decontamination area. Upon completion of decontamination and mobilization of drilling equipment to the drill site, exclusion and support zones as referenced in the Health and Safety Plan shall be identified and marked prior to commencement of drilling operations. All health and safety equipment (tables, water, eye wash station, etc.) will also be set up at this time and shall be located in an upwind direction. The Supervising Rig Geologist (SRG)/or rig technician shall be responsible for this effort. Also, the rig technician with the assistance of the SRG will be on site to monitor borehole and cuttings vapors during drilling.

### 5.2.3 Drilling Methods

Monitoring well drilling and construction methods will be selected to best meet the following criteria:

- Ability to reliably determine the occurrence and thickness of water-bearing zones and interlayered low permeability zones during drilling;
- Ability to accurately place monitoring well screens at desired depths;
- Ability to meet all local, state, and U.S. Environmental Protection Agency (U.S. EPA) requirements for well construction and development;
- Ability to collect samples for laboratory analysis of selected chemical compounds;

- Ability to obtain samples of geologic material and provide for the preparation of detailed lithologic logs; and

- Speed and overall reliability of the technology.

Drilling methods that may be used at McClellan AFB for installation of monitoring wells, observation wells, and subsurface soil sample recovery include the following:

- Hollow-stem auger (HSA);
- Direct circulation mud rotary;
- Air rotary casing drive;
- Dual tube percussion;
- Bucket auger; and
- Sonication

During SAP or FSP development for new RI projects, new technologies may be evaluated and recommended, if appropriate.

Precautions will be taken with all drilling methods to minimize the potential for cross-contamination of water-bearing zones. The precautions will include diminishing the flow of contaminated groundwater into the boring and performing grouting procedures in a manner that restores the integrity of the aquifer as quickly as possible.

Cuttings, waste, or drilling muds produced by any of the drilling methods will be managed in accordance with procedures described in Section 5.2.5.

**Hollow-Stem Augering**

The HSA drilling method employs a hollow helical steel drill tool that is rotated to advance the boring and lift formation materials (cuttings) to the surface. The flights for the hollow stem auger are welded onto steel pipe and a cutter head is attached to the "lead" (bottom) auger to cut the hole. During drilling, a center bit is inserted into the hollow area of the cutter head that prevents cuttings from entering the hollow portion of the auger. Generally, the center bit is flush with or extends no more than 1/2 foot below the cutter head. The center bit connects through the auger flights by small diameter drill rods

and is attached to the top-head drive unit of the drill rig. The top-head drive is powered by a truck mounted engine that mechanically rotates the entire flight of augers. The hollow opening allows the insertion of sampling tools (i.e., split-spoon sampler, MOSS®-system, etc.) and well completion materials with the augers in place to support the borehole. The HSA technique will be used when collection of analytical samples in the vadose zone is planned or on occasions when a single monitoring well is to be completed at the water table and a geophysical log is not required. Figure 5-1 shows a typical hollow-stem auger rig.

**Direct Circulation Mud Rotary**

The direct circulation mud rotary drilling method employs a bit that attaches to the lower end of a drill pipe and advances the boring as the drill pipe is rotated. In direct circulation rotary drilling, a drilling mud is pumped down through the drill pipe and out through the ports or jets in the drill bit. The mud consists of a mixture of water and bentonite powder. The mud then flows upward in the annular space between the hole and drill pipe, and carries the cuttings in suspension to the surface. At the surface, the mud is channeled across a shaker, and then into a settling pit or pits where most of the remaining cuttings drop out. The mud is then recirculated back down the borehole. The functions of the mud are to: transport the cuttings, support and stabilize the borehole, seal the borehole to prevent fluid loss, allow the cuttings to be separated at the surface, cool and clean the bit, and lubricate the bit. The mud weight and viscosity will be monitored and recorded at the start of drilling each day and at two-hour intervals during drilling unless mud conditions (e.g., increased or decreased viscosity, etc.) or drilling conditions (e.g., lost circulation) change rapidly in the borehole. Under normal drilling conditions, sand contents of 4 percent or less, viscosities of 35 to 40 seconds, and mud weights of 9 to 9.5 pounds/gallon should be maintained. Under abnormal conditions, such as lost circulation, caving, increasing solids content, etc., the FC or a Registered Geologist should be consulted. The rate of penetration depends on the type of subsurface formation, size of hole, type of bit, mud weight and viscosity, and weight on the bit. The use of the direct rotary method will allow resistivity and spontaneous potential geophysical surveys to be conducted. The subsequent geophysical survey will assist in the drilling and construction of the other wells in the cluster. After drilling the borehole, drilling mud is managed according to procedures described in Section 5.2.5.

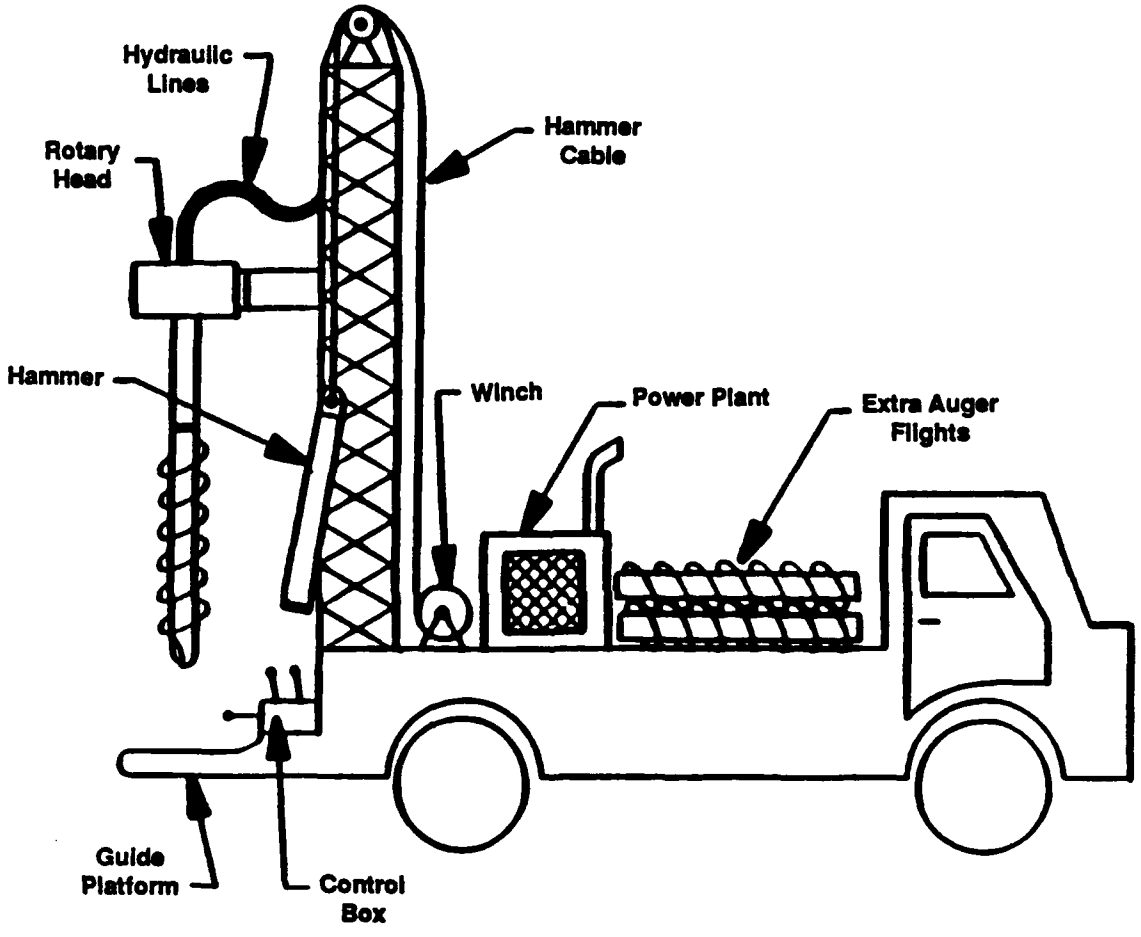


Figure 5-1. Typical Hollow Stem Auger Rig

0491-078-3

**Air Rotary Casing Drive**

This method of borehole drilling is preferred at locations where: drilling mud is not required for geophysical logging, a monitoring well will be constructed, and there is potential for downward migration of contaminated groundwater in the boring during drilling. The method is cost-effective for producing borings for well construction when continuous core and geophysical logs are not required. The air rotary casing drive drilling method uses a drilling bit attached to the lower end of a rotating drill pipe that is placed within a nonrotating outer "drive" casing. The drill bit advances the boring simultaneously as the nonrotating drive casing is driven into the ground. Air is forced under pressure downward through the drilling rods and bit and back to the ground surface through the annulus between the drive casing and the smaller rotating drill rod. The returning air continuously moves cuttings and groundwater from the area of the drill bit to the surface where they are discharged into a cyclonic separator, which separates the air from the formation cuttings to facilitate sampling and containment of drill cuttings. The outer casing is driven downward by repeated blows of a percussion hammer mounted on the drilling rig. The drive casing keeps the borehole open and reduces the flow of groundwater into the boring. Because of the reduction of groundwater flow into the boring, the potential for cross contamination between separate waterbearing zones penetrated during drilling is diminished. The drive casing is pulled out of the boring with hydraulic jacks during well construction. Groundwater brought to the surface during drilling is collected in a tank and transported from the drill site in a vacuum truck. The rate of penetration using this drilling method depends on the type of subsurface formation, size of the hole, type of bit, size of the drive casing, and the amount of pressurized air forced down the drill pipe. When using this method for drilling in the saturated zone, the drill bit shall never be advanced more than 1 foot deeper than the bottom edge of the drive casing. A typical air rotary casing drive rig is shown in Figure 5-2.

**Dual Tube Percussion**

Similar to the air rotary casing drive method, this method is effective for drilling borings for the construction of monitoring wells when continuous core and geophysical logs are not required. The potential for contaminant migration in borehole liquids is diminished by this method. The dual tube percussion method utilizes small diameter drill pipe and larger diameter drive casing with high pressure air as the circulating medium. Air is forced down the annular space between the inner drill pipe and outer drive

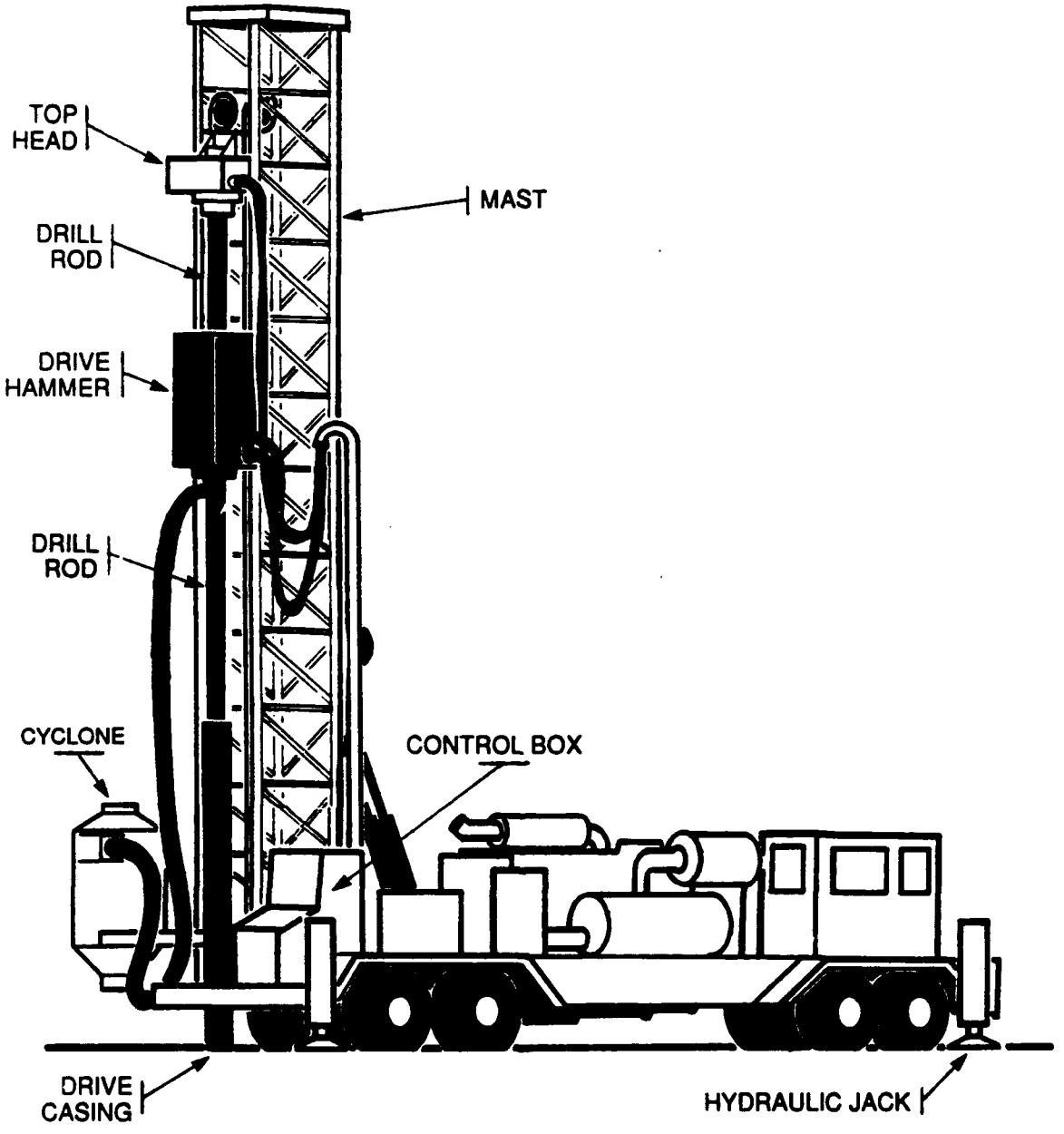


**Figure 5-2. Typical Air Rotary Casing Drive Rig**

0491-078-4

casing and cuttings are returned to the surface up the inside diameter of the drill pipe. The drill pipe is advanced by repeated blows of an aboveground percussion hammer. Impact from the hammer is applied to the outer drive casing only, therefore very little space exists between the outer drive casing and surrounding formation (soil). The drive casing keeps the borehole open and as in the air rotary casing drive method, diminishes the potential for cross contamination of water-bearing zones. The cuttings are discharged into a cyclonic separator, which separates the air from the formation cuttings to facilitate sampling and containment of drill cuttings. Groundwater produced during drilling is collected in a tank and transported from the drill site in a vacuum truck. The rate of penetration depends on the type of subsurface formation, size of hole, size of the drive casing, and the volume of air forced down the drill pipe.

**Bucket Auger**

This method of drilling will be used at locations in which a larger diameter (20 to 25 inches) boring is needed for sampling or well construction to depths in the vadose zone. The bucket auger method utilizes a large (16-inch to 52-inch) diameter bucket auger to drill and remove soil. As soil is collected in a cylindrical bucket, the hinged bottom of which has auger-type cutting blades and teeth and is attached to the lower end of a kelly bar. The kelly bar extends by telescoping and moves up and down through a rotary table that is connected to the drill rig. Once the extension limit of the telescoping kelly has been reached, drill pipe must be added in order to drill deeper. When the bucket becomes full of soil, it is raised to the surface and dumped in an adjacent waste area (i.e., roll off bin). Bucket auger drilling is primarily utilized under conditions that require a large diameter borehole in deposits that will not collapse if they are not supported by drive casing, conductor pipe, or drilling mud and in which groundwater will not be encountered. The bucket auger drilling method will be utilized when a large diameter hole is needed for the emplacement of conductor pipe. Management of cuttings follows the procedures described in Section 5.2.5.

**Sonication**

This method will be used for drilling at locations where continuous core samples must be collected from the vadose zone. The method is most effective in rapidly producing borings sufficient for soil, soil gas, and Hydropunch® groundwater sampling with no additional cuttings generation. The sonication (sonic) drilling method employs a sonic

drill head that advances the drill stem into the soils by vibration. The drill head produces vibrations normal to the ground surface by rotating (9,000 revolutions per minute) two 50-pound counterweights. Operational frequency is between 70 and 150 hz. At optimum frequencies (between 120 and 150 hz) the drill head produces approximately 30,000 pounds of downward/upward force. The drill bit attached to the bottom of the drill stem moves downward by breaking cohesive forces in soils or rock with vibrations. A split-spoon sampler or a solid drive point can be latched into the bottom of the drill stem to collect core samples or drill to a desired sampling depth. Soil is either forced into the split-spoon sampler or is forced aside as the drill stem is advanced. In hardpan or bedrock, the bit is rotated to cut through the material.

### 5.2.4 Sample Collection Methods

Core or soil samples may be collected while drilling with any of the above drilling methods. The core or soil sampling system selected depends on the type of drilling method being used and the type of sample that is desired (i.e., a continuous core sample for lithologic logging or a soil sample from a discrete depth for physical or chemical testing).

#### Split-Spoon Sampling

Hollow-stem augering, bucket augering, and sonication are the drilling methods that will be utilized in conjunction with split-spoon sampling techniques to collect subsurface soil samples. Two split-spoon sampling techniques can be used. They are: 1) a wireline MOSS® system; and 2) surface drop hammer or wireline downhole drop hammer.

The wireline MOSS® system is a downhole soil sampler that is capable of recovering up to 5-foot long soil samples. The sampler is lowered down the hollow opening of the auger to the lead auger and "latched" into place. The bottom of the sampler is set in place flush with the cutter head and advances with the rotating auger. As the rotating auger is advanced downward, the soil or rock advances upward into the sampler. After the desired interval has been sampled, the sampler is lifted back to the surface via a wireline system. The sampler may be lined with stainless steel/or brass liners (sleeves). The sampler will be lined with stainless steel or brass liners to isolate and maintain integrity of the samples when chemical or physical testing is required. When detailed lithologic descriptions are desired and no physical or chemical testing will be required, a split-spoon "dry core" (no sleeves) sampler will be used.

The surface drop hammer system normally utilizes an 18-inch to 24-inch long split-spoon sampler to collect soil samples. The augers are advanced to predetermined depths and the center bit, attached to the drill rods, is pulled out of the hole. The sampler, lined with sleeves, is attached to small diameter, flush threaded pipe and lowered to the bottom of the hole. The 140-pound surface drop hammer mounted on the drilling rig at the surface is dropped repeatedly, and the sampler is driven approximately 18-inches into the ground. Blow counts are measured every 6-inches to determine the physical characteristics of the material encountered. Fifty blow counts is considered formation refusal. If 50 blows are counted for a 6-inch interval, the sampler is removed and the drill stem is advanced through the material. A 140-pound drive hammer mounted above the sampler, but inside the boring (downhole hammer) may also be used for driving the sampler into the ground.

Samplers can hold various diameter (2-inch, 2.5-inch, 3-inch) sleeves as well as different length (3-inch or 6-inch) sleeves. The outside casing of the sampler opens longitudinally to insert/remove the sleeves. After the sampler is removed from the hole, liners are separated, removed from the holder, and those selected for analysis are capped with Teflon® tape and covered with PVC caps. The capped liner is then labeled and placed in a Ziploc® bag for shipping to the laboratory to prevent cross-contamination. Samples collected for nonvolatile chemical analysis will either be left in the liner and capped, or removed from the liner and placed in an appropriate container with minimal disturbance. The container will be sealed with a Teflon®-lined cap. A small portion of the sample will always be collected in labelled sample bags and retained for visual inspection. The lithology of the sample will be recorded on a boring log.

The split-spoon sampler will be decontaminated by: 1) washing in a detergent and potable water solution using a brush; 2) rinsing with potable water; 3) rinsing with deionized reagent water; 4) rinsing with methanol; and 5) rinsing with cyclohexane. Clean liners will then be placed into the sampler in preparation for collecting the next sample. After completion of the boring, the sampler will be decontaminated by steam-cleaning.

The stainless steel liners will be prepared prior to use by the following four-step process:

- Washing in a solution of detergent and potable water;
- Rinsing with potable water;

- Rinsing with deionized water; and
- Baking at 106°C for a minimum of eight hours.

For sampling of unconsolidated and uncemented sands or gravel deposits, a split-spoon sampler equipped with a sample catcher is used to prevent sample loss. The catcher is placed in the lower portion of the sampler. As the sampler is pulled to the surface, the catcher prevents unconsolidated or uncemented material from falling out the bottom of the sampler. The catcher will be decontaminated in the same manner used to decontaminate the split-spoon sampler.

**Continuous Coring**

Continuous coring techniques are used to collect soil/or formations when using the direct circulation mud rotary, hollow-stem auger, or sonication drilling methods. Continuous coring using a split-spoon sampler will be used to recover 5-foot long soil cores. The sampler is lowered down the hollow opening of the drill stem and latched into place. The bottom of the sampler is set in place flush or slightly ahead of the bottom of the bit and advances as the drill stem moves downward. As the drill stem is advanced downward, soil and/or rock are forced up into the split-spoon sampler is retrieved using a wireline or rod system. The sampler may either be lined with 2.5-inch diameter sleeves (3- or 6-inch long stainless steel or brass), or a split-spoon "dry core" (no sleeves) sampler may be used. If a rod system is employed, the split-spoon sampler may also be pushed ahead of the drill stem to collect undisturbed soil samples. If the dry core sampler is used, samples to be submitted for analysis will be sleeved at the surface after sample selection.

Continuous coring using a solid core barrel will be used to recover from 5- to 20-foot-long cores for the sonication drilling method. The sampler is lowered down an open borehole or down the hollow opening of the outer casing and vibrated into the soil. As the core barrel is advanced downward, soil and/or rock are forced up into the core barrel. After the core barrel has been driven to the desired depth, the outer casing is vibrated down around the core barrel until the bottom of the outer casing is at the same depth as the bottom of the outer casing is at the same depth as the bottom of the core barrel. Cores are then placed into boxes, labeled to indicate boring, date, depth interval, and core, and stored in a core storage area. In unconsolidated formations (soils), a retainer is inserted into the shoe of the core barrel to prevent the loss of soil or rock as the barrel is pulled to the surface.

Samples collected by either split-spoon or continuous coring techniques will be logged by a Supervising Rig Geologist (SRG) using the logging protocol defined in this document.

### 5.2.5 Cuttings and Groundwater Disposal

A licensed waste transportation company will transport borehole cuttings, drilling fluids, and groundwater removed from the boreholes in accordance with the McClellan AFB Soil and Debris Management Plan (Radian, 1991). During the drilling operations, the cuttings will be monitored for organic vapors, and contained in steel, plastic-lined, 55-gallon drums or 20-cubic-yard capacity roll-off bins. Bentonite drilling fluids will be collected in tanks located on site. Groundwater produced during the drilling will be collected in leak-proof metal bins or tanks, transported by vacuum truck, and discharged to either the Industrial Wastewater Treatment Plant (IWTP) or the Groundwater Treatment Plant (GWTP) at McClellan AFB. The timing and transportation of groundwater produced in drilling activities will be coordinated with McClellan AFB EM.

Drill cuttings and cores will be monitored for organic vapors in the field by the contractor's personnel and documented on the drilling logs. The frequency of monitoring organic vapors depends upon the drilling method employed. Organic vapor screening will be performed on all core or other samples taken from a boring. In borings drilled by air rotary casing drive, dual tube percussion, or hollow-stem auger, screening readings will be taken at the boring whenever drill string joints are broken to add new lengths. Cuttings from hollow-stem auger, air rotary casing drive, dual percussion, or bucket auger methods will be screened at approximately every 5 feet. The cuttings will be preliminarily characterized by monitoring for organic vapors emissions with a photoionization detector (PID) or flame ionization detector (FID), and, if required, species-specific Draeger® detector tubes. Proper management and disposal of cuttings will be determined after subsequent sampling and analysis of cuttings conducted in accordance with the McClellan AFB Soil and Debris Management Plan.

Drilling mud will be collected in water-tight, large capacity tanks at the drill site. All cuttings, soil, debris, waste, or drilling mud generated during drilling activities will be characterized and managed in accordance with the McClellan AFB Soil and Debris Management Plan. Any variation in the handling and management of materials generated in drilling activities must be approved in writing by McClellan AFB EM.

### 5.2.6 Recordkeeping

The SRG will work under the direct supervision of a California Registered Geologist, although the California Registered Geologist may not be in the field continuously.

The SRG shall be responsible to ensure that all recordkeeping associated with the drilling activities is completed. Examples of forms to be used during drilling projects are illustrated in Figures 5-3 through 5-9. The following forms shall be completed:

- Log of Drilling Operations;
- Well Completion Log;
- Time and Materials Log;
- Daily Field Report including equipment maintenance;
- Well Development Log;
- Photoionization/Flame Ionization Detector Screening Data Sheet; and
- Direct Reading Indicator Tubes Field Data Sheet.

In addition to completing these forms, the SRG will be responsible for keeping a daily log of events and observations in a field notebook. Contractors may use different forms; however, equivalent information must be recorded.

The Log of Drilling Operations will include descriptions of all subsurface materials encountered while drilling. All subsurface materials shall be classified and logged in accordance with Unified Soil Classification (USC) System and Wentworth Scale. The lithology shall be recorded in the field log in the following order:

1) Predominant lithologic type with major modifiers (i.e., gravelly sand, silty sand, clayey silt, silty clay);

2) Grain size (based on the size scale adopted by Wentworth);

Monitoring Well #: _____

## LOG OF DRILLING OPERATIONS

Page ___ of ___

TOTAL DEPTH _____ START DATE _____ FINISH DATE _____
GEOLOGIST _____ APPROVED BY _____ R.G.# _____
DRILLING COMPANY _____ DRILLER _____
DRILLING METHOD _____ EQUIPMENT _____
DRILL BIT TYPE AND SIZE _____
BORING LOCATION (ST. ADDRESS OR DESCRIPTION) _____

| Depth Below Surface (ft) | Sample Interval/Type | PID/FID Reading B.G./D.H. | Blow Count | Lithologic Description<br>Color, Texture, Moisture, etc. | Drilling Notes |
|---|---|---|---|---|---|
| | | | | | |

**NOTES**
PID = Photoionization Detector
ppm = parts per million
B.G. = Background Reading
D.H. = Down Hole Reading

**Figure 5-3. Log of Drilling Operations**

FIELD LOG – WELL CONSTRUCTION SUMMARY

Well/Borehole No.: _____
Project Name: _____
Project No.: _____

Location: _____ Logged By: _____
Completion Date: _____ Development Method: _____
Elevation of Measuring Point: _____ Drilling Method: _____

Depth (Feet) | Well Schematic

Stick Up Height: _____ Ground Surface Elevation: _____
Stick Up Elevation: _____

Surface Completion Type: _____
Borehole Diameter: _____

RISER PIPE

Type/Materials: _____
Diameter: _____
Interval (Depths): _____

ANNULAR GROUT

Type: _____
Interval (Depths): _____
Approximate Quantity: _____

ANNULAR SEAL

Type: _____
Interval (Depths): _____
Approximate Quantity: _____

FILTER PACK

Type: _____
Gradation: _____
Source: _____
Interval (Depths): _____
Approximate Quantity: _____

SCREEN

Type: _____
Diameter: _____
Interval (Depths): _____
Slot Size: _____

WATER LEVEL

Date Measured: _____
After Development: _____ Yes _____ No
Static Water Depth: _____ Feet _____ Elevation

Total Well Depth: _____ Feet _____ Elevation
Total Borehole Depth: _____ Feet _____ Elevation

Well/Borehole No.: _____

**Figure 5-4. Well Completion Log**

TIME AND MATERIALS LOG

Page 1 of 2

McClellan AFB

Boring/Well No. _____

Well No. _____ Completion Date _____

Total Depth Drilled _____ Screen Interval _____

Supervising Rig Geologist _____

| DRILLING TIME: | | WELL COMPLETION TIME: | |
|---|---|---|---|
| Mobe | _____ | Sand Pack | _____ |
| Rig Setup | _____ | Bentonite Seal | _____ |
| Drilling | _____ | Grout | _____ |
| Standby | _____ | Wellhead | _____ |
| Rig Decon | _____ | Site Cleanup | _____ |
| Other | _____ | Other | _____ |
| Subtotal Drilling | _____ | Subtotal Completion | _____ |

TOTAL _____

NOTES: _____

_____

_____

_____

_____

Figure 5-5. Time and Materials Log

Page 2 of 2

TIME AND MATERIALS LOG
(Continued)

Boring/Well No. _____

| | UNIT | QUANTITY |
|---|---|---|
| Screen | | |
| Stainless Steel Casing | | |
| PVC Casing | | |
| Sand | | |
| Bentonite Pellets | | |
| Portland Cement | | |
| Bentonite Powder (Gel) | | |
| Security Cover | | |
| Utility Box | | |
| Lock | | |
| | | |
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NOTES: _____

**Figure 5-5. Time and Materials Log (continued)**

DAILY FIELD REPORT

McClellan AFB Boring/Well No. _____

Date _____ Reported by _____

S M T W T F S

Summary of Events and Observations:

Summary of Subcontractor Activities:

Total Time - Activity

Figure 5-6. Daily Field Report

WELL DEVELOPMENT LOG
McClellan AFB

Page 1 of 2

Well No. _____ Reported By: _____

Screen Type/Interval _____ / _____

Screen Diameter/Slot Size _____ / _____

Static Water Level Prior to Development _____

Development Started _____ Development Ended: _____

Quantity of Water Discharged During Development _____

Equipment/Tools Used _____

| Time | Development Method | Observations and Remarks |
|---|---|---|
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Figure 5-7. Well Development Log

WELL DEVELOPMENT LOG
(continued)



Meter Start _____ Pump Intake (ft.) _____

Meter End _____

Total Gallons Purged _____

| Time | Elapsed Time | Discharge Rate (gpm) | Depth to Water | Drawdown | Notes |
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Figure 5-7. Well Development Log
(continued)

PHOTO-IONIZATION DETECTOR
SCREENING DATA SHEET

AREA: _____

DATE: _____ TIME: _____

SCREENER'S INITIALS _____ INSTRUMENT I.D. _____

| | SPECIFIC LOCATION | SCREENING VALUE (ppmv) |
|---|---|---|
| 1. | Ambient (average background) | |
| 2. | | |
| 3. | | |
| 4. | | |
| 5. | | |
| 6. | | |
| 7. | | |
| 8. | | |
| 9. | | |
| 10. | | |
| 11. | | |
| 12. | | |

Comments:

Figure 5-8. Photolonization Detector Screening Data Sheet

DIRECT READING INDICATOR TUBES
FIELD DATA

DATE: COLLECTOR'S NAME:

CLIENT / LOCATION:

| SAMPLE LOCATION | TIME | TUBE TYPE/MFG. | # OF PUMPS | CONCENTRATION | COMMENTS |
|---|---|---|---|---|---|
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**Figure 5-9. Direct Reading Indicator Tubes Field Data Sheet**

3) Minor modifier(s) (i.e., some silt, trace clay, etc.);

4) Color (based on Munsell Soil Color Chart);

5) Relative moisture content (i.e., dry, damp, moist, wet, saturated); and

6) Other descriptive terminology as appropriate, such as, but not limited to:

   a) Relative density or consistency;
   b) Observed bedding;
   c) Visual evidence of contamination;
   d) Distinctive mineralogy (i.e., micaceous); and
   e) Sorting or grading.

In addition to continuously logging the encountered subsurface materials, pertinent information regarding drilling operations will also be recorded on the log form. Such entries may document drilling times, rig "down" time, problems with drilling methods, etc. Because many of the boreholes will be drilled to construct monitoring wells, the occurrence and quantity of groundwater encountered while drilling will be documented. Depending upon the goals of the investigation, lithologic samples may be collected at a minimum of every 5 feet and stored in labeled sample bags. Samples collected in this manner are intended for general lithologic information only and will be required to be representative of the drilled interval. Samples of materials that will be sent to laboratories for physical or chemical parameter measurements will be contained in appropriate glass containers or sleeves.

When collecting samples during mud-rotary drilling, uphole velocities of the drilling mud will be determined to ensure that samples are obtained from desired depths. One of two methods will be used to determine the uphole velocities. Real-time measurements will be made if possible. After each drill pipe connection, the drilling mud will be circulated until air that was entrained in the drill pipe from making the connection travels through the drill pipe, into the borehole, and back to the surface. The travel time will be recorded and used to determine when to collect the sample for a desired depth.

The second method that may be used is to determined uphole velocities based on borehole diameter, drill pipe diameter, and pump output. A nomogram to determine

uphole velocities using this information is presented in Groundwater and Wells, Fletcher G. Driscoll, Ph.D., p. 387, 1986.

The termination depth (TD) for a monitoring well shall be determined by the SRG after consultation and concurrence of the contractor's registered geologist or hydrogeologist, the FC, and the McClellan AFB EM Engineer. Identification of a favorable screen interval in the planned depth range of the well will be the primary factor for selecting TD. Favorable conditions for a screen interval selection include:

- Groundwater contained in a monitoring zone of interest for water level measurements or water quality samples;
- Sufficient permeability to provide discharge needed for water quality sampling or aquifer testing; and
- Adequate penetration into the saturated zone (wells to be screened in the uppermost groundwater zone will be drilled to a depth to allow 10 to 15 feet of screen below the water table and at least 5 feet above the water table).

Should there be any questions regarding selection of a screen interval, the SRG will confer with the FC or Project Director (PD) prior to instructing the driller to terminate a borehole. If possible, the FC should be present as each well approaches TD. A sample of the formation material present in the proposed screen interval shall be recovered and retained by the SRG.

### 5.2.7 Staff Responsibilities

Two geologists and two field technicians will be required during sonication drilling to assure that boreholes are logged consistently and accurately and that sample integrity is maintained. A supervising geologist and one technician are generally assigned for other drilling methods.

**Supervising Rig Geologist (SRG)**

- Supervises drilling, sampling, and well installation or borehole abandonment activities.
- Point of contact for all drilling and sampling activities between the contractor and the driller.
- On-site health and safety officer when the project Health and Safety Officer is no present.
- Screens soil samples with PID.
- Inspects samples for physical evidence of contamination.
- Determines percent recovery of the cored interval.
- Makes decisions regarding sample selection for analyses based on lithology, PID measurements, and visual inspection.
- Keeps daily log of operations in field notebook, complete daily field reports, time and materials log, and soil sample PD reading data sheet.

**Assistant Rig Geologist (ARG)**

- Describes soil cores.
- Records other pertinent information on drilling log form (e.g., PID readings, discoloration, odor, and waste observed).
- Records intervals from which samples were taken for analysis, including soil gas analyses.
- Prepares field notes and well logs for entry into geologic database.
- Records percent recovery of the core.

- Records methods, measurements, and materials used in borehole abandonment.

- Labels core boxes and places core into boxes.

- Assists SRG in making field decisions regarding drilling, sampling, etc.

**Field Technician #1**

- Cover sleeves with Teflon® tape and plastic caps.

- Completes Soil Sample Data Sheets.

- Places sample labels on sample containers that will be submitted for analyses.

- Assists SRG/ARG in performing downhole soil gas sampling.

- Completes Soil Gas Sample Data Sheets.

- Assists in handling and break-down core barrel following removal from the borehole.

- Assists in packaging of samples for laboratory shipment.

**Field Technician #2**

- Performs health and safety monitoring measurements in the immediate work zone and at the top of the drill pipe following each "core run" using a PID.

- Completes health and safety PID Screening and Directed Reading Indicator Tube data sheets.

- Extracts core from sleeves.

- Decontaminates stainless steel sleeves and sampling tools (e.g., core sampler).

- Decontaminates other miscellaneous sampling equipment.

- Assists in handling and break-down of core barrel following removal from the borehole.

- Assists in packaging of samples for laboratory shipment.

For drilling methods other than sonication, the geologist performs all SRG and ARG duties, and the technician performs all technician duties.

### 5.2.8 Borehole Abandonment

At the completion of each borehole that is not developed into a monitoring well, the borehole will be grouted to the surface by pumping a neat cement mixture of Type-I Portland cement consisting of 4 pounds of powdered nonbeneficiated bentonite, 8 gallons of water, and one 96-pound sack of cement. Bentonite will be thoroughly mixed with water producing a "lump free" mixture prior to adding cement. The grout will be mixed using a mechanical mixer. The final grout mixture should weigh approximately 13 to 14 pounds/ gallon. The density of the grout will be measured using a mud scale. In 20-foot borings, the entire drill string will be pulled from the borehole, and a tremie pipe with a maximum outside diameter of 2-inches will be lowered to the bottom of the boring. As the grout is pumped, the tremie will be positioned such that it is below the top of the grout in the boring. With the exception of borings drilled by sonication, all borings greater than 20 feet will be grouted through the tremie pipe placed inside the drill pipe. The bottom of the tremie pipe will always be below the calculated grout level to prevent bridging of grout within the drill stem. Where sonication is used, the drill stem or overdrive casing will be used as the "tremie." As the drill stem is pulled, it will be vibrated to ensure there is no bridging of grout inside the drill stem. The bottom of the drill pipe will always be positioned below the top of the grout seal to prevent possible "caving" from overlying formation. The volume of grout required to seal a borehole will be calculated using the following formula and recorded on the SRG's field log:

$V = (3.14) * (r)^2 * (L) * (7.48 \text{ gallons/ft}^3)$

V = Volume of borehole in gallons;
r = Inside radius of borehole in feet; and
L = Total depth of borehole to be grouted in feet.

## 5.3 Well Installation

Monitoring well installation procedures documentation, and construction materials are described below.

### 5.3.1 Well Installation and Completion

All groundwater monitoring wells and piezometers will be constructed in a similar manner, regardless of the drilling method used. The following materials will be used:

- Screen--Johnson Type 304 stainless steel, wire-wrapped, 10-foot or 20-foot lengths, 4-inch inside diameter (2-inch inside diameter for piezometers) 0.020- to 0.010-inch slot sizes. Stainless steel centralizers will be set at the top and bottom portions of the screen.

- Casing--Johnson Schedule 5, Type 304 stainless steel, flush-threaded, 10-foot or 20-foot lengths, 4.026-inch inside diameter at coupling. Stainless steel casing will be used between the screen and the highest anticipated groundwater level. Stainless steel casing centralizers will be set at the bottom and top of the screen interval at every 40-foot length of casing above the top of the screen interval.

- Casing--Johnson Schedule 40 PVC, flush-threaded, 10-foot lengths, 4.026-inch inside diameter will be used from the water table to the ground surface in wells less than 190 feet deep. Low carbon steel casing will be used in wells greater than 190 feet deep. For all piezometers, 2-inch inside diameter low carbon steel casing will be installed from the top of screen interval to the surface. Stainless steel casing centralizers will be set at every 40 feet from the preceding centralizer in all wells and piezometers.

- Sand Pack--Monterey sand, 8x16 mesh or Lonestar, Number 1C for 0.010-inch slotted screen and Lonestar, Number 3 for 0.020-inch slotted screen.

- Sand Bridge--Monterey sand, 30 mesh.

- Bentonite Seal--Pelletized, nonbeneficiated sodium bentonite or bentonite chips.

- Sanitary Seal--Neat mixture of Type I Portland cement with approximately 4 pounds of powdered nonbeneficiated bentonite and 8 gallons of water per 94-pound sack of cement.

- Security Casing--Eight-inch diameter by 5-foot long steel casing with locking lid.

- Waterproof Utility Box--Will be used to protect wellhead for "flush" completions only.

Sieve analyses are performed in order to select the appropriate filter pack and screen slot size for extraction well construction. The sieve analyses are performed by collecting a sample of the formation material and passing it through a series of sieves of progressively smaller size. The cumulative weight of particles retained by each sieve is then plotted as a percent of the total sample weight against grain size to select the appropriate well construction materials. A thorough discussion of the sieving procedure is presented in Groundwater and Wells (Driscoll, 1986). Although a sieve analysis of grain size distribution is desirable in construction of a groundwater production well; it is not practical or cost-effective to conduct sieve analyses during drilling to optimize the design of each monitoring well. Although it is anticipated that most monitoring wells to be constructed at McClellan AFB will function satisfactorily with 0.02-inch screen slots, 0.01-inch slot screens may be selected in the field when completions are required in finer-grained monitoring zones penetrated in areas or depths not previously investigated. Screen slot sizes of 0.01 to 0.02 inch will be available for construction to ensure a sediment-free, representative groundwater sample for monitoring. Slotted screen selection will be made in the field on the basis of samples collected during drilling.

The preferred slot width of 0.02 inches for factory-prepared perforated casing (screen) was selected on the basis of 1984 Stage 2-1 sieve analysis and the satisfactory performance record of monitoring wells with screens of that slot width. Sieve analyses were performed on samples from 29 reconnaissance borings completed on and adjacent to McClellan AFB in 1984. Analyses of the grain size distribution in those samples indicated that a slot of 0.02 inches would pass ten percent or less of the grains in the water-bearing zone in which the screens were installed. The monitoring wells with 0.02-inch slotted screens, which have been periodically purged and sampled, discharge water at adequate pumping rates without producing measurable amounts of sand or silt. Screens with a slot size of 0.01 inches may be used in wells constructed in finer grained deposits.

Prior to placement of the screen and casing, the borehole depth is verified with a stainless steel-weighted surveyors' tape. The surveyors' tape must be free of debris, grease, tape and any artificial chemical compound. The screen and casing are emplaced through the hollow portion of the augers (HSA rig) or into the mud-filled borehole (direct rotary rig) or inside the drive casing (air rotary casing drive rigs). The casing and screen is suspended in the boring to prevent bending of the casing under its own weight, until the boring is sufficiently filled to support the casing's weight. Stainless steel centralizers are positioned at the top and bottom of the screen and at 40-foot intervals on the well casing up to ground surface. Stainless steel casing is used to the highest anticipated water table elevation. The highest anticipated water-level elevation is estimated for each monitoring well from: 1) the depth of the saturated stratum first encountered during drilling; 2) the observed decrease in the regional water table elevation of approximately 1 to 2 feet per year; and 3) seasonal fluctuations in water levels of approximately 1 to 5 feet from March (highest measured annual levels) to September-November (lowest measured annual levels). Polyvinyl chloride casing is used above the stainless steel casing in wells screened at depths equal to or less than 190 feet. In wells completed at depths greater than 190 feet, low carbon steel casing is used in the vadose zone because of the greater weight bearing capability of the steel threads. Low carbon steel is used above the stainless steel screen up to ground surface for all piezometer wells. All screens and casing are flush-joint threaded.

The sand pack is then placed around the screen through a tremie pipe to a height ranging from 1 to 2 feet above the top of the screen. The well is then surged to settle the sand and the height of sand adjusted to be at least 1 (no more than 1 foot for piezometers) and not more than 2 feet above the top of the screen. The drill string (HSA rig) or drive casing (air rotary or air percussion rig) will slowly be withdrawn and recovered

as the sand pack is installed. The drill string or drive casing will not be withdrawn above the level of the sand pack during installation. As the sand pack is being installed, a stainless steel-weighted surveyors' tape is used to sound the top of the filter pack. A 1- to 3-foot sand bridge is then added through the tremie pipe.

After the filter pack and sand bridge have been placed to support the screen and casing, the well will be checked for proper alignment by passing a length of PVC pipe (measuring 3.75 x 10 feet) through the casing to the bottom of the well. In the event that the PVC pipe length "binds" or resists downward or upward movement in the casing, steps will be taken to align the casing. Any misaligned casing will be straightened or replaced. Casing alignment procedures must allow free movement of the PVC pipe length or the well must be abandoned and reconstructed. After determining that the casing is properly aligned, the drill rig hoist or sand line is used to keep upward tension on the casing while the bentonite seal and grout are applied. This prevents casing movement and minimizes the potential for the casing to bend or become misaligned.

After checking the casing alignment, a 3- to 5-foot bentonite seal is constructed using the tremie pipe. The bentonite seal consists of hydrated bentonite pellets; however, a bentonite "slurry" should be utilized when: 1) the screen interval is at a depth that will not allow the pellets to reach the top of the sand bridge before complete hydration has occurred; and 2) when bentonite pellets will not sufficiently screen thick sand intervals. The bentonite "slurry" consists of Monterey 1C sand, Benseal®, bentonite chips, and water. The "slurry will be thoroughly mixed in a ratio of: 1 bag of 1C sand/20 pounds of Benseal®/5 pounds of bentonite chips/sufficient water to produce a viscous "slurry." The "slurry" is added through the tremie pipe, and the tremie pipe is always no less then 1 foot from either the top of the sand bridge or bentonite slurry. The quantities of sand and bentonite introduced to the well are measured and compared to a calculated estimate of the materials required based on well/borehole dimensions.

After sounding the top of the sand bridge and bentonite seal, a grout or cement-bentonite sanitary seal is pumped into the annulus between the well casing and outer casing. The grout is pumped into the annulus from the tremie line located and initially placed 7 to 10 feet above the bentonite seal. Only nonbeneficiated bentonite is used in the cement-bentonite grout mixtures. The cement, bentonite, and water will be mixed in the specified ratios to obtain a smooth, thick, pumpable grout mixture that will provide an effective seal and prevent excessive grout loss to the formation. The drill stem or drive

casing is gradually pulled up and recovered as the level of grout rises in the borehole. No more than 20 feet of drill stem or drive casing should be removed in any one recovery.

Grout should be emplaced in the well in one continuous operation except when an interval of the formation absorbs 1.5 to 2 times the normal calculated volume of grout. Grouting procedures should then cease for the day to allow time for the grout to set up. Grouting should proceed the next working day using the method described above. Figure 5-10 shows generalized well construction diagram for wells in which the weight of screen and casing does not exceed the weight bearing strength of PVC threads.

The wellheads for monitoring wells will be completed aboveground or as flush completions as shown in Figures 5-11 and 5-12, respectively. Monitoring wells completed aboveground will have a 8-inch diameter locking steel casing installed around the well casing. Aboveground completions may be appropriate in locations at which wells have been constructed in topographically low or poorly drained areas and entry of runoff into the wellhead is possible. For aboveground completions, the protective steel casing is set in a 2-foot-square cement pedestal. Four 3-inch-diameter steel protective posts are positioned around those wells where such protection is considered necessary.

The casing for all flush-completed wells is set several inches below grade. Each wellhead is enclosed within a steel "valve" box equipped with a threaded lid. Each valve box contains a water-tight seal between the box and the lid that prevents surface water from entering the well. The valve boxes are set slightly above the surrounding ground surface, so that surface water flows away from the wellhead.

The well number is stamped on the lid of the steel protective casing. All wells are secured with identically keyed padlocks; keys are provided to McClellan AFB EM. The top north side of the well casing is permanently notched during well completion to indicate a reference point from which all future water level measurements will be made. The location of the notch will be noted on the well completion form. Water-level measurements and surveying will be made from the bottom of the notch.

**Surveying**

At the completion of drilling operations, a licensed land surveyor determines the vertical and horizontal position of the reference point. The elevation of each wellhead

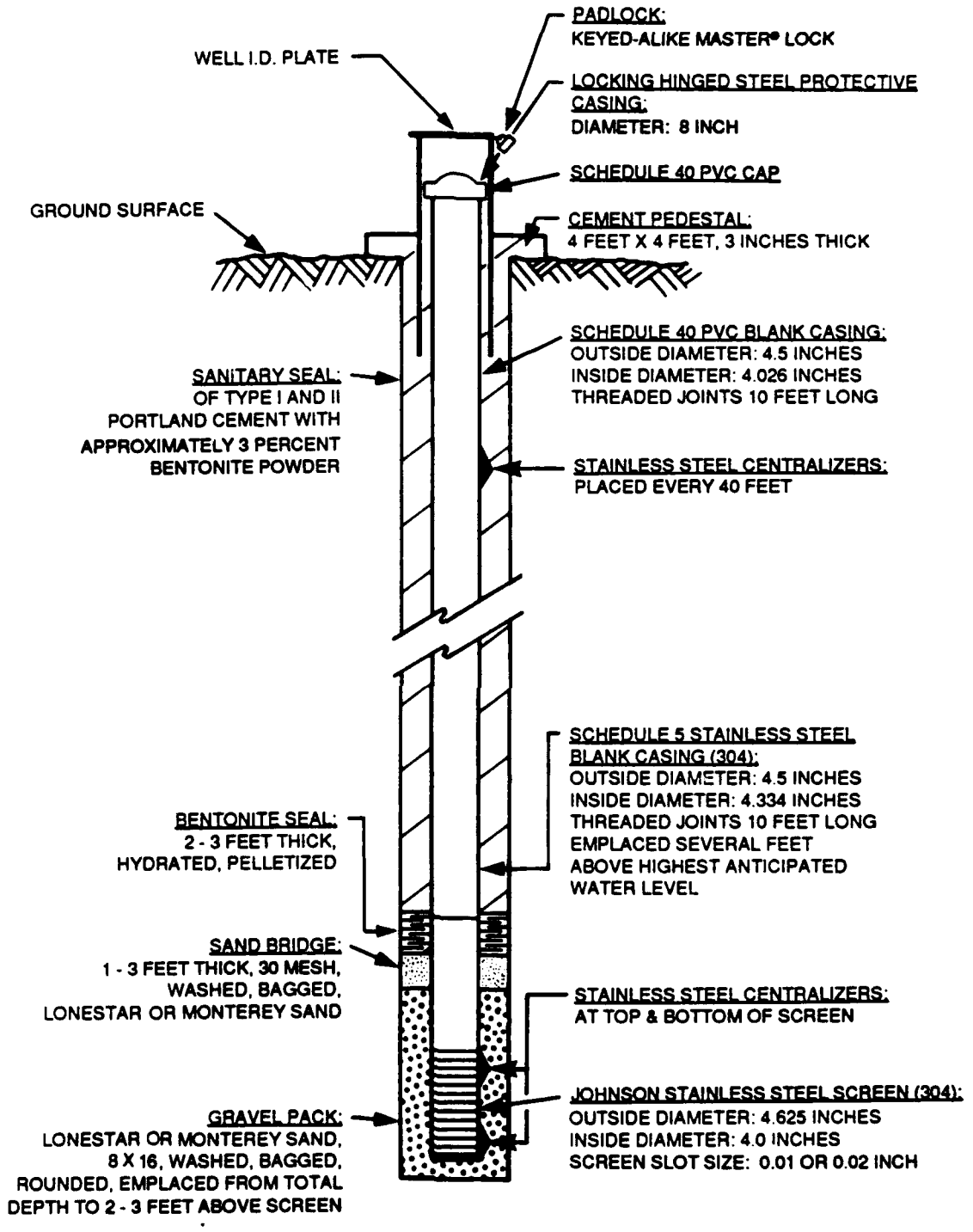


**Figure 5-10. Schematic Well Construction Diagram for Groundwater Monitoring Wells**

0991-078-15A

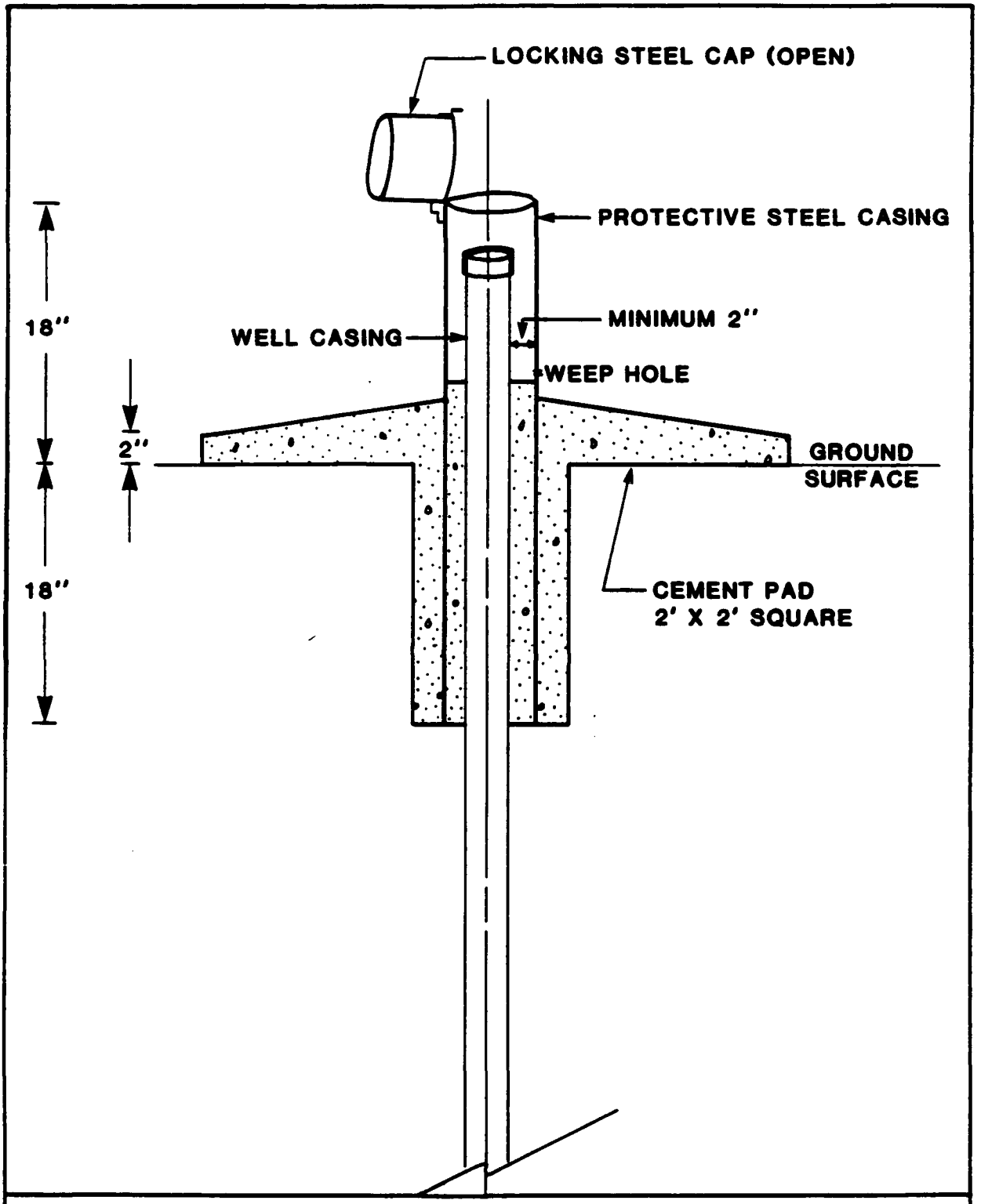


Figure 5-11. Aboveground Completion for Monitoring Wells

0491-078-16

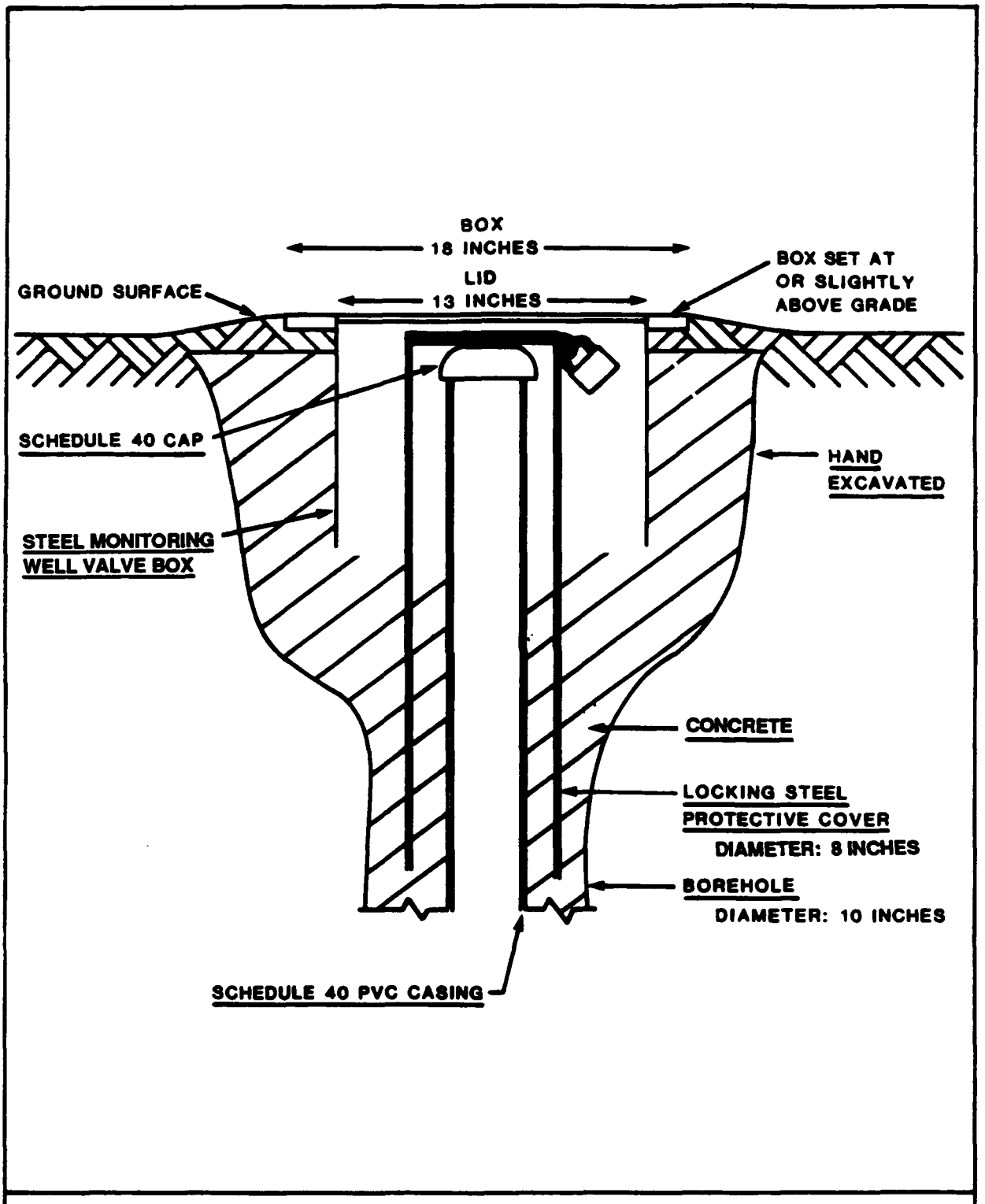


Figure 5-12. Surface Completion for Monitoring Wells

0991-078-17A

and the ground surface is determined to an accuracy of ±0.01 foot. Horizontal control is provided to ±1.0 foot.

### 5.3.2 Monitoring Well Development

All groundwater monitoring wells are developed to remove any fine-grained sediment from the screen and remove fluids introduced into the formations during drilling. Development procedures consist of bailing, surging, and pumping, and are accomplished under the supervision of the SRG or qualified field technician. During pumping of the well, drawdown data are obtained to determine specific capacity and estimate transmissivity and hydraulic conductivity.

Prior to development of each well, all downhole equipment (i.e., bailers, electric submersible pump, submersible pump cable, drift indicator, bailers, surge block, water level indicator, and development rig cable) is treated with Liqui-Nox® or equivalent laboratory-grade detergent and thoroughly steam cleaned to prevent cross-contamination between monitoring wells. The total well depth and depth to the water level are measured and recorded. The plumbness (straightness) of each monitoring well is determined using a Christensen well casing drift measurement device. Measurements are taken three times if the well is less than 150 feet below ground surface (BGS), or every 50 feet if the well is more than 150 feet BGS. Each well casing must meet a vertical declination criterion of 3 degrees.

Initially, each well is bailed with a sand-pump bailer to remove sediment that may have accumulated inside the screen as a result of formation disturbance during drilling or from surging after placement of the sand during well construction. Continued bailing is accomplished utilizing a minimum 10-foot long, 3-inch diameter, stainless steel, dart valve bailer with a capacity of 3.7 gallons. Bailing continues until the amount of sediment recovered from the well decreases to less than an estimated fluid ounce of sediment in one bailer load. The groundwater pH, conductivity, and temperature are measured and recorded during this process.

After bailing, each well is surged with a close-fitting, vented, surge block. As a surge block is moved up and down inside the well casing, it forces water back and forth across the screen openings to break down bridging of fine-grained material in the sand pack and adjacent formation. Surging is accomplished by lowering and rapidly raising the surge block and occurs for no less than 30 minutes. After surging for several minutes, the material

brought into the well is removed by additional bailing. The bailing and surging procedure is repeated until the amount of sediment entering the well decreases to a level deemed acceptable to the SRG.

Following bailing and surging, each well is pumped with an electric submersible pump. A 1/3 horsepower (HP) pump, a 2.5 HP pump, or a 5 HP pump is used for pumping. Selection of the pump horsepower depends upon well yield that is anticipated from the estimated permeability of deposits adjacent to the screened interval of the well. Wells are pumped at discharge rates ranging from approximately 2 to 20 gallons per minute. Actual pumping duration and discharge is controlled by the yield of each well and the amount of development required. All wells are pumped until pH, conductivity, and temperature parameters have stabilized and the well is free of sediment as determined by visual inspection by the SRG. Two successive readings of pH, conductivity, and temperature will not vary by more than $\pm 0.1$ pH unit, $\pm 0.5°C$, and $\pm 10$ micromhos, respectively, for the parameters to be considered stabilized.

## 5.4 Aquifer Test Methods

Properly planned and carefully conducted aquifer tests provide data used to determine hydraulic characteristics of an aquifer and a well. Pumping tests and slug tests are the two types of aquifer tests performed at McClellan AFB. A pumping test consists of pumping a well at a known rate and recording the drawdown in the pumping well and, if available, nearby observation wells at specific times. Recovery of the water levels in the pumped well and observation well are also recorded. A slug test is performed by manually removing or adding a known volume of water from a well and monitoring the water-level response. Slug tests are appropriate for wells yielding such low volumes that pumping with a submersible pump cannot be sustained.

Aquifer parameters may be determined by performing single-well and multiple-well aquifer tests. In single-well tests, water-level data from one well are used to calculate aquifer parameters. These tests are conducted in areas where there are no other wells screened in the same zone and located nearby. Single-well aquifer tests include both pumping tests and slug tests. Multiple-well aquifer tests are conducted by pumping one well, and monitoring water levels in observation wells screened in the same zone and in other observation wells screened in zones above and below the pumped zone. Multiple-well tests

test a larger portion of the aquifer than do single-well tests and thus yield more representative aquifer parameters.

### 5.4.1 Pumping Test Procedures

Prior to the actual pumping test, the following factors are addressed on a task-specific basis:

- Pumping test well configuration -- In multiple-well pumping tests, the well grouping distances and directions of the observation wells from the pumping well are specified based upon well construction and groundwater flow information.

- Pumping discharge point -- should be far enough away not to interfere with pumping test. Discharge of water will be coordinated with McClellan AFB EM. The water will either be collected into a vacuum truck and discharged at the Groundwater Treatment Plant or into the Industrial Wastewater Line. Selection of the discharge point will be based upon the presence of contaminants in the groundwater and the acceptance criteria established for each facility.

- Ambient water-level monitoring -- a period of ambient or background water-level collection performed to assess the magnitude and timing of water-level fluctuations due to natural and artificial phenomena (rainfall, barometric pressure).

- Pumping rate optimization testing -- preliminary experimentation with equipment and personnel to determine the maximum anticipated drawdown, optimum sustained pumping rate, and the best method to measure yield.

Proper planning and experimentation with the equipment and personnel during preliminary testing reduces the probability of errors occurring during the actual pumping test. In order to allow the aquifer to return to pretest conditions following preliminary testing, 24 to 72 hours are allowed for recovery, depending on type of aquifer.

The measurements taken during the actual pumping test are recorded on Aquifer Testing Field Sheets (Figure 5-13). These include static water levels at various intervals during the pumping period, and water levels after the pump is stopped. Since measuring the depth to water in the wells is an integral part of a pumping test, water levels are measured as accurately as possible many times during the course of the test. Pressure transducers and data loggers are used to measure and record water levels. Data are reviewed during the test to check if transducers are functioning and to determine that the pumping rate is adequate to provide data needed for analysis. Flow rate meters are used to determine discharge rate. The accuracy and precision of hydrologic measurements is discussed in Section 4.7. In addition, manual water-level meters are used to confirm automated water-level measurements. During the first one or two hours of the test, readings are taken at brief time intervals, since water levels usually drop rapidly immediately after pumping begins. As pumping continues, the time between readings will gradually be increased. The test is continued for a minimum of 4 hours; however, the duration of the pumping portion of the test must meet the objective of the test set forth in the specific SAP.

All equipment to be placed into the wells is decontaminated according to procedures detailed in Section 5.6.2. When placing the transducers in the wells, care must be taken to not exceed the maximum submergence depth of the transducer. The maximum submergence depth is determined by the pounds per square inch (PSI) rating. Proper placement is verified by using the data logger to take readings as the transducer is being lowered below the water surface and checked using a manual water-level meter. The transducer is set in the well at a depth that is lower than the maximum drawdown anticipated during pumping, but less than the maximum depth for the transducer PSI rating. This is particularly important in the pumped wells where large drawdowns are anticipated. In the observation wells, the transducer's sensor should be placed 5 feet below the greatest level of drawdown anticipated. Transducers are selected by dividing the maximum height of the water column above the transducer by 2.31 feet/psi. That calculated pressure should be within the range of the transducer. The transducer readings must be accurate to $\pm 2$ percent of the full scale pressure reading.

The transducers will be checked using manual measurements prior to any testing. After static conditions have been reached, the transducer will be moved up or down a measured distance and the resulting change in pressure noted. This change will be converted to a length and compared to the manually measured length to ensure that the pressure transducer is operating properly.

Page ____ of ____

## AQUIFER TEST DATA FIELD SHEET

Owner _____ Address _____ County _____ State _____

Date _____ Measured by _____

Well No. _____ Distance from pumping well _____ Type of test _____ Test No. ____

Measuring equipment _____

Serial numbers of equipment used _____

| TIME DATA | WATER LEVEL DATA | DISCHARGE DATA |
|---|---|---|
| Pump on: Date _____ Time _____ (t) | Static water level _____ | How Q measured _____ |
| Pump off: Date _____ Time _____ (t) | Measuring point _____ | Depth of pump/air line _____ |
| Duration of aquifer test: | Elevation of measuring point _____ | Previous pumping? Yes ____ No ____ |
| Pumping _____ Recovery _____ | | Duration _____ End _____ |

| Date | Clock Time | Time Since Pump On | WATER LEVELS | | | | Remarks |
|---|---|---|---|---|---|---|---|
| | | | Pumping Well | Observation Wells | | | |
| | | | | 1 | 2 | 3 | |
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**Figure 5-13. Aquifer Test Field Data Sheet**

Once the data logger has been programmed for measurement frequency and calibrated, the transducer reading is set to a reference level. All subsequent water-level readings from that transducer are referenced to this initial level. The transducer is allowed to equilibrate for several minutes with the temperature of the water in the well before the reference level is set. In addition, once the reference level is set, the transducer is not moved. The depth of the transducer in each well should be measured before and after the pumping test. The data logger is programmed for the appropriate test parameters, such as length of test, measurement frequencies, start time, number of input channels to read. Programmable features include the test number, inputs to be monitored, and the type of data to be collected on each input channel. The programmable features may vary from test to test; however, a logarithmic sampling rate is used for the early stages of all pumping tests.

Prior to starting the aquifer test, the data logger self-test routine is run to ensure that the data logger and all input channels are operating properly. If the self test indicates that the instrument is operating correctly, the aquifer test is started.

To begin each aquifer test, the water level in the pumped well and each observation well is measured and recorded. The start time is recorded and the pump and data logger are turned on. The data logger is started at the same time as the pump using a built-in delayed start feature. During the test, water levels are also measured with a manually operated water-level meter. These measurements provide a method of checking the accuracy of the transducer readings and provide backup data if the data logger fails. The pumping rate is checked periodically and recorded in the test log to ensure that the well is being pumped at the appropriate discharge rate.

The data logger can be programmed to monitor the recovery at the end of the pumping portion of the test. When this option is used, the logarithmic sampling interval is restarted without beginning a new test. The pump is then shut off; the new step started; and the recovery monitored for the same length of time as the pumping portion of the test or as specified in the SAP. At the completion of the recovery portion of the test, the pump is pulled from the pumping well and the transducers removed from the observation wells. Between each test, the pump will be decontaminated in accordance with procedures outlined in Section 5.6.2 of this QAPP.

After the test is completed, data are transferred electronically onto floppy disks from the logger and made available for analysis. These disks and hard copies of the data are stored in the project file.

### 5.4.2 Slug Test Procedures

The hydraulic parameters of an aquifer can be determined from the rate of rise or fall of the water level in a well after a certain volume or "slug" of water is suddenly added to or removed from a well. The rate at which the initial static water level is re-established is a function of the permeability of the formation near the well screen. This method of analysis, called a slug test, is particularly suited for wells in which aquifer tests cannot be performed with an electric submersible pump because the wells do not produce sufficient water to sustain continuous pumping. In slug tests performed at McClellan AFB, a closed-end pipe is used to displace the water column.

A pressure transducer is placed in the well to monitor water-level changes, which are recorded by a data logger. The transducer cable is enclosed in a PVC pipe to prevent the slug from damaging the cable or becoming entangled in it. The slug is constructed of PVC pipe. The ends of the pipe is fitted with flush-threaded PVC end caps. A nylon rope is attached to a bolt that has been threaded through the slug. All equipment to be placed into the wells is decontaminated in accordance with procedures detailed in Section 5.6.2.

Using the nylon rope, the slug is lowered into the water column as quickly as possible without causing the water in the well to be greatly disturbed. The slug is lowered below the water surface until maximum displacement of water has occurred. After the water level rises in response to the slug, the water level in the well is monitored by the data logger as it falls back to the initial static level. The slug is removed from the well after the pre-displacement water level has been reached. The water level will initially drop as the slug is being removed from the water, and then rise toward the initial static water level in the well. The rise in water level is recorded by the data logger. All field measurements are recorded on the Aquifer Test Data Field Sheet (Figure 5-13).

The data from each test are evaluated during the test and are downloaded to a computer after test completion. Plots of the rise or fall of the water level in the well versus time are generated for each test. The resulting curves are then analyzed using a suitable

method of slug test analysis. Various methods are available for confined and unconfined aquifer conditions.

## 5.5 Sample Storage and Preservation, and Handling

Sample containers are purchased precleaned and treated according to U.S. EPA specifications for the appropriate methods. Sampling containers that are reused (e.g., soil sleeves) are decontaminated between uses by the U.S. EPA-recommended procedures described in Section 5.2.4. Cleaned containers are stored separately at the contractor's staging area to prevent exposure to fuels, solvents, and other chemicals used to support RI activities. Amber glass bottles are routinely used where glass containers are specified in the sampling protocol. Table 5-1 lists sample storage and preservation requirements for each method and matrix.

All samples are labelled and field parameters and observations are documented in the field at the time of collection according to the procedures described in Section 6.1. Chain-of-custody and shipment and handling procedures described in Sections 6.2 and 6.3 are followed to ensure that samples can be tracked and sample possession and results documented through the analysis and reporting process. Information about sample shipment such as laboratory addresses, packing materials, return shipment of coolers, and arrangement for Saturday delivery, and transfer of field data to the database and reporting staff are provided in the field task instructions prior to the beginning of the sampling event.

## 5.6 Groundwater Sampling

Groundwater samples are collected from monitoring and extraction wells and analyzed for organic and inorganic constituents as required for each sampling and analysis task.

The objective of the groundwater sampling protocol is to obtain samples that are representative of the groundwater surrounding the well screen, so the analytical results reflect the composition of the groundwater as accurately as possible. In order to achieve this objective, all factors that may affect the physical and chemical integrity of the sample must be controlled before, during, and after sample collection. Sample preparation and preservation requirements are shown in Table 5-1. These factors are discussed in detail in the following subsections.

## TABLE 5-1. WATER AND SOIL SAMPLE STORAGE AND PRESERVATION REQUIREMENTS

| Reference Parameter | Method(s) | Holding Time | Container(s)[a] | Preservation | Storage Requirements |
|---|---|---|---|---|---|
| **ORGANIC COMPOUNDS:** | | | | | |
| Purgeable Halocarbons | SW8010 | 14 days | Three 40-mL glass vials with Teflon® seals (w)[b]; 4-oz widemouth glass bottle with Teflon® liner or stainless steel sleeve (s)[c] | None | 4°C |
| Volatile Total Petroleum Hydrocarbons/BTXE | SW8015 Modified | 14 days | 1-Liter glass bottles (w); stainless steel sleeves (s) | pH <2 with HCl (w) None (s) | 4°C |
| Extractable Total Petroleum Hydrocarbon | SW8015 Modified | 14 days until extraction, 40 days after extraction | 1-Liter glass bottles (w), stainless steel sleeves (s) | None | 4°C |
| Purgeable Aromatic Compounds | SW8020 | 14 days | Three 40-mL glass vials with Teflon® seals (w); 4-oz widemouth glass bottle with Teflon® liner or stainless steel sleeve (s) | pH <2 with HCl | 4°C |
| Organochlorine Pesticides and PCBs | SW8080 | 7 days until extraction, 40 days maximum | Three 40-mL glass vials with Teflon® seals (w); stainless steel sleeve (s) | None | 4°C |
| Organophosphorus Pesticides | SW8140 | 7 days until extraction, 40 days after extraction | 250 mL widemouth glass jar with Teflon® lined cap (s); 2.5 L amber glass jar (w) | None | 4°C |

(Continued)

TABLE 5-1. (Continued)

| Reference Parameter | Method(s) | Holding Time | Container(s)[a] | Preservation | Storage Requirements |
|---|---|---|---|---|---|
| **ORGANIC COMPOUNDS: (Continued)** | | | | | |
| Chlorinated Phenoxy Acid Herbicides | SW8150 | 7 days until extraction, 40 days maximum | 250 mL widemouth glass jar with Teflon® lined cap (s); 2.5 L amber glass jar (w) | None | 4°C |
| Purgeable Organic Compounds | SW8240 | 14 days | Three 40-mL glass vials with Teflon® seals (w); 4-oz widemouth glass bottle with Teflon® liner or stainless steel sleeve (s) | pH <2 with HCl (w) | 4°C |
| Base/Neutral and Acid Extractables | SW8270 | 7 days until extraction, 40 days maximum | Glass bottles with Teflon® seals (w); 8-oz glass bottle with Teflon® liner or stainless steel sleeve (s) | None | 4°C |
| Dioxins and Furans | SW8280 | 30 days for extraction, 45-day maximum | 1-Liter glass bottle with Teflon® seals (w and s) | 0.008% $Na_2S_2O_3$ if residual chlorine is present | 4°C |
| Polynuclear Aromatic Hydrocarbons | SW8310 | 14 days until extraction (s), 7 days until extraction (w), 40 days maximum | 250 mL widemouth glass jar with Teflon® lined cap (s); 1 L glass bottle (w) | None | 4°C |

(Continued)

TABLE 5-1. (Continued)

| Reference Parameter | Method(s) | Holding Time | Container(s)[a] | Preservation | Storage Requirements |
|---|---|---|---|---|---|
| **ORGANIC COMPOUNDS: (Continued)** | | | | | |
| Total Organic Carbon | SW9060 | 28 days | 120 mL widemouth glass jar with Teflon® lined cap (s); 125 mL polyethylene bottle (w) | None[b]<br>HCl or $H_2SO_4$ to pH <2[c] | 4°C |
| 1,2-Dibromoethane | E504 | 28 days | Two 40 mL glass vials with Teflon® seals (w) | None | 4°C |
| Nitrosamines | E607 | 7 days until extraction, 40 days after extraction | 250 mL widemouth glass jar with Teflon® lined cap (s); 2.5 L amber glass jar (w) | None<br>.008% $Na_2S_2O_3$[c] | 4°C<br>Dark |
| Volatile Organic Compounds in Air | Radian GC/ FID-PID-HECD Method (TO14) | 14 days | Stainless steel canister | None | None |
| Organic Lead Compounds | HML 338 | 14 days until extraction, 30 days after extraction | 250 mL glass jar with air-tight Teflon® lined lid (w); 500 mL glass bottle with air-tight lids (s) | None | 4°C |
| **INORGANIC COMPOUNDS/PARAMETERS:** | | | | | |
| Conductivity | E120.1/SW9050 | 28 days (off site) or field test | 250 mL widemouth glass jar or 250 mL polyethylene bottle (w) | None | 4°C |
| pH | E150.1 | Field Test | 500-mL polyethylene bottle or directly from bailer (w) | None | None |

(Continued)

TABLE 5-1. (Continued)

| Reference Parameter | Method(s) | Holding Time | Container(s)[a] | Preservation | Storage Requirements |
|---|---|---|---|---|---|
| **INORGANIC COMPOUNDS/PARAMETERS: (Continued)** | | | | | |
| Soil pH | SW9045 | Analyze as soon as possible | 250 mL glass jar (s) | None | 4°C |
| Total Dissolved Solids | E160.1 | 7 days | One 500-mL polyethylene bottle (w) | None | 4°C |
| Temperature | E170.1 | Analyze immediately | 2 L polyethylene bottle (w) | None | Analyze immediately |
| Anions | E300.0 | 28 days | 500 mL polyethylene bottle | None | N/A |
| Alkalinity | E310.1 | Field Test | 500-mL polyethylene bottle (w) | None | N/A |
| Nitrogen-Nitrite-Nitrate | E353.2 | 28 days | 250-mL widemouth glass jar (s); One 500-mL polyethylene bottle (w) | pH <2 with $H_2SO_4$ (w) | 4°C |
| PLM Asbestos[b] | E600/ M-4-82-020 | None | 250 mL widemouth jar or Ziploc® plastic bag (s) | None | None |
| Gross alpha, beta, gamma radioactivity | SW9310 E901.1 | 6 months | Glass or polyethylene bottles (w); stainless steel sleeves (s) | pH <2 with $HNO_3$ (w) | 4°C |
| ICP Scan for Metals | SW6010 | 6 months | One 500-mL polyethylene bottle (w); stainless steel sleeve (s) | pH <2 with $HNO_3$ (w) | 4°C (s) |

(Continued)

TABLE 5-1. (Continued)

| Reference Parameter | Method(s) | Holding Time | Container(s)[a] | Preservation | Storage Requirements |
|---|---|---|---|---|---|
| **INORGANIC COMPOUNDS/PARAMETERS:** (Continued) | | | | | |
| Arsenic | SW7060 | 6 months | Aliquot taken from 500-mL polyethylene bottle (w); stainless steel sleeve (s) | pH <2 with $HNO_3$ (w) | 4°C (s) |
| Chromium VI | SW7196 | 24 hours | 250 mL widemouth glass jar or stainless steel sleeve (s); Polyethylene or glass bottles (w) | None | 4°C |
| Lead | SW7421 | 6 months | Aliquot taken from 500-mL polyethylene bottle (w); stainless steel sleeve (s) | pH <2 with $HNO_3$ (w) | 4°C (s) |
| Mercury | SW7470 SW7471 | 28 days | Aliquot taken from 500-mL polyethylene bottle (w); stainless steel sleeve (s) | pH <2 with $HNO_3$ (w) | 4°C (s) |
| Selenium | SW7740 | 6 months | Aliquot taken from 500-mL polyethylene bottle (w); stainless steel sleeve (s) | pH <2 with $HNO_3$ (w) | 4°C (s) |
| Cyanide | SW9010/ SW9012 | 14 days | 1-Liter polyethylene or glass bottle (w); stainless steel sleeve (s) | pH >12 with NaOH (w) | 4°C |

(Continued)

TABLE 5-1. (Continued)

| Reference Parameter | Method(s) | Holding Time | Container(s)[a] | Preservation | Storage Requirements |
|---|---|---|---|---|---|
| **PHYSICAL PROPERTIES:** | | | | | |
| Grain Size Distribution | ASTM D422-63 | Analyze as soon as possible | Stainless steel sleeve | None | None |
| Specific Gravity | ASTM D854-83 | Analyze as soon as possible | Stainless steel sleeve | None | None |
| Permeability | ASTM D2434-68 | Analyze as soon as possible | Stainless steel sleeve | None | None |
| One Dimensional Consolidation | ASTM D2435-80 | Analyze as soon as possible | Stainless steel sleeve | None | None |
| Moisture Content | ASTM D2216-80 | Analyze as soon as possible | Stainless steel sleeve | None | 3°C to 30°C |

[a] All containers are pretreated and cleaned before being purchased by the laboratory.
[b] Sample container for water samples.
[c] Sample container for soil/solid samples.

E = U.S. Environmental Protection Agency.
HML = California Department of Toxic Substances Control Hazardous Materials Laboratory.
PLM = Polarized light microscopy.
SW = SW846 Third edition.
ASTM = American Society for Testing and Materials.

NOTE: Sample containers for gas phase samples, air canisters, sorbent tubes, or filters are stored at ambient temperatures and treated according to method specifications.

**Planning and Scheduling**

Prior to all field sampling events, a schedule is prepared to maximize the efficiency of the field activities. During preparation of the sampling schedule, several factors are considered, including the condition and accessibility of the well, the level of contamination, and sample collection techniques to be employed.

The wells not equipped with dedicated equipment are sampled in order of increasing contamination, with the least contaminated wells being sampled first. The level of contamination is not a determining factor when establishing sampling priority for wells containing dedicated equipment (equipment for purging and sampling left in place at a well) because the potential for contaminant carryover from well to well is diminished. If additional field measurements are planned for the sampling event, such as radioactivity monitoring, arrangements for use of the field instrument must be made.

Field QC is another integral aspect of schedule preparation. Wells selected for QC sample recovery (duplicates, equipment blanks, and ambient blanks) will be incorporated into the sampling schedule. Because the wells sampled for each event vary, QC sample assignment occurs after the list of wells to be sampled has been finalized. Considerations for assigning QC sampling include the required type and frequency (often a maximum number of blanks and duplicates are specified in the SOW), historical presence of contaminants, well location, and ensuring collection of field QC samples using all sample collection procedures. Additional information about the type, frequency, purpose, and use of these samples is presented in Sections 10.0 and 13.0.

**Materials and Supplies**

Materials and supplies required for sampling activities are organized prior to the start of sampling. A materials and supplies list is reviewed and the items checked before the sampling team leaves for the monitoring well sites. This procedure will encourage efficient use of time during the day. A list of typically required materials and supplies is presented in Table 5-2. In addition, safety equipment, including a first aid kit, always accompanies personnel in the field. A general safety equipment checklist is presented in Table 5-3.

**TABLE 5-2. SAMPLING EQUIPMENT CHECKLIST**

| |
|---|
| Specific instructions and maps for wells to be sampled |
| Quality assurance and field sampling procedures (the QAPP) |
| Trip blanks |
| pH meter |
| pH 4, 7, and 10 buffer solutions |
| Conductivity meter |
| Conductivity meter calibration solution |
| Thermometer |
| Radioactivity meter (if needed) |
| Chemical preservatives |
| Sample containers |
| Bailers (if sampling wells without dedicated pumps) |
| Bailer emptying device |
| Type II reagent-grade water |
| Laboratory-grade detergent (Alconox®) |
| Pesticide-grade methanol and cyclohexane |
| Water-level indicator |
| Calculator |
| Measuring tape (calibrated in hundredths of a foot) |
| Field notebook |
| Chain-of-custody forms |
| Plastic sheeting |
| Ballpoint pens |
| Sample bottle labels |
| Duct tape |
| Teflon® tape |
| Tool kit |
| Scissors |
| Wash tubs, buckets |
| Drinking water container |
| Towelettes |
| Garbage bags |
| Coolers |
| Ice |
| Protective mesh coverings for glass sample bottles |
| Foam containers for volatile organic analysis vials |

(Continued)

**TABLE 5-2. (Continued)**

| |
|---|
| Aluminum foil |
| Ziploc® bags |
| 10% nitric acid |
| Submersible pump and attachments (if sampling wells without dedicated system) |
| 10-foot lengths of plastic drop pipe with check valves and cam locks |
| Operating manuals |
| Control box |
| Compressor |
| Generator |
| Low submergence adaptor |
| Purge pump exhaust adaptor |
| 5-Gallon gasoline can -- spill proof |
| Air lines |

**TABLE 5-3. SAFETY EQUIPMENT CHECKLIST**

| |
|---|
| First aid kit |
| Fire extinguisher |
| Disposable coveralls |
| Safety glasses |
| Hard hats |
| Steel-toed rubber boots |
| Disposable polyvinyl chloride gloves |
| Butyl gloves |
| Draeger® tubes and pumps |
| Respirator and cartridges |
| Respirator cleaner |
| Heat monitors |
| Personal organic vapor badges |
| Safety forms |
| Copy of Health and Safety Plan |
| Organic vapor analyzer meter |
| Portable eyewash stations |
| HNu® Systems photoionizer |

NOTE: Additional equipment may be mandated by future revisions of the Health and Safety Plan.

**Sample Container and Trip Blank Preparation**

Prior to leaving for the monitoring well sites, prepare the trip blank samples as required. The purpose and preparation of trip blank samples are discussed in Section 10.0. When possible, prepare the sample containers with preservative at this time. Such preparation will save time and promote organization in the field.

**Recordkeeping**

Organized and accurate written records contribute to the reliability and comparability of field data. Field observations and data collected while sampling a well are entered on well-specific sampling data sheets contained in a bound logbook. Well maintenance check logs, including well security observations and visual surveys are maintained in the Groundwater Sampling and Analysis Plan (GSAP) project files for reference. In addition, a bound master log is kept to record sample tracking and analysis information, as discussed in Section 6.1. Samples also may be logged into an electronic master log system for producing chain-of-custody documentation and direct transfer of data to the project database.

### 5.6.1 Groundwater Sampling Equipment

One of three types of sampling equipment or system is used to collect groundwater samples from monitoring wells: a dedicated sampling system; submersible pump and/or bailer; or a truck-mounted sampling system that uses a discrete interval sampler (DIS) that was designed to sample deep wells (greater than approximately 250 feet). The dedicated sampling systems are placed permanently in individual wells, minimizing the potential to cross-contaminate other wells. The submersible pump and bailer or DIS system are used to sample wells without dedicated sampling equipment. For the pump/bailer system, the pump is used to purge the well, and a Teflon® bailer is used to collect groundwater samples; in some shallow wells, the bailer may be used to both purge and sample the well. The DIS system consists of a specially designed packer/bailer and purge pump system that is used to sample deep wells. The purge pump unit on the DIS system may also be used to purge shallow wells prior to sampling with a bailer. Standard operating procedures are followed for each type of sampling system to ensure that representative and comparable groundwater samples will be collected. A list of groundwater sampling equipment and components is shown in Table 5-4 and described in the following subsections.

**TABLE 5-4. LIST OF GROUNDWATER SAMPLING EQUIPMENT AND COMPONENTS**

| Equipment/Component | Model/Part Number | Manufacturer |
|---|---|---|
| Bladder pump | T1200 | QED Environmental Systems<br>P.O. Box 3726<br>Ann Arbor, MI 48106<br>Telephone: 313/995-2547 |
| Bladder pump screen | 34873EP | |
| Purge pump | HR4700 | |
| Driver/controller unit | 3111HRM | NOTE: Includes components manufactured by Briggs and Stratton Corporation and Pneumotive Air Power Products (see below) |
| Air compressor | Model No.: SGH-E101<br>Part No.: 160939 | Pneumotive Air Power Prod.<br>4601 Central Avenue<br>Monroe, LA 71203<br>Telephone: 318/387-4280 |
| Gasoline engine | 82232 | Briggs and Stratton Corp.<br>Milwaukee, WI 53201<br>Telephone: 414/259-5333 |
| Well cap -- 2-inch for bladder pump only | 2120A | |
| Well cap -- 4-inch for bladder pump only | 2120C | |
| Well cap -- 4-inch for bladder and purge pumps | 2150C | |
| Well cap -- 6-inch for bladder and purge pumps | 2150E | |
| Well cap -- 8-inch for bladder and purge pumps | 2150G | |
| Tubing -- Teflon®-lined polyethylene (1/2-inch O.D. and 1/4-inch O.D.) | PT5100 | |
| Tubing -- polyethylene (7/8-inch O.D. and 1/2-inch O.D.) | P5500 | |
| Tubing -- polyethylene (7/8-inch O.D. and 1/2-inch O.D.) | P5500 | |

(Continued)

TABLE 5-4. (Continued)

| Equipment/Component | Model/Part Number | Manufacturer |
|---|---|---|
| Teflon®-coated stainless steel cable | 8330 | |
| Teflon® elbow union (for bladder pump) | 34469 | |
| Discharge elbow (for purge pump) | 34536 | |
| Exhaust adapter | HR3500 | |
| Exhaust adapter hose assembly | 34664 | |
| Low submergence adapter | 3017 | |
| Water-level indicator | 6000 (or equivalent) | |
| Teflon® bailer | | |
| pH/Conductivity meters | EP-10/pH (or equivalent) | Myron L Company 6231C Yarrow Drive Carlsbad, CA 92008-4893 Telephone: 619/438-2021 |
| Thermometers | 15-000A (or equivalent) | Fisher Scientific 2170 Martin Avenue Santa Clara, CA 95050 Telephone: 408/727-0660 |
| Generators | EM4000<br>7600E | Honda<br>Wacker |
| Air Compressor | HP1500 (125 PSI) | Thomas |
| Electric submersible pumps | 1/2 HP Serial #M9J0494 and MX01 | |

### 5.6.1.1 Dedicated Sampling System

Well Wizard® groundwater sampling equipment, manufactured by Q.E.D. Environmental Systems, Inc., is used to collect groundwater samples from wells that are sampled on a regular basis (i.e., quarterly). The Well Wizard® equipment is designed to be "dedicated" or left in place in a monitoring well. This eliminates the need to use a submersible pump and sample collection equipment for many wells and decreases the probability of cross-contaminating groundwater samples and monitoring wells. The well Wizard® system involves dedicated equipment (permanently installed in the well) and portable components needed to operate. The dedicated equipment consists of a purge pump and bladder pump, or in shallower wells only, a bladder pump. The portable components required to operate the Well Wizard® system include the automatic controller and the oil-less air compressor driver.

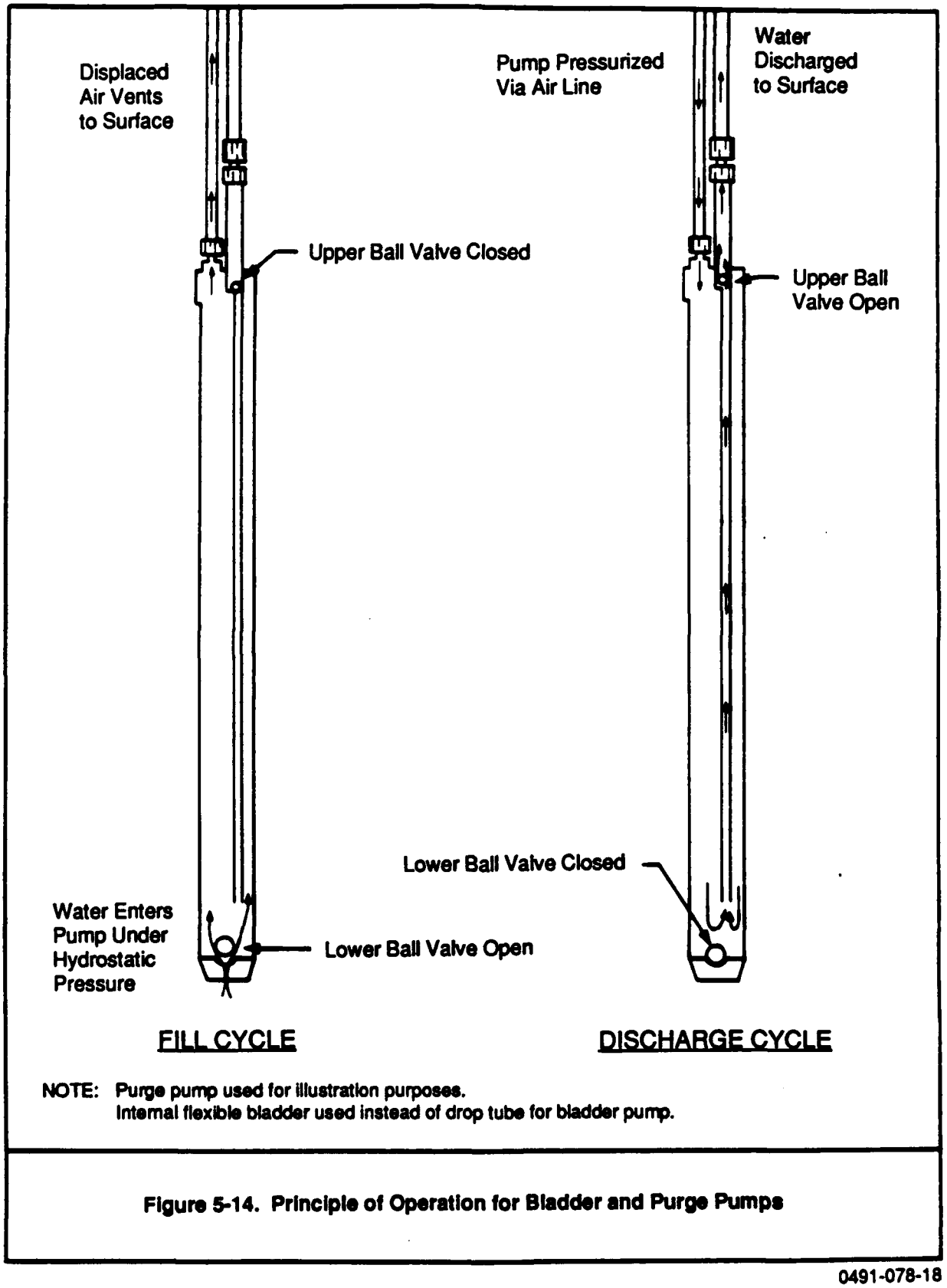
Both the Well Wizard® bladder and purge pumps use the positive gas displacement method to move water from the pump intake to the wellhead. Figure 5-14 illustrates the principle of operation for the gas displacement pumping method. The steps taken in operating the dedicated pump system are detailed in the Operations Manual, which is taken into the field when dedicated system wells are to be sampled.

#### Bladder Pump

A positive gas displacement bladder pump is used to recover groundwater samples. The bladder pump consists of a stainless steel body with an internal flexible Teflon® bladder. The bladder isolates the groundwater from the drive gas that would otherwise promote volatilization of any volatile organic compounds (VOCs) that may be present in the water sample. Each bladder pump is equipped with a stainless steel screen that covers the pump intake. The bladder pumps are installed within the screened intervals of the monitoring well, and are suspended from the well caps by the air line and discha.ge tubing.

#### Purge Pump

Groundwater sampling protocol requires that each monitoring well be purged to ensure that the sample consists of fresh formation water and not stagnant water that has been standing in the casing. Because the purging process often requires the removal of large quantities of water from a well and the bladder pump discharge rate is relatively low, a




NOTE: Purge pump used for illustration purposes.
Internal flexible bladder used instead of drop tube for bladder pump.

**Figure 5-14. Principle of Operation for Bladder and Purge Pumps**

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higher capacity purge pump is used for this purpose. Wells that have more than about 20 gallons of water stored in the casing will be equipped with a positive gas displacement purge pump in addition to the bladder pump discussed above. This will typically include the deep and/or large diameter wells that contain the largest volumes of water.

The purge pump is similar to the bladder pump operationally, but differs in that there is no bladder to prevent contact between the sample and drive gas. The purge pump is constructed of stainless steel and Teflon®. It is generally installed directly above the bladder pump or at a maximum submergence of about 50 feet, whichever depth is less. Like the bladder pump, the purge pump is suspended in the well by the air line and discharge tubing. However, a Teflon®-coated stainless steel cable is used as a safety line.

**Tubing and Well Cap**

The bladder and purge pumps each require both a discharge tube for delivery of the sample or purge water and an air tube for pressurizing and venting the pumps. The air line is alternately pressurized and vented at the wellhead by the oil-less compressor to allow the pump to discharge and fill, respectively. The sample discharge line conveys water to the wellhead for well purging or sample recovery. Polyethylene tubing is used for both pumps. Teflon®-lined polyethylene discharge tubing is used for the bladder pump to reduce the effects that leaching, adsorption, or desorption associated with less inert tubing materials might have on the groundwater samples.

Each well is equipped with a PVC cap. Depending on whether one or two pumps are used in a well, the cap is fitted with either two or four fittings for connection of the discharge tubing and air lines to the controller and driver. Figure 5-15 presents a graphic illustration of the two types of well caps.

A short length of Teflon®-lined polyethylene tubing is dedicated to each well to direct the bladder pump discharge water from the wellhead to the appropriate containers. This tubing is stored suspended in the well casing, and will be rinsed with Type II Reagent Grade water before each sampling activity. An access port is also provided in the cap to allow the use of a water-level probe without removing the cap.

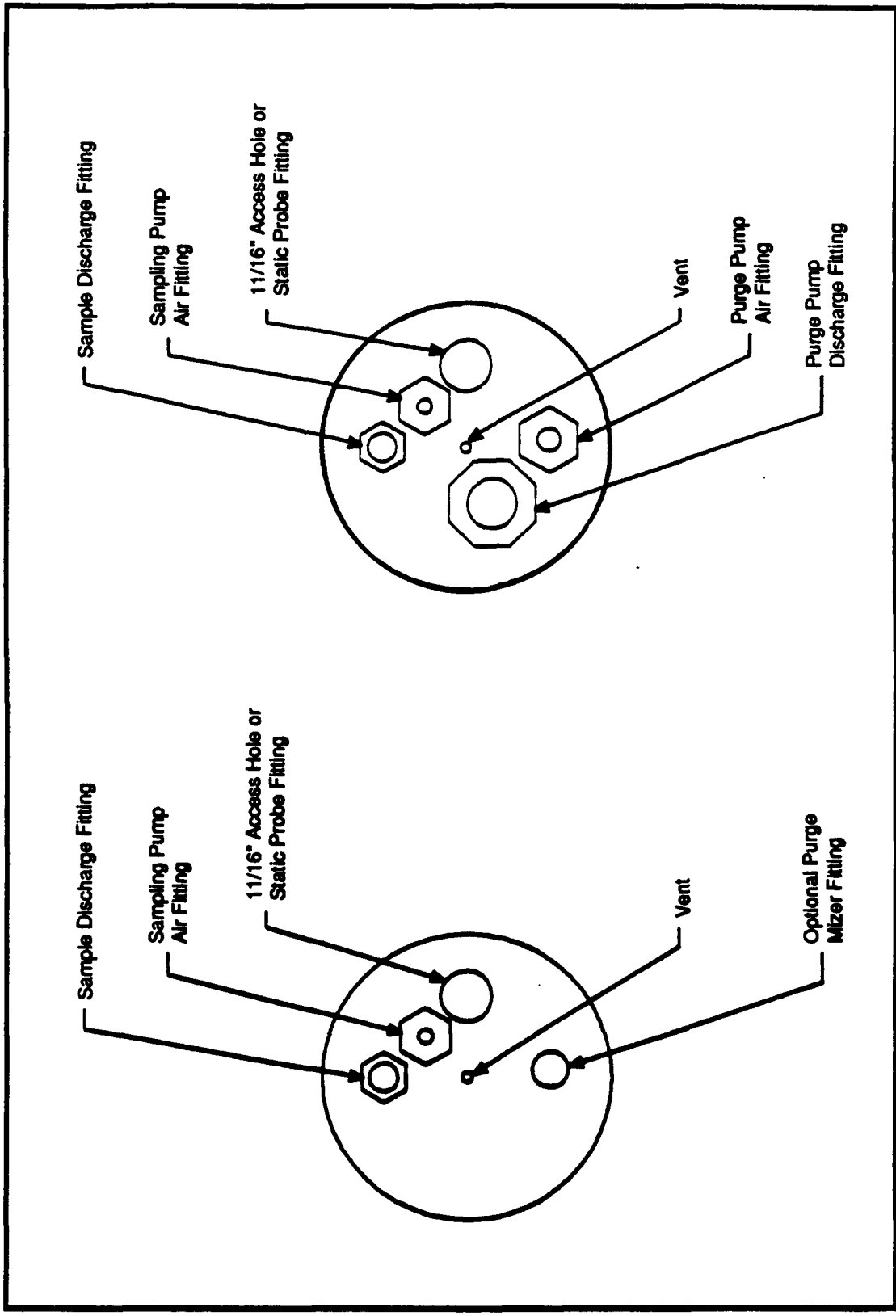


Figure 5-15. Well Cap Illustration

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### Driver Assembly

The driver assembly consists of a portable, three to five horsepower, air-cooled, gasoline engine that powers an oil-less, 100 psi, air compressor. The compressor supplies the drive air required to pressurize the bladder and purge pumps, forcing groundwater through the tubing to the surface. The pressure/vent cycle is regulated by the automatic controller device (discussed below).

### Automatic Controller

The automatic controller is a portable system component that regulates the air flow from the compressed air source (driver assembly) to the bladder and purge pumps. The automatic controller alternately vents and pressurizes the air line, allowing the pump to fill and subsequently discharge water, respectively. Figure 5-16 illustrates the control panel for the automatic controller for the Well Wizard®.

### Low Submergence Adaptor

The low submergence adaptor is a portable system component used to increase the discharge rate of those pumps installed with a minimum of submergence below the water level. Water will fill the pump more slowly in low submergence situations because the rate at which the pump is dependent on the hydrostatic head in the well. The low submergence adaptor, when connected to the automatic controller, induces a slight negative pressure in the air line during the fill cycle, thereby decreasing the time required to fill the pump and increasing the overall discharge rate.

### Purge Pump Exhaust Adaptor

When the bladder pump fills, exhaust air is forced through the air line and vented at the automatic controller. During operation of the purge pump, an exhaust adapter is used to vent the larger diameter purge pump air line at the wellhead to vent the exhaust air prior to the controller. Like the low submergence adapter, the exhaust adapter decreases the time required for the purge pump to fill and increases the overall discharge rate.

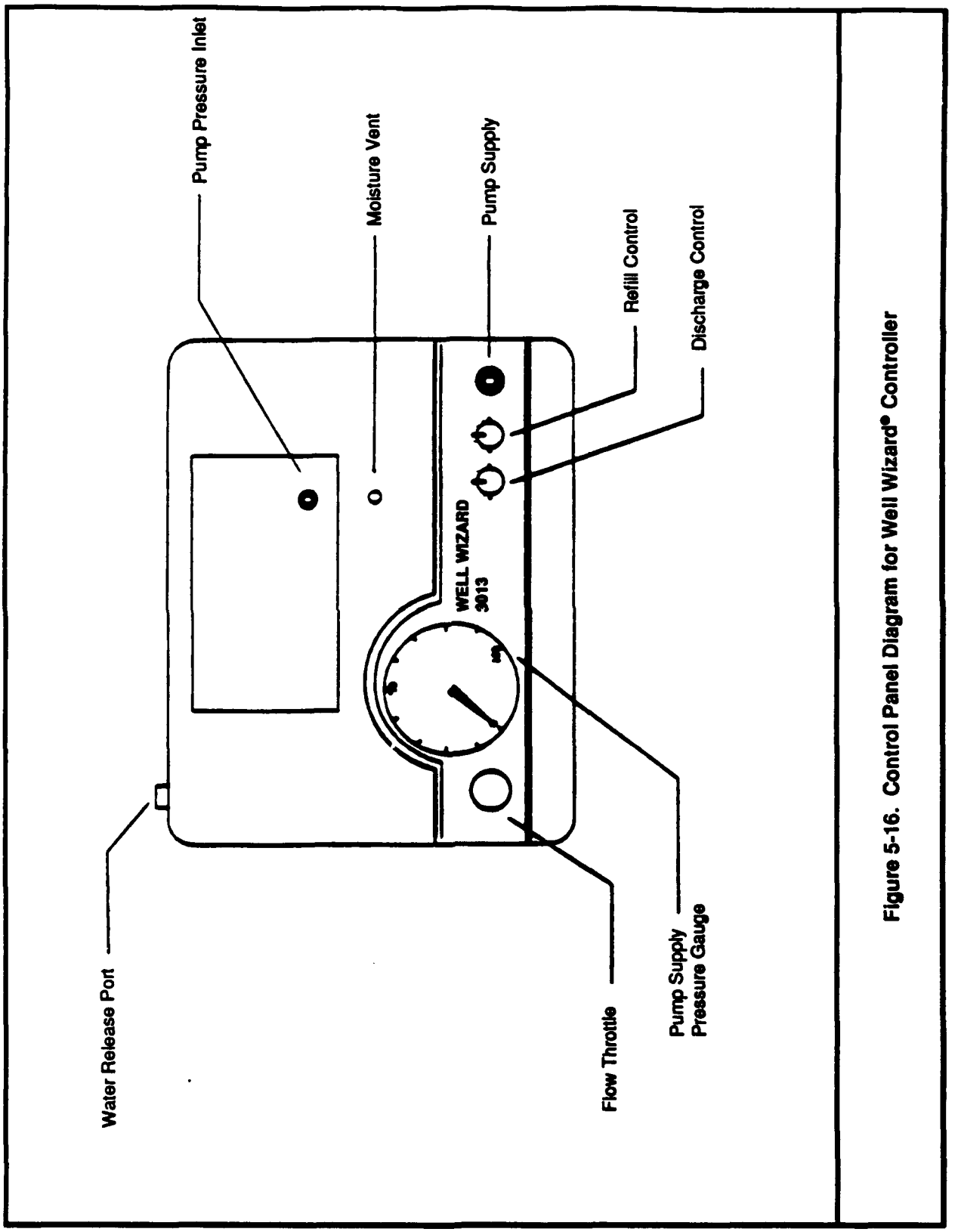


Figure 5-16. Control Panel Diagram for Well Wizard® Controller

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### 5.6.1.2 Bailer and Submersible Pump

If monitoring wells are not equipped with a dedicated sampling system, an electric submersible pump or a Teflon® bailer may be used to purge the well. Typically, the submersible pump is used to purge the deeper wells with a large water column. When purging is complete and the pump has been removed, the wells are sampled using a bailer with a braided stainless steel or disposable monofilament line. Shallow wells with a small water column, or wells that produce insufficient water to allow using a pump, are purged using a bailer. Before either a submersible pump or bailer is used to purge and sample a well, it will be decontaminated using the procedures described in Section 5.6.2. A petcock, or flow control valve attachment, is used to collect samples from the bailer after purging has been completed. This type of attachment allows collecting samples from the bailer with minimal disturbance or agitation that could result in loss of volatile organic compounds.

### 5.6.1.3 Discrete Interval Sampler System

The DIS system was designed by Radian for use in collecting representative groundwater samples from monitoring wells (greater than 250 feet deep) at McClellan AFB. The system is equipped with several features which significantly reduce the time required to purge and sample deeper wells, thereby optimizing the overall efficiency of the sampling process. The two primary features of the vehicle are a remote-operated sampler and packer unit, and a 2-inch, stainless steel, submersible purge pump.

The sample and packer unit is designed to be lowered into the well and to seal the casing, which isolates the water column just above the screen and within the screened interval. This design achieves three benefits in sample collection:

- The samples are collected from the screened interval;
- The sample chamber undergoes continuous conditioning during the purge process; and
- The volume of purge water is greatly reduced because only the lower portion of the well, the screened interval and a 5-foot length of casing above the interval, must be purged.

The sampler and packer unit consists of a sample collection chamber and an inflatable well casing packer. Materials used in the construction of these components are stainless steel, Teflon®, and Viton rubber. The sampler consists of a stainless steel collection chamber through which water flow is controlled by a set of Teflon® and stainless steel control valves. A remote actuated solenoid operates the control valves. The inflatable packer, which attaches to the bottom of the sample chamber, is manufactured of stainless steel and Viton rubber. The packer is used to isolate the fresh formation water entering the screened interval from the stagnant water in the remaining water column.

The submersible purge pump is constructed of stainless steel and Teflon®. The pump is controlled at the surface by a rectifier, a voltage converter that allows control of the pumping rate from as much as 6 gallons a minute to as little as 40 milliliters a minute. Because of its small size and light weight, the pump can be lowered to the water table using a reinforced hose.

### 5.6.2 Equipment Decontamination Procedures

In order to avoid cross contamination, all nondedicated sampling equipment including bailers, pumps, hoses, and water-level indicators are decontaminated prior to use and between samples using the following procedures. Equipment is washed using a laboratory-grade detergent (Alconox®) followed by rinses with drinking-quality water, ASTM Type II reagent water, pesticide-grade methanol, and pesticide-grade cyclohexane or hexane if the ambient temperature is below 45°F. Hexane is used when temperatures are below 45°F because cyclohexane freezes at that temperature and does not evaporate; hexane use is limited to these conditions because of health and safety concerns for the field samplers. Sufficient time must be allowed for the solvent to evaporate and the equipment to dry before use. A final rinse with ASTM Type II water is performed after solvents have evaporated to rinse off any residue. Equipment used to collect water samples for organics analysis is transported in aluminum trays (also decontaminated) and is not allowed to come in contact with plastic (e.g., plastic storage bags). All pH and conductivity meter probes and thermometers are thoroughly rinsed with ASTM Type II reagent water between uses.

Before dedicated pumps are installed, or the submersible pumps or DIS bailer/ packer assembly are used, they are cleaned using a steam cleaner or high pressure hot water system. The intake check valve areas receive extra cleaning efforts, by forcing steam through the pumps to clean the interior. The exterior of the pumps and discharge hose are

also steam cleaned. All dedicated groundwater monitoring systems must pass a rigorous cleaning procedure and must be certified clean by the manufacturer prior to installation. Since these sampling devices are permanently fixed in the wells, the possibility of cross-well contamination is eliminated. Dedicated sample tubing and pumps removed from wells for maintenance purposes are thoroughly steam cleaned prior to being reinstalled. Clean, disposable gloves are worn during and after decontamination, so equipment is not contaminated.

### 5.6.3 Field Procedures

Groundwater sampling field procedures include field instrument calibration, water-level measurements, well purging, and sample collection. Where possible, activities required to sample the monitoring wells are completed by a field team consisting of at least two people. This will expedite the sample collection process and promote safety at the well site. Field instrument calibration, water-level measurements, well purging, and sample collection are discussed below. In the case of low-yielding wells, purging and sample collection techniques are slightly different and are detailed within the well purging section. The procedures for sample collection using dedicated systems and the pump/bailer procedures follow those outlined in U.S. EPA's Compendium of Superfund Field Operations (1987); the DIS system design represents new technology, and the sampling procedures for this system differ from the existing procedures. Therefore, the operation and sample collection procedures for this system are described separately in Section 5.6.4.

#### Field Instrument Calibration

Conductivity meters, pH meters, thermometers, and organic vapor analyzers are used during groundwater sampling. The conductivity meters and pH meters are calibrated on a daily basis prior to sample collection and also at the end of each field day. The conductivity meter is calibrated according to manufacturers instructions with two standards bracketing the expected conductivity range. The pH meter is calibrated and adjusted with two buffer solutions which bracket the expected sample pH. A single point calibration check using the pH 7 buffer is performed at each well. If the meter drift is 0.1 pH units or greater, the two point calibration is repeated. Any instrument drift at the end of the day is also noted in the calibration field log. Field thermometers are calibrated monthly. Calibration procedures for the organic vapor analyzers are detailed in Section 7.2 of this QAPP.

**Water-Level Measurements**

The water-levels in all monitoring wells are measured within the first one or two days of sampling for each sampling task. This allows point-in-time assessment of the potentiometric surface during every sampling effort.

The depth to water is measured from a reference point at the wellhead (see Section 5.3) of each monitoring well prior to purging or sampling. In wells equipped with dedicated pumps, the water level is measured from the top of the well cap. The elevation of this surface will be calculated relative to the surveyed reference point and entered into the McClellan AFB database. In wells without dedicated pumps, the depth to water is measured from the surveyed reference point, a notch on the top north side of the PVC well casing, as discussed in Section 5.3. The reference point used for the depth-to-water measurement is specified on the sample-recovery log.

Water-level measurements are reported to the nearest 0.01 foot. The measurements are made with QED® electronic water-level meters. This measuring device has a flat polypropylene tape with stainless steel wire in the tape to prevent it from stretching. The visual or audio signal is activated when water is encountered. The depth to water is measured repeatedly until two consecutive measurements are consistent within 0.01 foot. In new wells or wells where a hydrocarbon layer is suspected, an Oil Recovery Systems® probe is used. This probe is capable of detecting hydrocarbon layers with a thickness of 1/32 inch (0.003 feet).

The following procedures will be followed when performing water-level measurements:

- Wear protective clothing and equipment as required by the Health and Safety Plan;
- Unlock the well cover;
- Don new clean disposable polyvinyl chloride gloves as an outside layer to prevent cross contamination;

- Decontaminate the water-level indicator and last 5 feet of tape using the previously mentioned procedures--since only the probe comes in contact with the well water, this is considered sufficient decontamination;

- Lower the electronic probe into the well until water is encountered and note the depth on the calibrated tape relative to the surveyed reference point;

- Repeat the water-level measurement until two consecutive measurements agree within 0.01 foot;

- Record the depth to water on the sample recovery log form; and

- Decontaminate the probe and the first 5 feet of the tape.

To promote organization and consistency in field data collection, one field team member measures the depth to water and calculate the required purge volume for the well, while the other organizes and sets up the sampling equipment, and prepares the field logs.

**Well Purging**

Well purging is an integral step in recovering samples that are representative of in situ groundwater chemistry. Each monitoring well is purged immediately prior to sample collection. This ensures the sample consists of fresh formation water rather than stagnant water that has been stored in the well casing.

During all sampling activities, well purging equipment is positioned so that any potential volatile organic sources, such as vehicles, gasoline-driven generators, or air compressors, and fuel tanks are downwind of the well. This avoids contamination caused by entrainment of volatile contaminants in the sample. Any potential sources of volatile organics that are unavoidable are noted on the well purging log. An example of a well purging log is presented on Figure 5-17.

McCLELLAN AFB THIRD QUARTER 1988 WELL SAMPLING DATA SHEETS

Well Number: MW-33S

Samplers: _____/_____ Date: _____ Arrived at Well: _____
Sample Time : _____
Weather: _____ Left Well : _____
Total Time : _____

---------- Historic Information ----------

Well Diameter (inches): 4 Depth to Water: 91.66

Well Volume (gal): 3.48 Date Measured: 06/30/88

Screen Depth (feet): pH: 6.8 Conductivity: 810

Date Sampled: 04/25/88 Sampler's Notes: SPLIT SAMPLES, AMBIENT, 601, 602

---------- Sample Specific Information ----------

Depth to Water measured from (circle one): Well Wizard / Top of / Sounding
Cap Well Tube

Pump Intake Setting (feet):
Total Depth (feet): 97.00

Depth to Water (feet): -( )
---
( )
X 0.65
---

Well Volume (gal): = _____(gal) x 3 = Min. Purge Vol.____(gal)

Parameters after Purging (circle): 601 602 604 624 625 9010 Metals

Temperature (degrees C) _____ pH: _____ Conductivity (umhos): _____
Purge Pump Controller
Reading (psi): _____ Total Volume Purged(gals): _____

---------- QA/QC ----------

( )Field Duplicates ( )Split Samples ( )Trip Blanks in custody ( )Ambient or Equipment Blanks

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 601 | 602 | 604 | 601 | 602 | 604 | | | | 601 | 602 | 604 |
| 624 | 625 | 9010 | 624 | 625 | 9010 | | | | 624 | 625 | 9010 |
| Metals | | | Metals | | | | | | Metals | | |
| I.D. | _____ | | I.D. | _____ | | I.D. | _____ | | I.D. | _____ | |

(check appropriate QA/QC and circle parameters sampled)

---

Comments/Field Notes:_____

_____

_____

_____

(PAGE 1 OF 2)

Figure 5-17. Groundwater Sampling Field Sheets

Well Purging Log

Monitoring Well No.:_____ Date:_____

Discharge Rate: (gpm) _____ Purge Method: Bladder, Purge, Bail

Sampler(s) Initials: _____ (Circle One)

| Time | Elapsed Time | Cumulative Volume Purged | pH | Conductivity (micromhos) | Temperature (°C) | Field Notes |
|---|---|---|---|---|---|---|
| | Start-Up of Purge | | | | | |
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Comments:

In those wells where purge pump is used for purging, operate the bladder pump until the stabilized purge parameters are matched. (See Sampling Guidelines)

NOTE WHEN SWITCHED TO BLADDER WAS MADE.

Meter No. _____

(PAGE 2 OF 2)

**Figure 5-17. Groundwater Sampling Field Sheets (continued)**

For wells equipped with dedicated pump systems, well purging is accomplished with a positive gas displacement purge pump, or with the bladder pump if the well is not equipped with a purge pump. If a well is not equipped with a dedicated pump system, it can be purged with a bailer or electric submersible pump or the DIS system. The bailer or submersible pump is positioned near the middle of the screened interval of the well to ensure that standing water is removed and fresh formation water is drawn into the well. The placement and operation of the DIS system is described in Section 5.6.1.3. Purged groundwater is collected in trailer-mounted 55-gallon tanks and discharged into the McClellan AFB GWTP for treatment and disposal.

When purging a well using a dedicated system, submersible pump, or bailer, purging is considered complete when the indicator parameters of pH, temperature, conductivity, and color have stabilized and a minimum of three wetted casing volumes have been removed from the well. The wetted casing volume of a well is calculated by using the following equation:

$$V = 3.14\ r^2\ L\ x\ 7.48\ \text{gallons/ft}^3$$

where:

V = One wetted casing volume (gallons);
r = Inside radius of casing (feet); and
L = Height of water column in well (feet).

To determine when stabilization has occurred, pH, temperature, and conductivity are monitored on a regular basis until two successive readings of all three parameters do not vary by more than $\pm 0.1$ pH unit, $\pm 1.0$°C, and $\pm 5$ percent change in micromhos, respectively. The volume of water discharged between the last two readings used to verify stabilization will be no less than 20 percent of the total amount of water purged. If stabilization does not occur, samples may be collected after a total of six wetted casing volumes have been removed from the well.

When purging and sampling wells equipped with dedicated systems, the same purging procedure is followed; however, after switching to the bladder pump, three additional gallons of water are removed. While purging water from the bladder pump, pH, temperature, and conductivity are monitored. When the parameters stabilize, a sample may be obtained. If, after removing one screened interval volume, the parameters still do not stabilize, collect a sample and note the discrepancy. Figure 5-18 presents a sampling

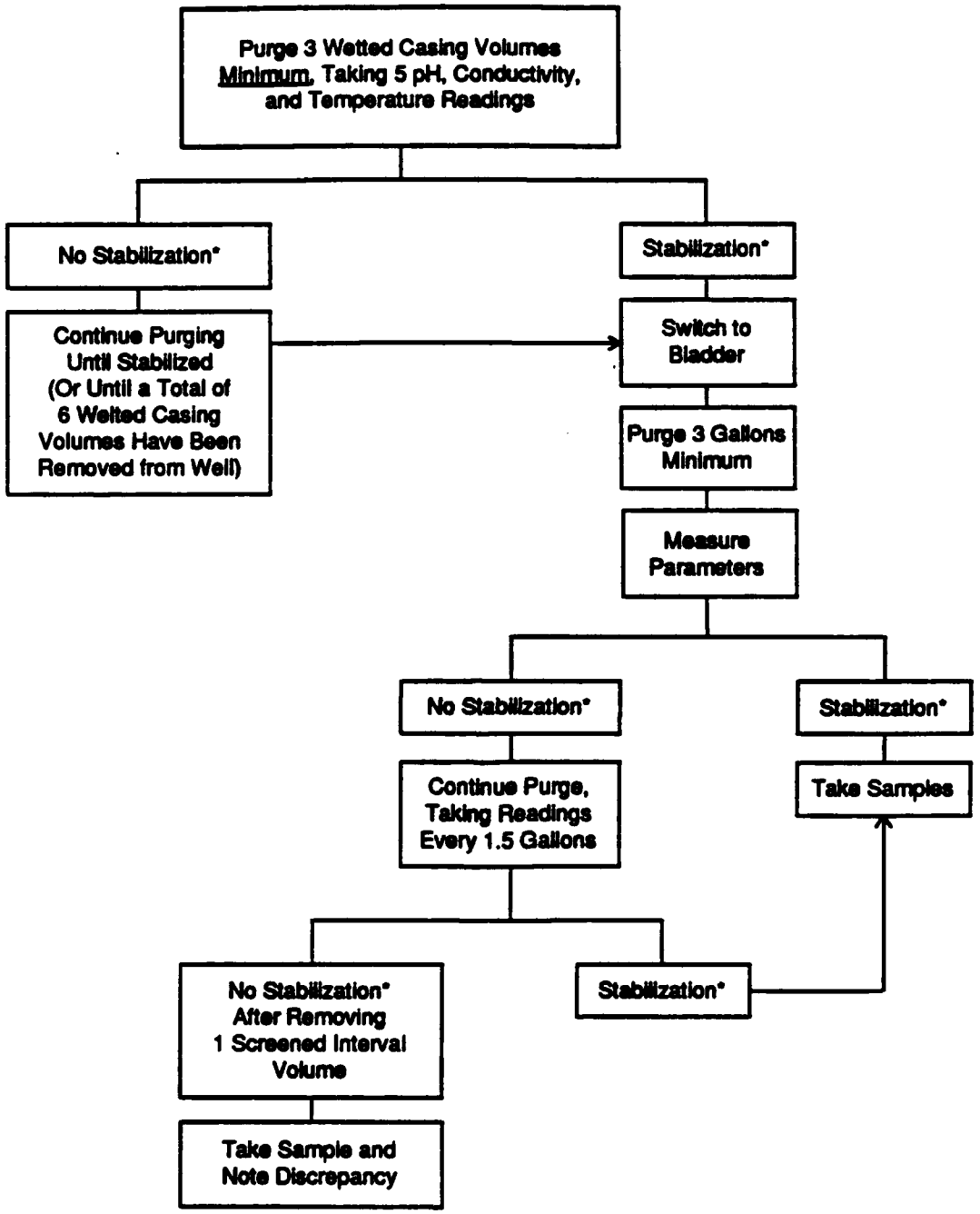


**Figure 5-18. Guidelines for Switching from Purge Pump to Bladder Pump**

0392-078-2B

decision flow chart summarizing guidelines used when switching from the purge pump to the bladder pump.

In low-yield wells which are purged dry before three wetted casing volumes have been removed, the sample shall be collected as soon as a sufficient amount of water has re-entered the well to obtain a minimum of three pH, conductivity, and temperature readings and to collect samples. The time at which the well was purged dry is recorded on the groundwater field sheets, as well as the volume of water removed prior to sampling.

A 5-gallon bucket (or similar container of known capacity) is used to measure the amount of water being removed from the well during the purging process. Elapsed time is noted as the container is filled, thereby allowing the calculation of the discharge rate. Record the total amount of water purged from each well on the field sheets. The water is directed into a "water wagon" for disposal at the McClellan AFB GWTP.

During purging, one field team member is responsible for handling field equipment and taking purging measurements. The other is responsible for recording observations and taking water parameter measurements.

**Sample Collection**

Groundwater samples are recovered in a prearranged priority, so all collection and handling takes place as efficiently as possible. Although the actual sample collection protocol will depend on the analytes of interest, it is important to be consistent in general sample collection procedures. Prior to using the bailer, or collecting a sample from the discharge line, wear new, clean disposable polyvinyl chloride gloves to avoid cross contamination. Typically, samples for volatile constituents are collected first. During sample collection, one member of the field team oversees the operation of field equipment and collection of samples. The other team member is responsible for recording field data, container labeling, etc. Such consistency will help minimize any errors that may compromise data validity or promote bias in the analytical results.

During the sampling procedure for wells equipped with dedicated pump systems, special attention is given to the pump discharge rate. All samples are collected from the Teflon®-lined discharge line connected to the bladder pump. Care must be taken to avoid taking the discharge line to any part of the sample containers or caps. Samples for

volatile organic constituents are collected only after slowing the delivery rate to 100 mL/min or less, or until nonturbulent flow (no bubbles) is obtained, to avoid aeration of the sample. For other analytes, adjust the discharge rate in accordance with the size of the sample container being filled. A slow rate of 150 mL/minute or less allows easy filling of smaller bottles. Large volume bottles can be filled at a greater flow rate. However, the sample delivery rate should not exceed the pumping rate used during well purging.

Wells not equipped with a dedicated pump system are sampled using a decontaminated Teflon® bailer. A new monofilament line or braided stainless steel line is securely attached to the bailer. The bailer is lowered slowly into the well, taking care to cause as little disturbance as possible to the water surface. Water is collected from the middle of the screened interval of the well. As the bailer is lowered and raised, take care to keep the line clean and off the ground surface. To minimize this problem, the monofilament line can be directed into a clean bucket or similar container as the bailer is being raised in the well. The bailer is filled and emptied twice with well water to condition it before collecting samples. A bailer emptying device is used to transfer water from the bailer into the sample containers.

Samples are collected from bailers using the flow control valve by attaching the valve to the bailer after purging is completed, and then slowly opening the valve until smooth, steady flow is obtained. The sample bottle is then placed directly under the bailer and attachment, so a minimum free-fall distance is allowed during sample collection. This will minimize the potential for agitation or aeration of samples that may cause loss of volatile organic compounds.

The sample bottles for VOCs must be filled slowly to prevent the entrapment of air bubbles; splashing or agitating the water is avoided. Take care to avoid touching the mouth of the discharge line, the top of the sample bottle, the inside of the cap, or the Teflon® septa. A septum that falls out of the cap onto the ground cannot be used. The bottle is filled completely such that a meniscus forms. The cap is screwed on and the bottle inverted, tapped firmly, and checked for the presence of air bubbles. If a bubble is present, the sample is discarded and a new sample collected; new preservatives are added if appropriate. If a bubble appears a second time in the same bottle, discard that bottle and prepare a new one. Accurate analytical results for volatile organic compounds may be compromised if there is any free air trapped in the sample container.

**Conductivity, pH, and Temperature**

Conductivity, pH, and temperature of water can change over the sample holding time. Consequently, these parameters are determined in the field at the time of sample collection. The pH and conductivity probes or cells are rinsed at least two times with the water to be tested prior to making the measurements. Groundwater temperature is measured concurrently with pH and conductivity. Values for pH, conductivity, and temperature are recorded with a minimum accuracy of $\pm 0.1$ pH unit, 5 percent in micromhos, and $\pm 1.0°C$, respectively. If radioactivity measurements are required, they are performed at the same time as the initial and final pH, conductivity, and temperature readings. The results are recorded in the comments.

**Sample Labeling**

Label all samples clearly with the following information:

- Project name/client;
- Well number or sample location;
- Sample type (analytical method);
- Preservatives used;
- Sampler's name and initials; and
- Date.

Labels are completed using a ballpoint pen and securely attached to the sample jar. Permanent markers (i.e., Sharpie®) are not used in the vicinity of sample collection activities because they contain volatile organic compounds that may contaminate the sample. To ensure future legibility and limit the potential for cross contamination in case of breakage, all sample jars are sealed and transported in individual Ziploc® bags.

**Filtration**

Filtration is required for samples recovered for dissolved metals analyses. Filtration is accomplished in one of two ways. Disposable 0.45 micron in-line filters are used if dedicated pump systems are in place. The filter is fitted to the discharge line immediately before collecting the samples which require filtration. If a well is sampled with a bailer, filtration is accomplished by pumping the sample through a 0.45 micron filter using

a portable peristaltic pump. If required, preservation of the sample is performed at this time. All filtration equipment is decontaminated prior to use, and rinsed with a 10 percent nitric acid wash.

**Sample Storage and Transportation**

All samples are stored at approximately 4°C from immediately after collection until analysis. In the field, samples are stored with ice or blue ice in coolers (ice chests). Protective foam or styrofoam packing is used to minimize the risk of breakage during transport, and to ensure that the samples do not freeze. Individual sample containers are sealed with a strip of paper tape extended across the container cap.

When packaging samples for commercial transport, an absorbent material such as vermiculite is used to minimize the effect of any breakage. Sample containers are packed in the upright position and separated by padded materials. Sample packaging requirements for hazardous materials requiring interstate transport are defined in the Code of Federal Regulations (CFR) 49, Chapter 1, Part 171 and are utilized during sample transport. These requirements outline in detail the proper classification and procedures for transportation of hazardous materials and are included by reference.

### 5.6.4 Discrete Interval Sampling System Procedures

The DIS system operation and sample collection procedures are described separately because this system operates under slightly different conditions from the dedicated pump or submersible pump/bailer systems. A demonstration of the DIS system was conducted. A study was performed to document that samples collected using this system are comparable to those collected by the other procedures. The system and operating procedures were approved for use by agency and Air Force representatives. The same pre-sampling operations are followed, and sample labeling, recordkeeping, filtration, and sample handling procedures described in Section 5.6.3 are followed.

**Initial Set-Up Procedures**

Position the truck at the wellhead. Next, remove the control pendant bar, the flexible high pressure nitrogen line, and the two purge water discharge lines from their storage compartment on the truck, and attach to their proper connecting points.

After the truck has been positioned, measure the water level using a standard water-level meter and record the information on the well sampling data sheet. This measure is critical for the proper calculation of the packer inflation pressure.

Once the water level has been measured, start the generator to provide power for the on-board systems. The master control panel (Figure 5-19) is used to control the electrical systems and the hydraulic system, which lowers the sampler system and the purge pump down the well.

Before each use, decontaminate the sampler and packer unit following the procedure described in Section 5.6.2.

The assembly of the sampler and packer unit follows the decontamination procedure and is done in three steps:

- The sample chamber assembly;
- Connection of the packer to the sample chamber; and
- Connection of the power control lead and packer inflation tubing.

**Sample Unit and Purge Pump Placement Procedure**

The sampler and packer unit assembly is then lowered to the determined depth. The placement of the sampler and packer unit to the proper depth is a critical step in the sampling procedure. For proper packer positioning, refer to well construction details for each well to determine the total depth of the well, and determine the depth of the screened interval. Place the bottom of the packer approximately 5 feet above the top of the screened interval as shown in Figure 5-20.

Following the placement of the sampler and packer unit, inflate the packer to the determined pressure (Figure 5-21). The inflation of the packer is a critical step; too little pressure will not inflate the packer sufficiently to seal off the well casing, allowing water to flow around the packer instead of through the sample chamber. Too much pressure can cause the packer to fail, destroying the packer and possibly damaging the well. The formula to use when calculating the proper inflation pressure is:

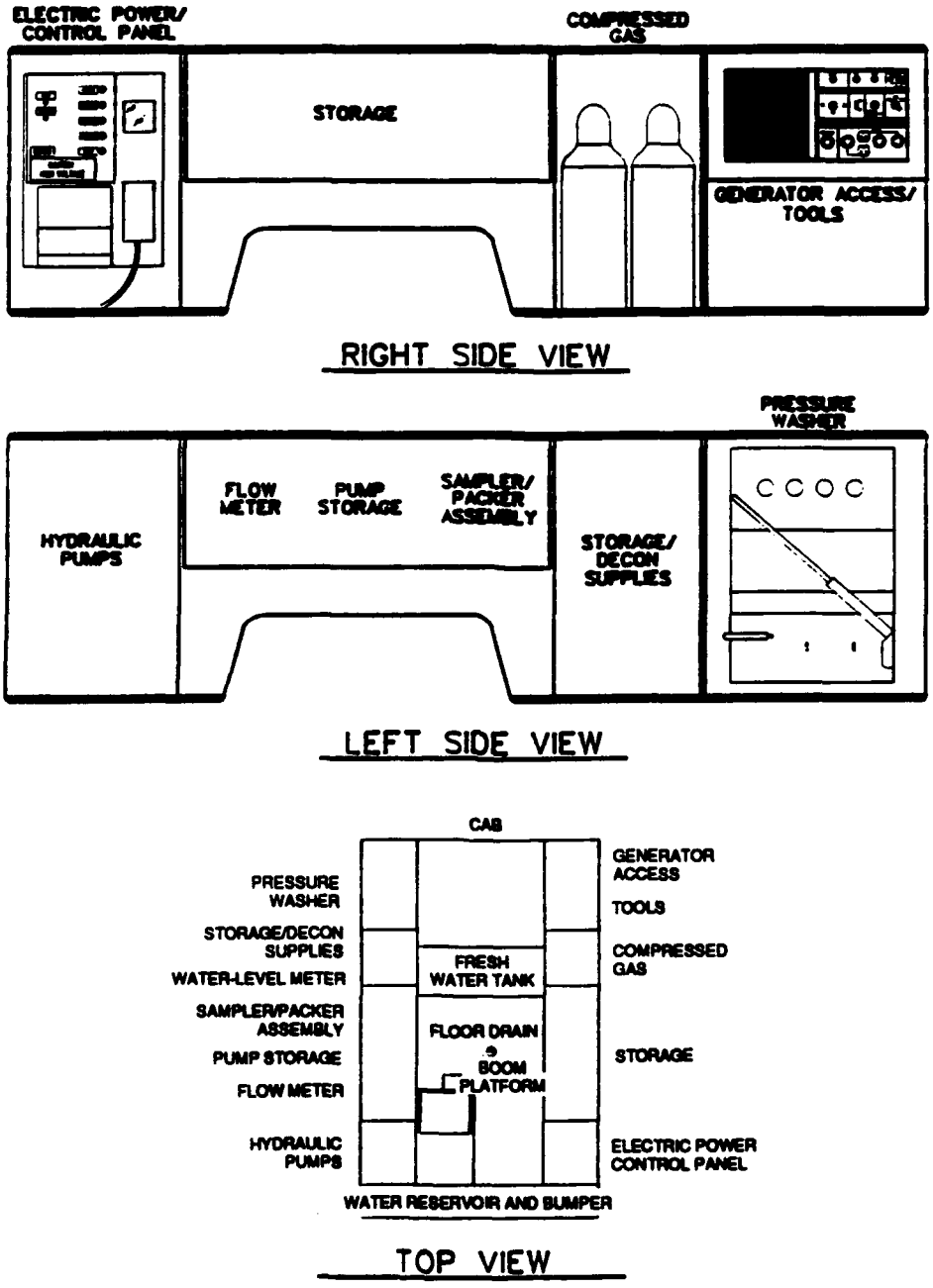


**Figure 5-19. Sampling Vehicle Master Control Panel**

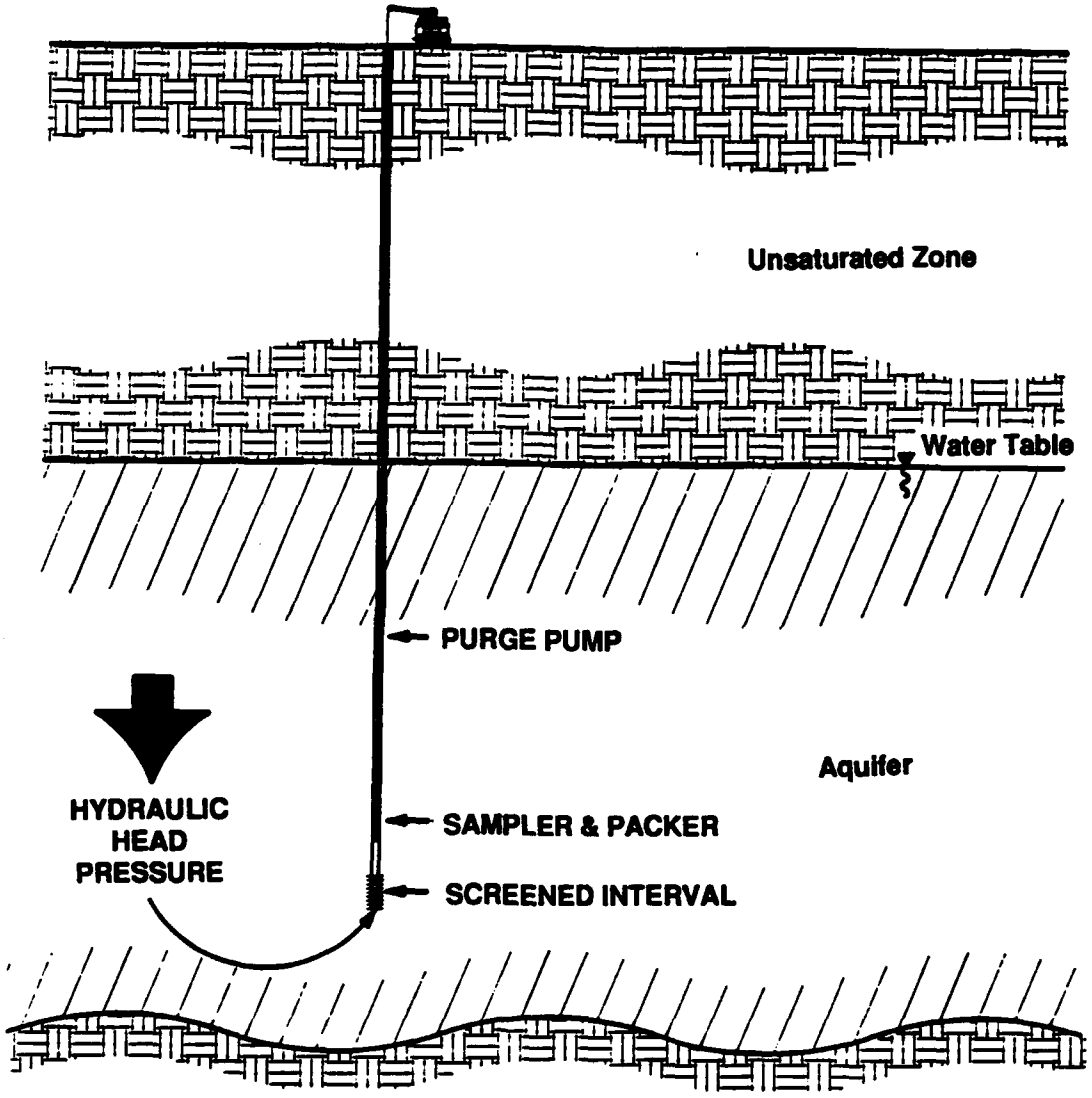


Figure 5-20. Sampling Schematic

0392-078-55

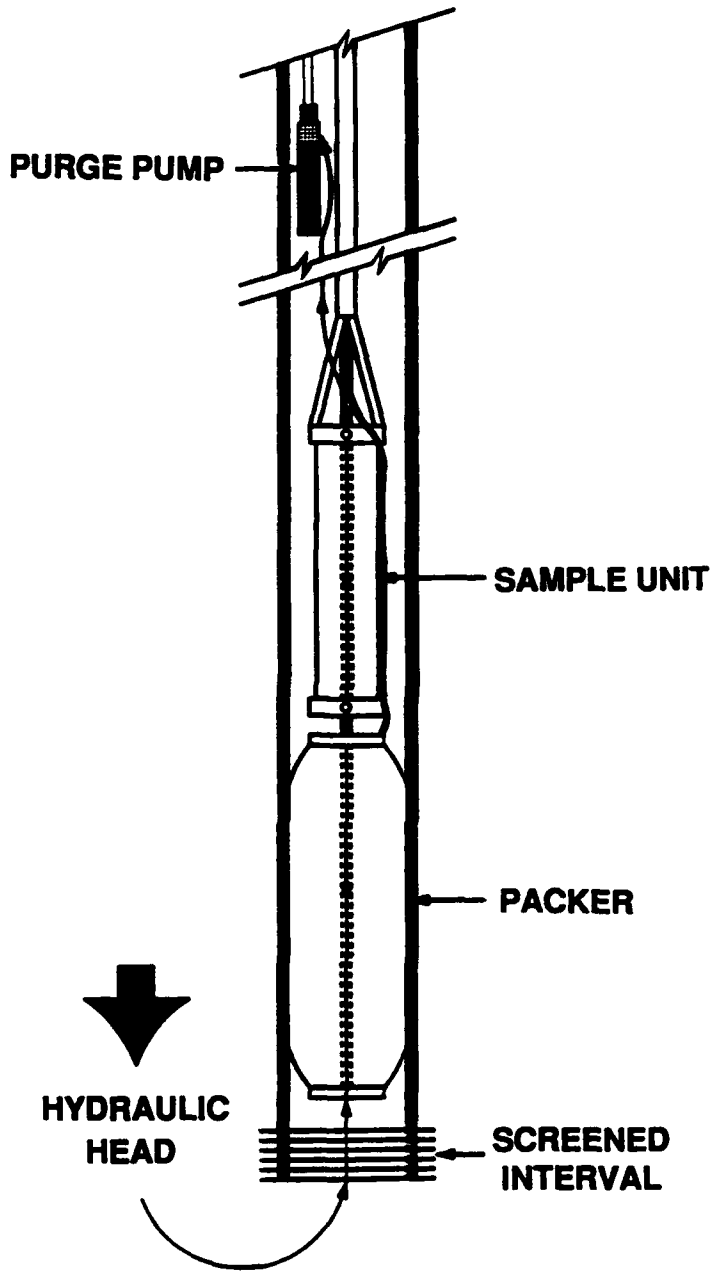


Figure 5-21. Closeup of Inflated Packer

0392-078-54

[{(P-S) * .433} * 1.1] + 35 = INFLATION PRESSURE (pounds per square inch)

P= PACKER SUBMERGENCE DEPTH (feet below measuring point)

S= STATIC WATER LEVEL (feet below measuring point)

After the packer has been inflated, lower the purge pump to the determined depth. The placement of the pump at the correct depth is important; insufficient submersion of the pump may cause the pump to encounter a "run-dry" situation in which the pump can no longer pump water from the well. Place the pump no less than 5 feet below the measured water level and no more than 15 feet below the measured water level (see Figure 5-20). Information about pump placement, packer inflation calculations, and pressure are recorded on the well purging field data sheets.

**Well Purging Procedure**

Begin the well purging procedure by opening the sampler solenoid. This is opened by turning the power switch in the Master Power Control cabinet to the "On" position.

After the sampler solenoid is open, turn the pump power control switch to the "On" position on the Master Control Panel. The flow rate of the purge pump is noted by viewing the on-board flow meter. The purge flow rate should be maintained at the maximum sustainable level by turning the rectifier knob on the Master Control Panel.

Once the purge pump is operating, the purge cycle will begin and the determined purge volume is evacuated. The purge volume is determined by multiplying the area from the bottom of the well to the bottom of the packer by 0.65 on a 4-inch diameter well and then multiplying that value by 5. This formula calculates five times the volume of water below the packer, ensuring that fresh formation water is drawn into the screened interval.

After the purge volume has been evacuated from the well, close the sampler solenoid. The purge pump is turned off only after the sampler solenoid has been closed. This ensures that only fresh formation water is in the sample chamber.

Once the sample chamber is closed, retrieval of the pump, sampler and packer begins. First, bleed the packer compressed air supply. Next, retrieve the pump. At this

time, the purge hose is decontaminated using the on-board steam cleaner. After retrieving the purge pump, bring the sampler and packer unit to the surface. The control hose is also decontaminated with the steam cleaner.

**Sample Collection Procedure**

Sample collection occurs after the sampler and packer unit has been retrieved and the packer has been removed, and sampling flow control valve is attached to the bottom of the sampler body. The sampler flow control valve is lifted into the open position and a sample of water is taken for parameter tests (pH, conductance, temperature). These parameters cannot be measured during the purging procedure because of the system configuration. Once these parameters have been recorded, the sample flow control valve is opened and the samples are collected.

After the samples for the well have been collected, the sampler, packer, and purge pump are disassembled and placed in the decontamination tray in the bed of the truck for decontamination. A dilute solution of Alconox® and drinking water is sprayed on the inside and outside surfaces to be decontaminated. Steam clean the inside and the outside of the parts until there are no more soap bubbles on the parts.

Measure the post-purge water level and log the measurement in the logbook.

## 5.7 Surface Water Sampling

Water samples are collected from surface water drainages according to the following procedures. The field preparation procedures for the sampling event include scheduling, QC sample assignment, sample container preparation, and development of documentation materials. Health and safety procedures described in the project Health and Safety Plan will be followed. These include procedures for monitoring, personal protective equipment, and physical hazards.

**Sample Locations**

Surface water drainages to be sampled include on-base storm drainage systems of Magpie Creek, Arcade Creek, and the "Unnamed Stream." Emphasis is placed on drainages that cross the base boundaries and that potentially receive contaminants from

former waste disposal sites. Specific sample locations and analytical parameters are identified in the SAP prepared for the surface water survey. All sample sites are confirmed by visual inspection during the preparation of the SAP. The site locations will be documented by measuring northing and easting from a surveyed reference point, or professionally surveyed if the DQOs require a greater level of accuracy. Photographs taken at the sites can be used to document site conditions at the time of sampling.

**Sample Collection**

Grab samples are collected from the midpoint of each drainage sampled, or from the edge of the pond or lagoon. The samples are obtained in a manner that minimizes the potential for cross-contamination by collecting samples directly into the containers. Sampling equipment will be decontaminated following the procedures described in Section 5.6.2. Background locations are sampled first, then the furthest downstream sample. The upstream locations closer to the suspected source or known discharge point are sampled last. This will minimize the potential for cross-contamination or carry-over from sampling equipment. In locations where both surface water and sediment samples are to be collected, the surface water are collected first. Samples are collected from the bank nearest the suspected source of contamination.

Prior to sample collection, field personnel will re-suit with new, clean, poly-vinyl chloride surgical-type gloves in order to avoid cross contamination of the sample or bottle. Water samples for VOCs are collected by submerging the sample containers. A decontaminated stainless steel bucket or Teflon® beaker is used when collecting composite samples for nonvolatile parameters, or when there is not adequate stream-flow to submerge the sample container. Water collected in the bucket or beaker is carefully decanted into the proper sample containers. Streambed augmentation is used if samples are to be collected for volatile analyses, and the flow or depth is not great enough to submerge the sample container. This consists of briefly damming the flow, or creating a depression in the streambed. The sample is collected after the disturbed sediment clears.

Surface water samples may also be obtained using a continuous automatic sampler. With a continuous sampler, an intake probe is secured at the sampling point and the sampler is preprogrammed to collect either individual or composite samples at designated times throughout the day.

After collection of each sample, all boots and waders are decontaminated according to the following three-step process: 1) wash equipment in detergent and potable water solution using a brush; 2) rinse with potable water; and 3) rinse with deionized water. Between sample locations, all sample collection equipment is decontaminated using the standard procedure presented in Section 5.6.2.

**Water Blanks and Duplicates**

Ambient (field) blanks are collected by pouring ASTM Type II Reagent Water directly into the sample container in the field. A trip blank, a sealed vial of Type II Reagent Water, accompanies samples collected for VOC analysis. One trip blank per cooler or other designated frequency in the SOW is collected. Surface water duplicates are collected as aliquots of a composite sample, except for volatile parameters that must be collected as sequential samples from the water body. Requirements for blanks and duplicates are presented in Section 10.0, Internal Quality Control, and in the SOW and work plan for the sampling activity.

**Field Measurements**

For all surface water bodies sampled, the depth of water, temperature, pH, and specific conductance are measured prior to collecting samples for laboratory analyses. The pH/conductivity meter is rinsed with Type II reagent water prior to sampling and between sampling locations.

Portable real-time organic vapor analyzers may be used to perform real-time screening of total hydrocarbon vapors. An organic vapor analyzer (OVA) with a flame ionization detector and a range of 1 to 10,000 parts per million (ppm) is used to detect total hydrocarbons. In addition to the OVA, an HNu® Systems photoionizer, with a range of 0 to 2,000 ppmv is used to detect nonmethane hydrocarbons. The instruments are calibrated daily using certified methane-in-air (OVA) or TCE-in-air (HNu®) gas standards. Further details on these instruments can be found in Sections 7.2 and 8.4 of this QAPP.

Radioactive material may be present at specific sampling locations, therefore, radioactive spectrum detector is available for use.

**Recordkeeping and Sample Handling Procedures**

All data collected in the field are recorded on sample field sheets. Pertinent field information, including date of last rainfall and amount (if within the past one to two weeks), the width, depth, and flow rate of streams, the surface water conditions, and the location of any discharge of pipes, sewers, or tributaries is recorded on field sheets. Sample control information is documented in a master log. Chain-of-custody records are completed subsequent to sample collection. Data collection and chain-of-custody forms are described in more detail in Section 6.0.

All samples will be stored at 4°C from immediately after collection until analysis. In the field, samples are kept in coolers (ice chests) kept cool with ice. The chain-of-custody record accompanies sealed samples to the laboratory.

## 5.8 Soil Samples

Surface and subsurface soil samples are collected during various phases of the RI. The rationale for sample collection, including sampling locations, analytical parameters, and number and type of samples is established in the SAP for the field effort. Sample collection is documented using sample logs and chain-of-custody forms as described Section 6.0. In addition, field data are documented on data sheets prepared for each sampling task and sampling locations are noted on site-specific maps. The field data sheets are described in the individual work plans. Lithologic logs are prepared during all drilling operations. Preparation of lithologic logs is discussed in Section 5.2. Health and safety procedures presented in the project Health and Safety Plan will be followed during all field procedures.

Surface and near-surface soils may be collected by a number of techniques, including surface scraping, hand augering, and core sampling. Subsurface soil samples are collected during drilling operations. The principal drilling technique to be used for subsurface soil sampling is hollow-stem augering, although mud rotary, air rotary, sonication, and dual tube air percussion may be used in some cases, depending upon the DQOs, and efficiency of the specific drilling procedures will be documented in the SAP. Samples are collected using split-spoon samplers, modified California samplers, Shelby tubes, or directly from cuttings. All of these methods can be used to collect discrete or composite soil samples. Soil samples to be analyzed for nonvolatile parameters can be composited either in the field or the laboratory; soil samples to be analyzed for volatile parameters cannot be

composited because of potential loss of those compounds. Examples of field data sheets used to document soil sample collection activities are shown in Figures 5-22 and 5-23.

**Surface Scraping**

Surface scraping may be used to collect soil samples from the ground surface, sidewalls and bottom of trenches or excavations, and scale or sediment from tanks, ponds, impoundments, or streams. The samples are collected using the following procedure:

1. Check all equipment and sample containers to ensure that the equipment is clean and that the containers are new and have been properly prepared.

2. Label container and initiate the chain-of-custody process and task/ sample-specific data sheet, as applicable.

3. Prepare surface for sampling by removing any vegetation, sticks, stones, etc.

4. For discrete samples:

   a. Scrape a thin layer of soil directly into the sample container using a stainless steel spoon. This should be done as quickly and with as little disturbance as possible in order to minimize the loss of volatile and semivolatile organics.

   b. Cap the container with a Teflon®-lined cap and place in a cooler at 4°C.

5. For laboratory composited samples (nonvolatile parameters only):

   a. Collect individual samples at each location to be composited as described in Step No. 4 above.

**OU B SOIL AND SEDIMENT SAMPLE DATA SHEET**
**HAND AUGER AND SOIL BORING SAMPLES**

Boring ID_____ Date____/____/____

IC ID_____ Samplers____/____/____

Site ID_____ Cooler ID_____
(field cooler)

Location type: Surface, Borehole, Channel/Ditch _____
(circle one) (shipping cooler)

**Location Measurements:** Reference Point_____ _____Ft. N,S, E, or W
(MW Number or Bldg. Number & Corner)

Borehole depth:_____ _____Ft. N,S,E, or W

Borehole diameter:_____ _____Ft. N,S,E, or W

Construction Method: HSA, Hand auger, Sonication, Slide hammer _____Ft. N,S,E, or W
(circle one) (circle direction)

| Field Sample ID | Blind Sample ID | Beginning Depth | Ending Depth | Time | Max PID |
|---|---|---|---|---|---|
| | | | | | |

Comments:

Analyses Required (circle one or more): SW6010, SW7060, SW7196, SW7421, SW7471, SW7740, SW3550/SW8015, SW5030/SW8015, SW8040, SW8080, SW8140, SW8150, SW8240, SW8270, SW8280, SW8310, SW9012, SW9045, SW9060, SW9310 (E900.1), E901.1, E607, HML338, ASTM D2216-80, ASTM D2434-68, ASTM D2435-80, ASTM D422-63, ASTM D854-83, E600/M4-82-20, Fish Toxicity

| Field Sample ID | Blind Sample ID | Beginning Depth | Ending Depth | Time | Max PID |
|---|---|---|---|---|---|
| | | | | | |

Comments:

Analyses Required (circle one or more): SW6010, SW7060, SW7196, SW7421, SW7471, SW7740, SW3550/SW8015, SW5030/SW8015, SW8040, SW8080, SW8140, SW8150, SW8240, SW8270, SW8280, SW8310, SW9012, SW9045, SW9060, SW9310 (E900.1), E901.1, E607, HML338, ASTM D2216-80, ASTM D2434-68, ASTM D2435-80, ASTM D422-63, ASTM D854-83, E600/M4-82-20, Fish Toxicity

| Field Sample ID | Blind Sample ID | Beginning Depth | Ending Depth | Time | Max PID |
|---|---|---|---|---|---|
| | | | | | |

Comments:

Analyses Required (circle one or more): SW6010, SW7060, SW7196, SW7421, SW7471, SW7740, SW3550/SW8015, SW5030/SW8015, SW8040, SW8080, SW8140, SW8150, SW8240, SW8270, SW8280, SW8310, SW9012, SW9045, SW9060, SW9310 (E900.1), E901.1, E607, HML338, ASTM D2216-80, ASTM D2434-68, ASTM D2435-80, ASTM D422-63, ASTM D854-83, E600/M4-82-20, Fish Toxicity

**Figure 5-22. Example Soil or Sediment Sample Field Data Form**

Boring:_____
Date:_____

SOIL SAMPLE HEADSPACE READINGS

| Depth (ft) | PID Reading (ppmv) | Sample Number | Notes |
|---|---|---|---|
| .25 | | | |
| .5 | | | |
| .75 | | | |
| 1.0 | | | |
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| 6.0 | | | |
| 6.25 | | | |
| 6.5 | | | |

**Figure 5-23. Example of Soil Sample Headspace Data Form**

b. In the laboratory, homogenize each sample designated to be included in the composite and combine an equal weight of each in the composite.

6. For field composited samples for nonvolatile parameter analysis:

a. Collect an equal amount of soil from each location to be included in the composite by scraping a thin layer of soil into separate sample jars, and then empty all of the jars into a stainless steel bowl.

b. Homogenize the soil by thoroughly mixing with a stainless steel spoon.

c. Divide the homogenized soil into four equal quarters and collect the composite sample from one of the quarters.

d. Cap the labelled container with a Teflon®-lined cap and place in a cooler at 4°C.

7. Complete chain-of-custody and field data sheets. Log sample into logbook.

8. Decontaminate sampling equipment between each sample following the procedure described in Section 5.6.2.

9. Document sample locations on a map.

**Hand Augering**

Hand augering is used to collect soil samples from depths up to 10 feet BGS, although the technique can sometimes be used to a depth as great as 30 feet BGS. This method is not appropriate for collecting samples for volatile parameter analysis because undisturbed samples are not obtained, and volatile compounds may be lost (see the following core sampling method). Hand augers equipped with 3-inch diameter cylindrical stainless

steel bits are used. A typical hand auger kit is shown in Figure 5-24. Soil samples are collected as follows:

1. Check all equipment and sample containers to ensure that the equipment is clean and that the containers are new and have been properly prepared.

2. Label container and initiate chain-of-custody and task/sample-specific data sheet, as applicable.

3. Auger to the desired sampling depth. Soil for lithologic inspection should be placed on plastic sheeting.

4. For discrete samples:

   a. At the desired sampling depth, collect soil in the bit and empty into an stainless steel bowl. Typically, a 1-foot column of soil will be collected. However, if only a short column of soil is desired, the soil can be emptied directly into the sample container. Level 1 measurements using an OVA, OVM, or HNu® are taken and recorded on field data sheets at this time if required in the SAP. Follow the procedures in Section 8.4.

   b. Homogenize the soil by mixing with a stainless steel spoon. Minimize mixing as much as possible, or eliminate when sampling for semivolatile organics.

   c. Divide the homogenized soil into four equal quarters and collect the sample from one of the quarters. More than one quarter may be used if additional sample is needed for all parameters.

   d. Cap the container with a Teflon®-lined cap and place in a cooler at 4°C.

5. Backfill hole with native material, or fill hole with cement grout, if required by the SAP.

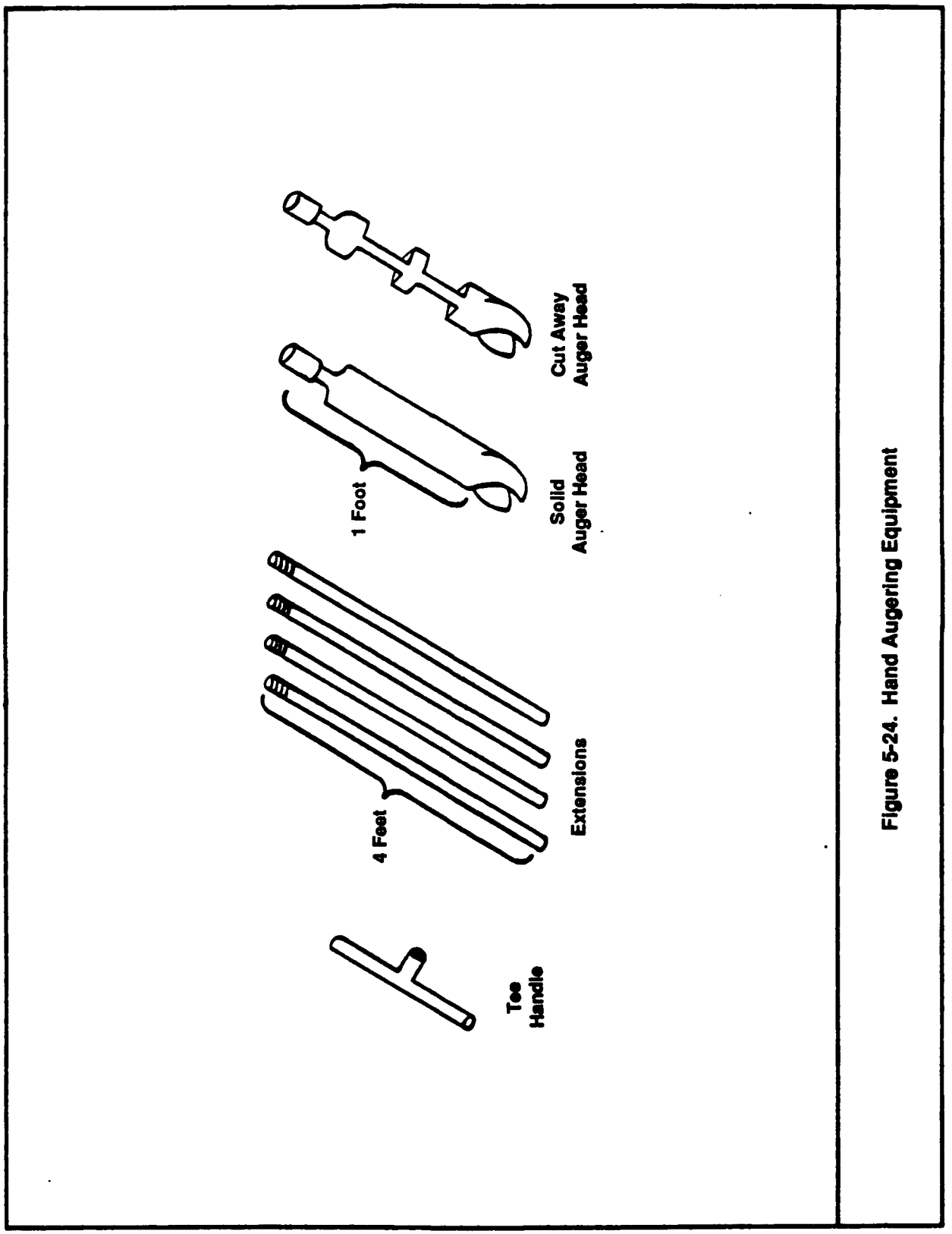


Figure 5-24. Hand Augering Equipment

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6. For laboratory composited samples (nonvolatile parameters only):

   a. Collect individual samples at each location to be composited as described in Step No. 4 above.

   b. In the laboratory, homogenize each sample designated to be included, and combine an equal weight of each in the composite.

7. For field composited samples (nonvolatile parameters only):

   a. Collect an equal amount of soil from each location to be included in the composite. If soil from different depths within the same auger hole are to be composited, place soil from each depth into a stainless steel bowl. If soils from the same depth in different auger holes are to be composited, collect soil from each location as described in Step No. 4 above and place in separate sample containers. Then empty all of the samples into the metal bowl after all locations have been sampled.

   b. Homogenize the soil by thoroughly mixing with an stainless steel spoon.

   c. Divide the homogenized soil into four equal quarters and collect the composite sample from one of the quarters.

   d. Cap the container with a Teflon®-lined cap and place in a cooler at 4°C.

8. Complete chain-of-custody and field data sheets. Log sample into logbook.

9. Decontaminate sampling equipment between each sample following the procedure described in Section 5.6.2.

10. Document sample locations on a map.

**Core Sampler with Soil Sleeves**

Core sampling with soil sleeves will be used to collect soil samples from depths up to 10 feet BGS. The core sampler with slide hammer handle is designed to collect relatively undisturbed soil samples by driving a cylindrical cup (similar to the bit of a split-spoon sampler) into the ground using a sliding, weighted handle. This method is appropriate for collecting samples for volatile and semi- or nonvolatile parameter analysis. The 6-inch-long cup contains stainless steel soil sleeves in which the soil is retained. Soil sleeves from 1 to 6 inches in length and 2 inches in diameter are available. Sampling subsurface soil requires drilling to the desired sample depth with a hand auger. The soil lithology is recorded on the boring log shown in Figure 5-3. Soil samples are collected as follows:

1. Check all equipment and soil sleeves to ensure that the equipment, including the sleeves have been properly cleaned. Ensure that the containers are new and have been properly prepared.

2. Initiate chain-of-custody and task/sample-specific data sheet, as applicable.

3. Drill to the desired sampling depth with a hand auger, if necessary. Soil for lithologic inspection should be placed on plastic sheeting.

4. At the desired sampling depth, drive the core sampler into the ground with a sliding hammer action.

5. Remove the core sampler and open the cup. Remove the soil sleeves from the cup and cap with Teflon® caps or Teflon® tape covered with PVC caps. Level 1 measurements using an OVA, OVM, or HNu® are taken and recorded on field data sheets at this time, if required in the SAP. Follow the procedures in Section 8.4.

6. Label each soil sleeve and place samples in a cooler at 4°C.

7. Backfill hole with native material, or fill hole with cement grout if required in the task-specific work plan.

8. For laboratory composited samples (nonvolatile parameters only):

   a. Collect individual samples at each location to be composited as described above; and

   b. In the laboratory, homogenize each sample designated to be included in the composite and combine an equal weight of each in the composite.

9. For field composited samples (nonvolatile parameters only):

   a. Collect individual samples at each location to be composited as described above;

   b. Remove an equal weight of soil from each sleeve to be composited and place in an stainless steel bowl;

   c. Homogenize the soil by thoroughly mixing with a stainless steel spoon;

   d. Divide the homogenized soil into four equal quarters and collect the composite sample from one of the quarters; and

   e. Cap the container with a Teflon®-lined cap and place in a cooler at 4°C.

10. Complete chain-of-custody and field data sheet.

11. Decontaminate sampling equipment between each sample depth following the procedure described in Section 5.6.2.

12. Stainless steel soil sleeves are cleaned prior to sampling by the following four-step process:

   a. Wash sleeves in a solution of nonphosphate detergent and potable water;

b. Rinse with potable water;

c. Rinse with distilled or deionized water; and

d. Bake at 160°C for a minimum of 8 hours.

**Hollow-Stem Augering and Split-Spoon Sampling**

Hollow-stem augering or sonication drilling will be used to collect subsurface soil samples from depths exceeding 10 to 30 feet BGS. The sampling sites will be marked and surveyed according to the procedure described in Section 5.2. Hollow-stem augering will be used in conjunction with a split-spoon sampler to collect soil samples as follows:

1. Initiate chain-of-custody and boring log. Initiate task sample specific data sheet, if any.

2. By using a conventional drill rig, as shown in Figure 5-1, a hole (typically 8 or 10 inches in diameter) will be bored to the required sampling depth. At the desired depth, the drive tip of the bit (and drive shaft) will be removed from the hole. A downhole hammer (or a drill stem with an external hammer) fitted with a split-spoon sampler will be lowered inside the auger stem to the sampling depth. The sampler will be driven into the soil approximately 18 inches and then removed, with the soil sample retained in the split spoon. No fluids will be used to advance soil borings.

3. A Dames & Moore (D&M) split-spoon sampler (Figure 5-25) with stainless steel liners will be used to obtain samples at 5-foot intervals or other designated intervals for chemical analysis, visual observation, lithologic characterization, and soil vapor screening. This type of sampler allows for the collection of relatively undisturbed samples and minimizes losses of volatile and semivolatile species.

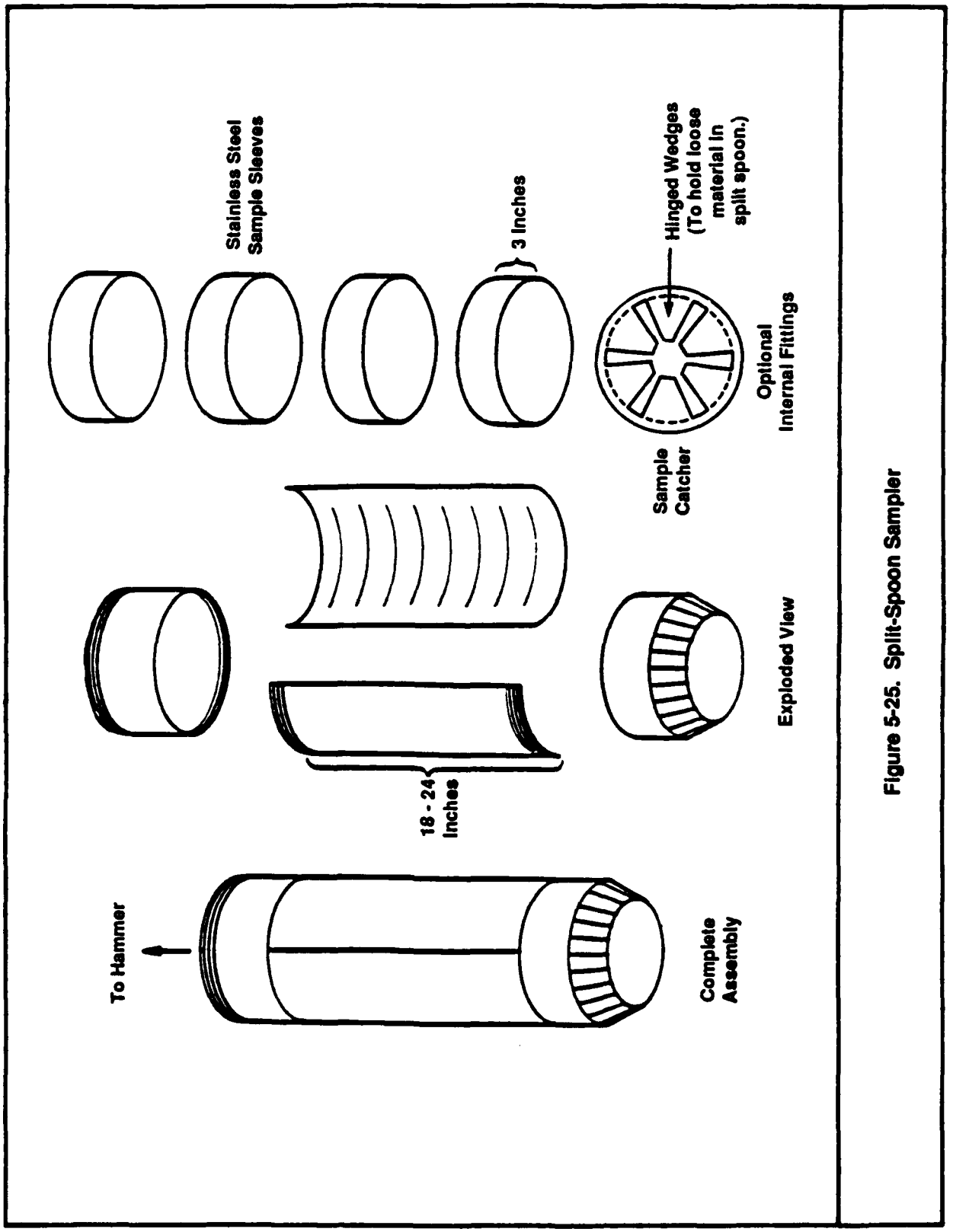


Figure 5-25. Split-Spoon Sampler

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The sampler is 18 inches long and 3 inches in diameter and contains six stainless steel liners approximately 3 inches long. The sampler is split lengthwise to insert/remove the liners. As the sampler is driven into the soil, samples are retained in the stainless steel liners.

4. After the sampler is removed from the hole, liners are separated, removed from the holder, and those selected for analysis are capped with Teflon® caps or wrapped with Teflon® tape and covered with PVC caps. Level 1 measurements using an OVA, OVM, or HNu® are taken and recorded on field data sheets at this time if required in the SAP. Follow the procedures in Section 8.4.

5. If samples are to be retained for nonvolatile chemical analysis, the soil will either be left in the liner and capped, or placed in an appropriate container with minimal disturbance. The container will be sealed with a Teflon®-lined cap.

6. A portion of the sample will be collected in properly marked sample bags and retained for visual inspection. Information on the soil lithology will be recorded on a boring log (Figure 5-3).

7. After each sample is collected, the split-spoon and tip will be decontaminated by the procedures described in Section 5.6.2.

8. The stainless steel liners will be prepared prior to use by: 1) washing in a solution of detergent and potable water; 2) rinsing with potable water; 3) rinsing with deionized water; and 4) baking at 106°C for a minimum of 8 hours.

9. In loose formations, a split-spoon sampler equipped with a sample catcher will be used to prevent sample loss. The catcher will be decontaminated in the same manner used to decontaminate the split-spoon sampler.

10. Label sleeves and place samples in cooler at 4°C.

11. The hollow-stem auger flights and split-spoon sampler will be steam cleaned between borings.

12. At the completion of each boring, the borehole will be abandoned by backfilling with native material to a depth of 10 feet BLS or 10 feet below the lowest extent of visible contamination, whichever is deepest, and grouting to the surface with cement, or as described in Section 5.2.8 if designated in the SAP.

13. Complete boring log and field data sheets.

## 5.9 Sediment Sampling

Sediment samples may be collected from dry streambeds by surface scraping, hand augering, or core sampling using a core sampler with stainless steel sleeves. When sediment cores are taken, the percent recovery shall be recorded in the field log. These methods have been described in Section 5.8. Sediment samples may be collected using a PVC tube or Ponar dredge when water present in the stream prevents the use of the above referenced sampling methods. Each of these techniques allows for the collection of discrete samples, with the option of compositing samples in either the field or the laboratory. The selection of the optimum sediment sampling technique will depend on the field conditions and the desired sample depth. The appropriate sampling procedures will be selected by the field task leader, and will be selected to satisfy the DQOs and accommodate the site conditions, and health and safety concerns. The rationale for sample collection, including sample locations, analytical parameters, type of equipment, and number and type of samples will be described in the SAP. Sample labeling, field data recording, compositing, QC requirements, and equipment decontamination procedures are the same as those described in Section 5.8.

### PVC Tube

A PVC or stainless steel tube may be used to collect sediment samples from beneath a liquid layer. The tube is driven down through the liquid into the underlying sediment to the desired sampling depth; a rubber mallet is used to drive the tube to depth if necessary. A peristaltic pump is used to remove the liquid from the casing. If there is any difficulty in removing the liquid (i.e., there is an insufficient seal on the bottom), the casing can be driven down further. After the liquid has been removed, the PVC tube can be with-

drawn with the sediment sample plug in the bottom. If the sediment is too fluid to remain in the tube, a hand auger barrel can be extended down through the casing to collect a sediment sample. The sediment is placed directly into the sample container or a stainless steel bowl for compositing.

### Ponar Dredge

A Ponar dredge (Figure 5-26) may be used to collect sediment samples from impoundments or flowing streams. This type of sampler has a jaw-type mechanism that is tripped from above to collect the sediment sample. The dredge is lowered through the liquid to the sediment with the jaws in the open position. The dredge is lowered slowly to minimize disturbance of the sediment upon contact. As the dredge is retrieved, the jaws close and the isolated sediment is brought to the surface, where the sample is transferred to the sample container or stainless steel bowl.

## 5.10 Soil Gas Sampling

Soil gas sampling is performed using a variety of sampling techniques. These techniques include measuring soil gas concentrations at the ground surface; in a slide hammer or hand-auger hole; in split-spoon samples; in shallow soil gas probes; downhole measurements in borings; and in soil gas monitoring wells. Additionally, emission flux measurements can be made at the ground surface, in borings, or in an exposed trench. The analytical methods currently anticipated for use with soil gas samples in the McClellan AFB RI/FS program are discussed in Section 8.0.

The selection of the optimum soil gas sampling technique will depend on the DQOs for the site or field effort, the site characteristics and accessibility, and the intended use of the analytical results. Measurements can be made using portable real-time organic vapor analyzers or direct reading sorbent tubes for Level 1 analyses, soil gas syringe samples can be collected for Level 2 analyses, or stainless steel canister or sorbent tube samples can be collected for Level 3 analyses. The rationale for sample collection, including sample locations, analytical levels and parameters, and number and type of samples collected, will be described in the SAP developed for each sampling task. The selected analytical parameters will determine the quantity of the sample, the container or media type, and the preservation required.

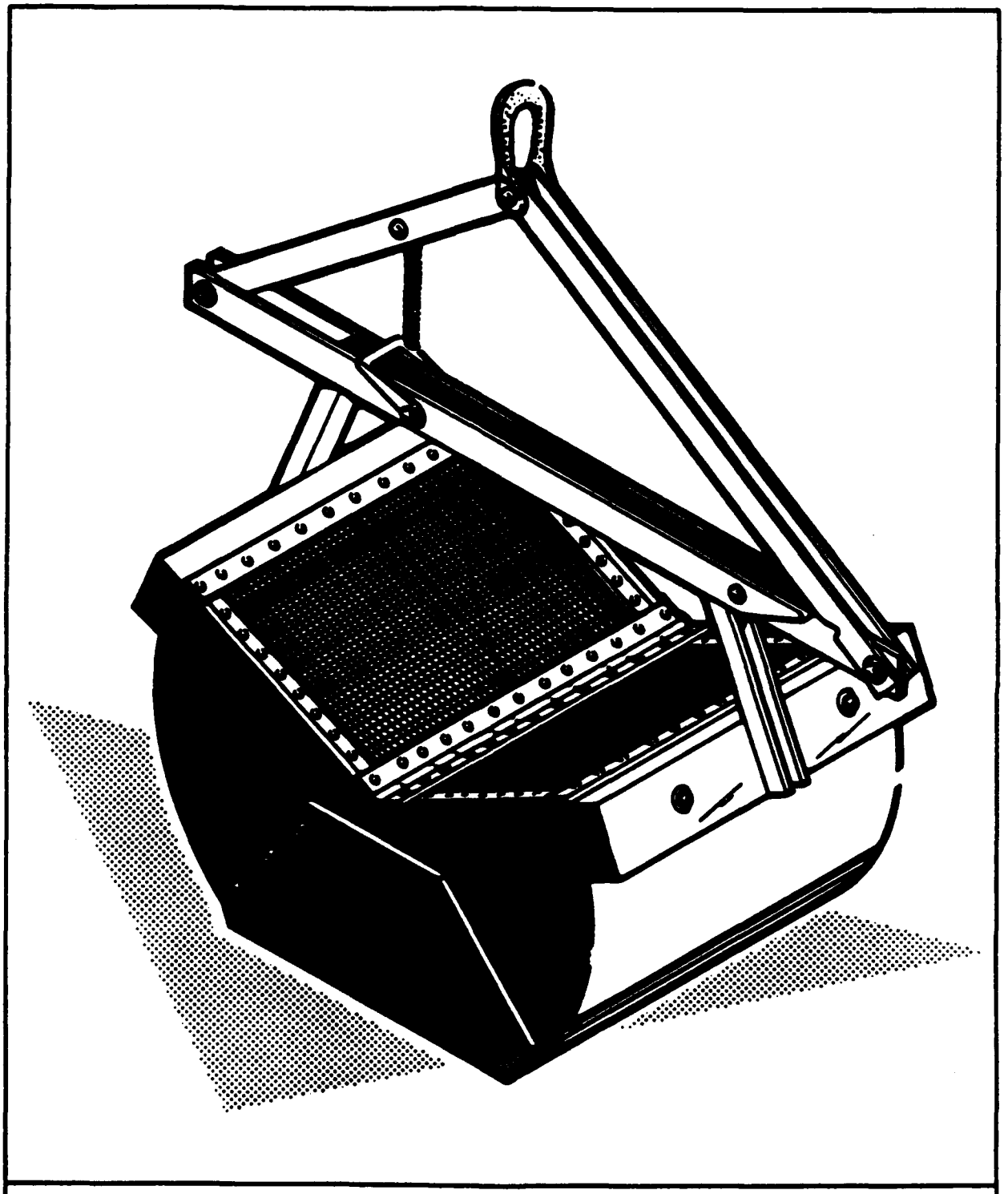

Figure 5-26. Ponar Dredge

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Sample collection will be documented using field log books, field data forms, and chain-of-custody forms. Site locations will be surveyed in the field by measuring northing and easting from a surveyed reference point, or locations can be professionally surveyed if greater accuracy is required. Preparation for any of the shallow or downhole soil gas sampling field efforts includes obtaining digging permits, developing detailed task instructions showing sample locations and QC requirements, example data sheets, training in documentation and field procedures, and a health and safety meeting. Digging permits are required to ensure that underground objects or utilities will not be encountered. Permits are requested by the field task leader and are coordinated by McClellan AFB EM.

### 5.10.1 Ground Surface Screening

The measurement of gas concentrations at the ground surface is performed in the initial phase of testing for potential surface emissions. This is intended as a preliminary survey to identify large variations in emissions and locations for additional measurements. Surface screening is performed by walking across a site on the grid pattern (established for the site in the SAP), using a sampling instrument to detect areas that have elevated concentrations of VOCs in the soil gas near the surface. Sampling can be performed by using portable real-time analyzers such as an OVA, OVM, and HNu®, or MicroTip™, or a sample can be collected in a stainless-steel canister or pump with sorbent tubes. The sampling is performed as follows:

1. Initiate field data sheet and map according to the procedures in Section 6.0 and the field task instructions. Ensure that instrument is properly calibrated and operating correctly (refer to Section 7.2). If samples are to be collected, initiate chain-of-custody or sample log and ensure that all sampling equipment is clean.

2. Standing upwind of the site (at least 3 to 5 yards upwind of the investigation area boundary, or as specified in the SAP), hold the input probe in the air, approximately 10 feet above the ground, and away from any equipment, etc. Note the reading, or average reading after one minute. This is the initial ambient reading. Record location and reading on site map and data sheet.

3. Walk the site on a grid pattern with the inlet probe of the sampling device within 3 inches of the surface. Walk the site slowly, noting any reading exceeding three times the ambient reading. After verifying the reading by remeasuring the gas at that location, record exceedances on a site map and data sheet.

4. After walking the site, repeat ambient reading at the same location as the initial measurement, provided that the wind direction has not changed. If the wind direction has changed, select a new upwind location for the final ambient measurement. Record location and reading on site map and data sheet.

5. Complete data sheet.

6. If samples were collected, complete chain-of-custody and label sample following the procedures described in Section 6.0 and the field task instructions. Purge sampling equipment with upwind ambient air for 5 minutes or until reading is at ambient level, whichever is longer, before initiating next sample.

### 5.10.2 Shallow Soil Gas Sampling Methods

The methods that may be used at McClellan AFB for shallow soil gas sampling include the following:

- Slide hammer hole method;
- Hand auger hole method;
- Hand-driven probe method;
- Pneumatic probe method; and
- Hydraulic probe method.

Slide hammer, hand auger, and the hand-driven methods are best used for small site investigations where only a few samples are needed, or in remote areas where other methods cannot be used. The pneumatic and hydraulic methods are designed for extensive soil gas investigations where up to 15 samples are required per day.

**Shallow Soil Gas Equipment Decontamination Procedures**

Sampling equipment used to produce sampling holes (slide hammer, hand auger) is decontaminated by first brushing soil from the equipment, and then washing with detergent and rinses with tap and distilled water. Sampling probes and retractable tips are decontaminated by steam cleaning or high pressure hot water washes between uses. Stainless steel or Teflon® sampling tubes are decontaminated between uses by purging with ambient air for 5 minutes or until field FID/PID readings are at ambient levels, whichever is longer. The vacuum of the field instrument or the vacuum pump is used to purge the tubing. Daily equipment blanks are collected in syringes to verify that decontamination is effective (when a field gas chromatograph is being used for the field effort). Teflon® tubing is replaced after five uses, or as needed if blanks indicate contamination. Septa used for syringe sample collection are replaced daily.

Glass sampling syringes and stainless steel sampling manifold fittings are baked in the GC oven overnight. Syringe blanks are analyzed to ensure that bakeout has removed all volatiles.

**Slide Hammer or Hand Auger Hole Method**

Shallow soil gas may be sampled using a slide hammer to drive a rod into the soil, providing a hole that will accept a sampling tube. The slide hammer is a steel rod with a weighted sliding handle used to drive the rod into the ground, as shown in Figure 5-27. The rod is 3 to 6 feet in length with a 5/8-inch diameter head. The weighted handle of the rod is used to drive the rod into the ground with repeated downward blows.

Shallow soil gases may also be measured and collected from hand auger holes in conjunction with soil sampling. Sampling can be performed at several depths within each hole, if desired. The maximum hand auger depth is usually 10 feet BGS, although depths up to 30 feet BGS may be achieved with some augers. Samples may be collected at designated intervals within the hole. The field procedure for hand auger soil gas sampling is as follows:

1. Initiate field data sheet and chain-of-custody or sampling log, if applicable, following the procedures in Section 6.0 and the field task instructions.

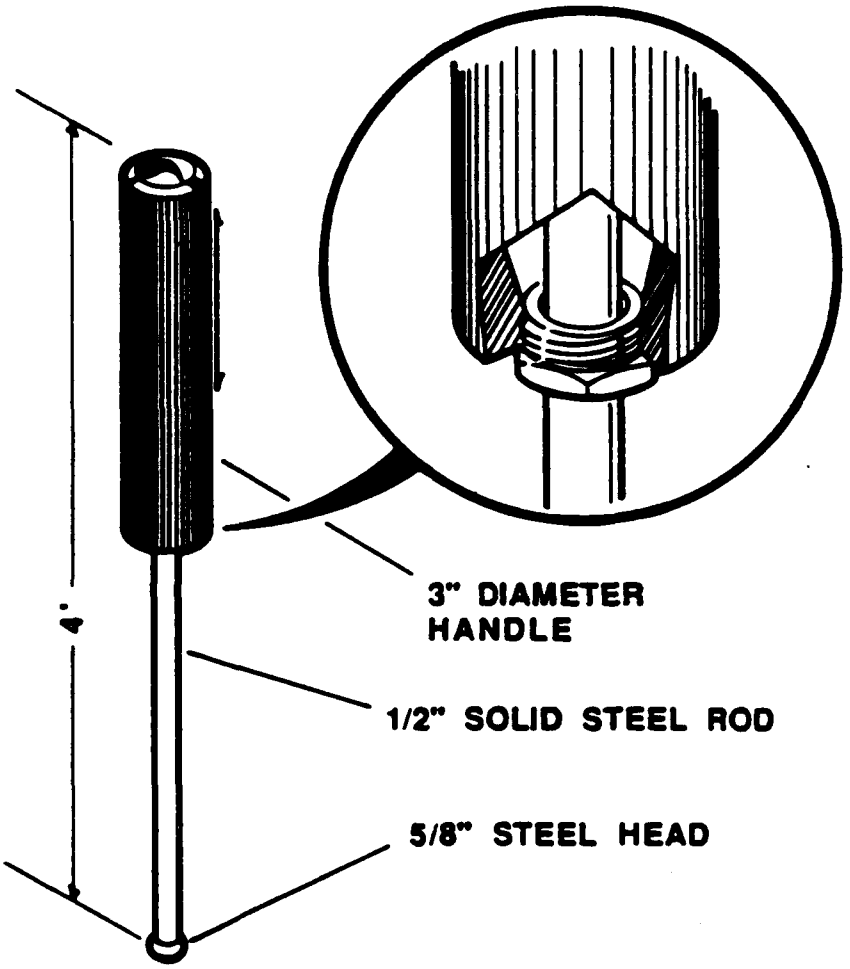


Figure 5-27. Slide Hammer

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2. The slide hammer or hand auger is used to prepare a hole to the desired depth.

3. A 1/4-inch Teflon® or stainless steel tube is inserted down the hole. Tube depth is measured relative to the depth of the auger stem or slide hammer to prevent soil clogging of the end of the tube.

4. Soil gas samples are drawn through 1/4-inch Teflon® or stainless steel tubing connected to the field organic vapor analyzer or a vacuum pump.

5. If a discrete sample for laboratory analysis is needed, disconnect field instrument and connect the sampling device to the tubing.

   a. Cover the boring opening with wood or plastic that has a hole cut in it to allow the sample tubing to pass through; this prevents soil gases from mixing with the ambient air or escaping from the boring.

   b. Withdraw a sample through the tubing at a rate specified for the sampling apparatus and/or analytical method.

   c. To collect syringe samples for Level 2 field gas chromatographic analysis:

      1) Install a septum in the sampling line upstream of the field instrument. The field instrument is left in place during sampling to assure the continued flow of soil gases at the septum.

      2) Purge the syringe three times with soil gas.

      3) Collect a sample.

d. To collect canister or sorbent samples for Level 3 analysis in an off-site laboratory:

1) Disconnect the field instrument from sampling line.

2) Connect the sampling device (canister or inlet to sorbent tube) to the sampling line; a Swagelok® tee fitting can be used to preconnect the canister or sorbet tube.

3) Collect the sample.

6. Complete chain-of-custody, if applicable, and field data sheet.

7. Decontaminate the sampling tube and other equipment following the procedures in Section 5.10.

8. Backfill the hand auger hole by using native soil or grout, as specified in the SAP.

**Hand-Driven and Pneumatic Methods**

The hand driven probe or pneumatic driven probe methods may be used for extensive shallow soil gas sampling investigations. These methods are semi-portable and can be used in relatively remote areas or areas that have confined access. The probes consist of a hollow 3/4-inch outside diameter chrome/molybdenum steel alloy tube attached to a perforated, retractable, stainless steel probe tip or disposable steel soil gas sampling tip. The probes are driven into the soil to a depth of 3 to 6 feet BGS by using a slide hammer, sledge hammer, or a pneumatic hammer attached to the top of the probe (Figures 5-28 and 5-29). Once the probe is driven to the required depth, it is retracted a minimum of 3 inches to expose the perforated probe tip to the soil, or detach the disposable probe steel tip and leave a void space for sample collection below the probe. The tubing is then purged using a vacuum pump. The negative pressure in the tubing is monitored by a vacuum gauge or flow meter to determine whether soil gas is being drawn into the tubing.

A sample is collected for Level 2 analysis using a glass 50-cc syringe equipped with a Mininert® valve and hypodermic needle. The syringe is inserted through a septum

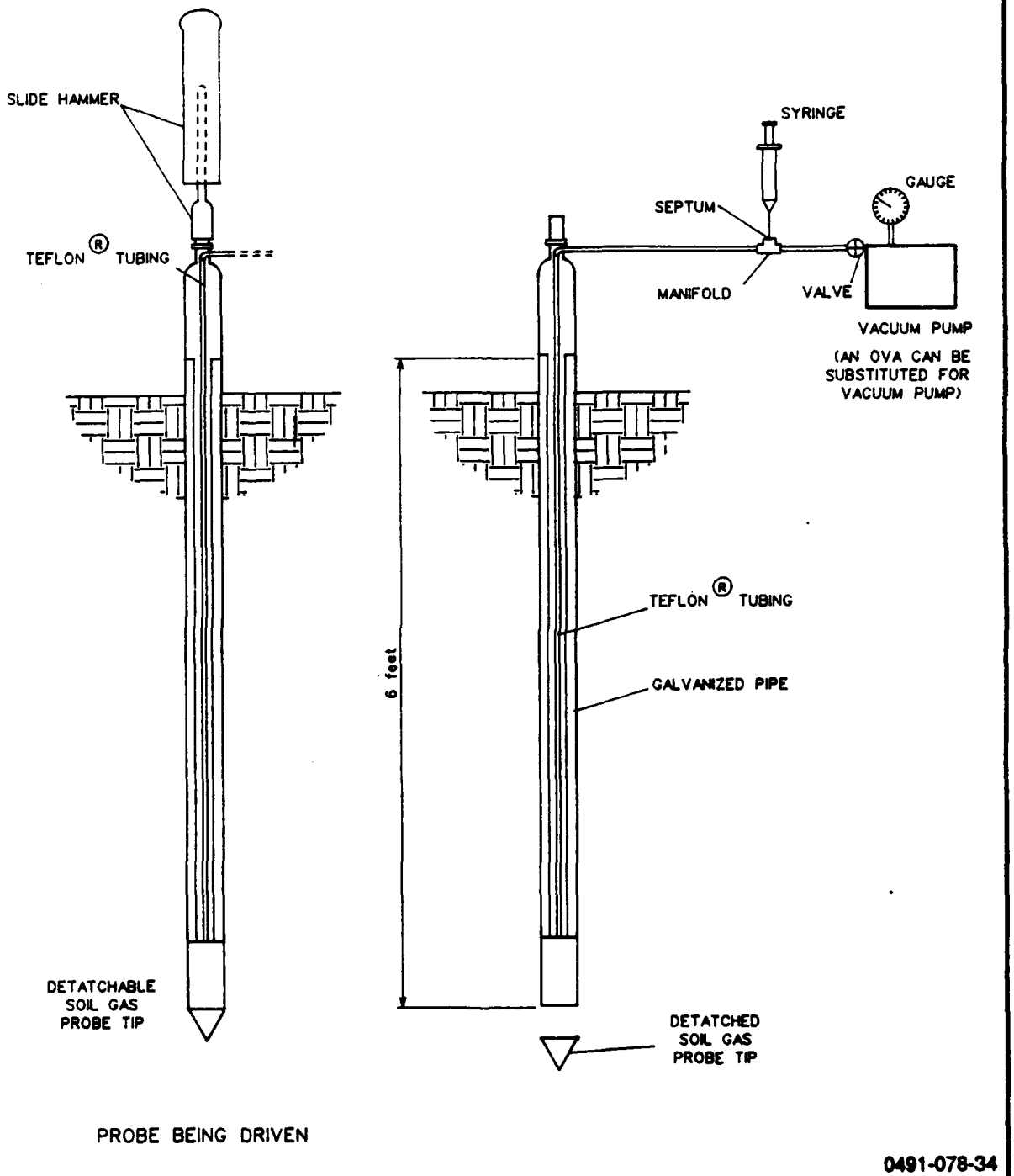


**Figure 5-28. Hand-Driven Soil Gas Probe Apparatus**

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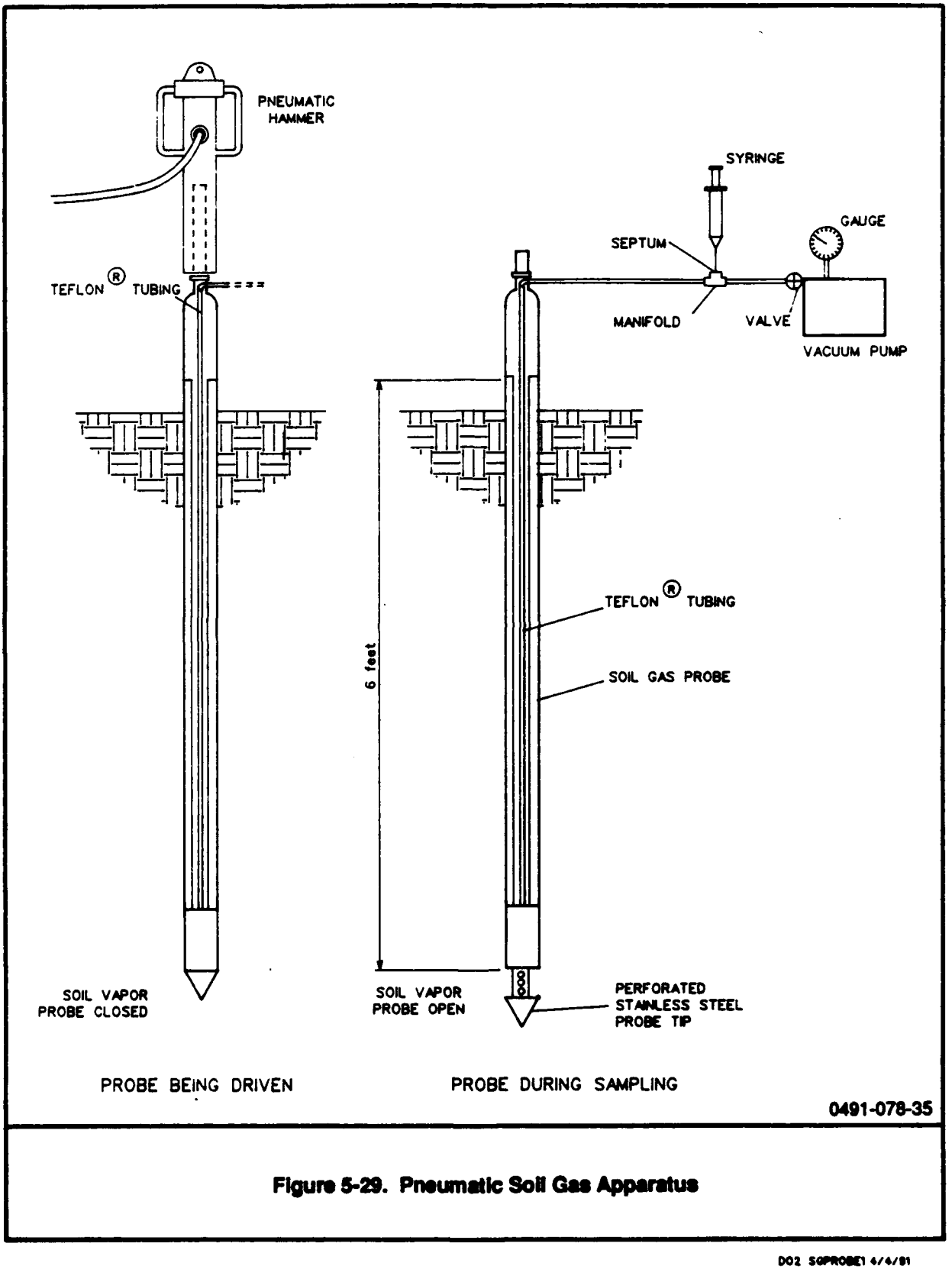



**Figure 5-29. Pneumatic Soil Gas Apparatus**

attached to a manifold, which consists of Teflon® tubing and a stainless steel tee. The soil gas samples are then brought to the field laboratory for analysis. A canister sample can also be collected for Level 3 analysis at an off-site laboratory by connecting a sampling line from the canister to the sampling manifold.

Below is a description of the sample collection Standard Operating Procedure.

A. Probe Placement

1. Measure probe location from a known reference point, locate on-site map.

2. Initiate field data sheet/chain-of-custody form following the procedures described in Section 6.0 and the field task instructions.

3. Retrieve a decontaminated soil gas probe, sampling tip, and Teflon® tubing; assemble sampling probe.

4. Purge Teflon® tubing with vacuum pump for 3 to 5 minutes. Collect an equipment blank if designated by the field task instructions and schedule.

5. Using the selected probe driving method, drive probe 3 to 6 feet BGS. If an obstruction is encountered, contact the Field Coordinator immediately.

B. Sample Extraction

1. Attach Teflon® tubing extending from probe to sampling manifold and vacuum pump intake (connect canister sampling line to manifold).

2. Retract probe 2 to 3 inches to expose sampling ports on tip.

3. Turn on vacuum pump to measure pressure (should be -10 to -29 inches of Mercury [Hg]). If the pressure is greater than -10 inches of Hg, check for leaks or improper seal around the probe. If the pressure is less than -29 inches of Hg, retract probe 2 to 3 inches while pulling a vacuum until more than -29 inches of Hg is obtained. If these conditions are not met, relocate probe because there is obstruction at the tips, and the formation is too tight to allow collection of a soil gas sample reading.

4. Purge 3 to 5 tubing volumes of soil gas. Use the graph showing purge times for tubing length and vacuum readings provided in the task instructions to determine purge requirements. Monitor flow meter to determine when completed purging.

5. Close the valve and turn off the vacuum pump.

C. Sample Collection

1. To collect a syringe sample:

   a. A hypodermic syringe equipped with a Mininert® valve and hypodermic needle is inserted through a Teflon® septum and into the Teflon® tubing connected to the probe.

   b. The syringe is purged a minimum of three times by extracting 40 to 50 milliliters of soil gas into the syringe, closing the Mininert® valve, removing the syringe from the septum, and injecting the gas into the air (the gas can also be injected into an organic vapor monitor for real-time readings of soil gas concentrations).

   c. Collect sample by withdrawing 10 to 50 cc of soil gas into the syringe, close Mininert® valve, and remove from septum. Collect second syringe sample, if required.

d. Cover syringe with foam insolation; label sample with sample control number and complete field data sheet/ chain-of-custody form following the procedures described in Section 6.0 and the field task instructions. Sign over sample data sheet/chain-of-custody form to soil gas runner or deliver sample to field lab for analysis.

2. To collect a canister sample:

a. Open vacuum gauge valve on top of canister to observe initial pressure. Record initial pressure.

b. Slightly open side valve on canister to draw sample into canister. Soil gas should be slowly drawn into the canister. Close valve slightly if a hissing sound is heard.

c. Watch canister vacuum gauge. When pressure is between negative 7 and 5 inches of Hg, close both valves. Cap sampling port on canister and record final pressure.

d. Complete field data sheet and chain-of-custody form following the procedures described in Section 6.0 and the field task instructions. Sign canister and chain-of-custody form over to soil gas runner for shipment to the laboratory for analysis.

D. Demobilization and Decontamination

1. Disconnect vacuum pump from Teflon® tubing.

2. Extract probe with jack/puller, place probe in used probe bin. The jack/puller mechanism consists of a clamp that is placed around the probe, and is connected to a hydraulic jack that is operated to extract the probe.

3. Fill hole with concrete or fill material, as designated in the SAP.

4. Purge Teflon® tubing with vacuum pump for 3 to 5 minutes. If OVM readings exceed 1 part per million by volume (ppmv), replace tubing.

5. Mark location with field tag.

6. Decontaminated all equipment following the procedures described in Section 5.10.

If the QC schedule indicates a field duplicate sample is to be collected, a second probe is placed within 10 feet from the original location using the same procedures. If nested duplicates are designated, a second sample is collected from the first probe.

**Hydraulic Method**

The contractor performs or subcontracts with a qualified soil gas sampling subcontractor for hydraulically driven soil gas probes. The contractor or the soil gas sampling subcontractor is equipped with a hydraulic probe driving capability and field laboratory instrumentation to provide a means of obtaining Level 2 soil gas analysis by real-time field analysis.

A field laboratory or mobile field laboratory consisting of a vehicle equipped with GC using FID, PID, or ECD with packed and capillary columns are used. The equipment are operated by a chemist and hydrogeologist. Soil gas sampling subcontractors are supervised by base contractor field personnel.

Soil gas samples are collected from the vadose zone through steel probes. A specialized hydraulic mechanism is used to push and withdraw the sampling probes by transferring the weight of the vehicle onto the probe. The probes are 7-foot lengths of 3/4-inch diameter steel pipe fitted with detachable steel drive points. A percussion hammer can be used to assist in driving probes through cobbles or through unusually hard soil. The vehicle has gasoline-powered generator(s) to provide the electrical power (110 volts AC) to operate the field equipment.

The probes (similar to those shown in Figures 5-28 and 5-29) are driven into the soil to a depth of 3 to 8 feet BGS by the hydraulic pusher/puller mechanism. The probe is then retracted a few inches to provide a void space and allow soil gases to enter and be purged by withdrawing two probe volumes before sampling. A gas sample is then taken using a glass syringe inserted through a section of silicon tubing. Gas samples only come into contact with steel surfaces, and are never in contact with potentially sorbing materials. A vacuum gauge monitors the negative pressure in the sampling line to ensure that there is no impedance to gas flow. The gas samples are injected into the GC for immediate analysis.

One or two 10-cc air samples are collected from each sampling probe after 1 to 4 minutes of pumping. These 10-cc samples are subsampled according to analytical requirements, and replicates are injected into the gas chromatograph for documentation of reproducibility. Where there are multiple contaminants that require different sample sizes for chromatographic analysis, more than two injections may be necessary.

Below is a description of the Standard Operating Procedure.

A. Probe Placement

1. A clean probe (pipe) is removed from the "clean" storage area.

2. The soil gas probe is placed in the pusher/puller mechanism.

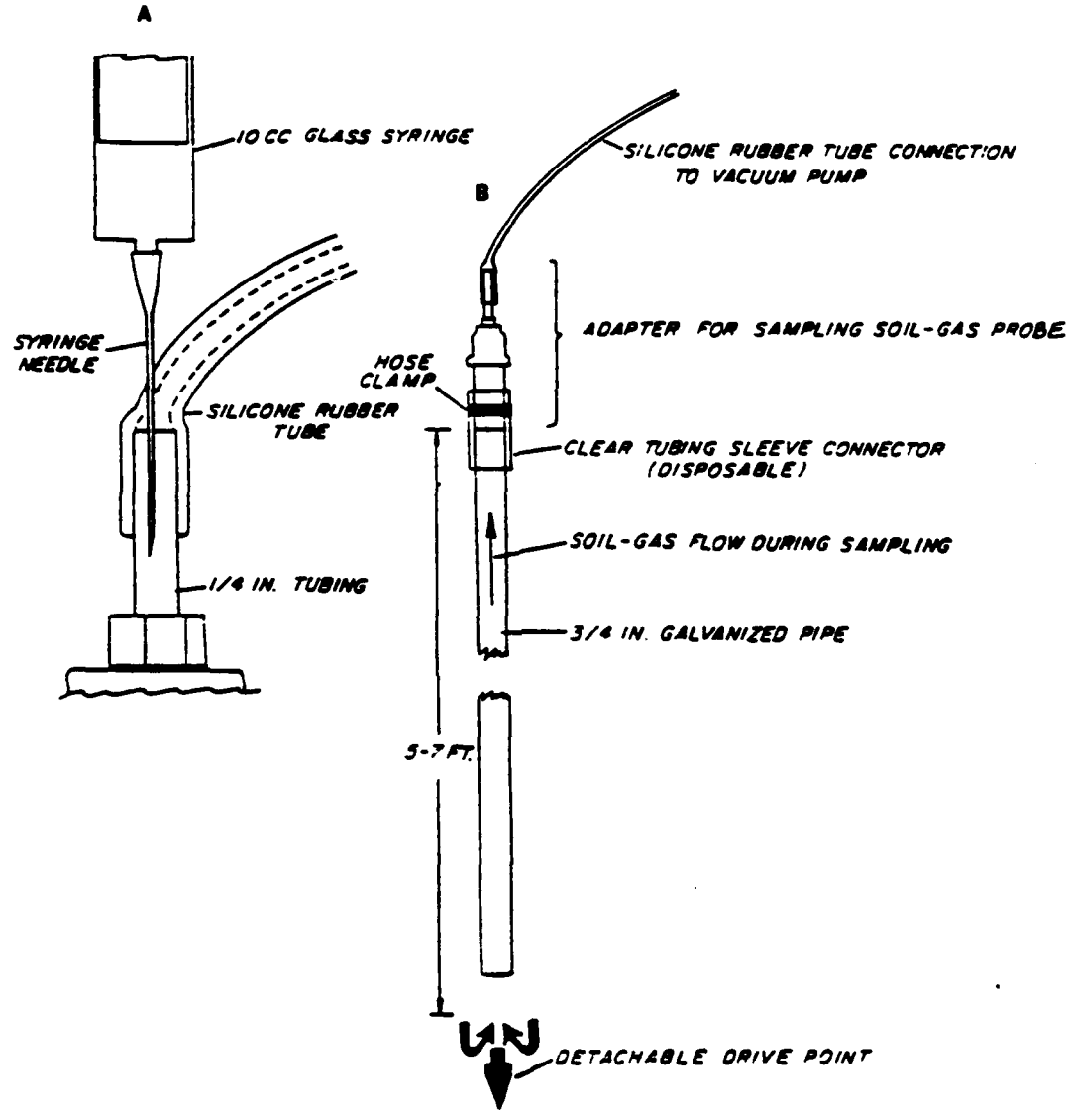
3. A drive point is attached to the bottom of the probe.

4. A driving mechanism is used to push the probe into the ground.

5. If the mechanism cannot push the probe into the ground a sufficient depth for sampling, a hydraulic hammer is used to pound the probe into the ground.

B. Sample Extraction

1. An adaptor (Figure 5-30) is put onto the top of the soil gas probe.




A. Close up of syringe soil gas sampling through evacuation line.

B. Diagram of soil gas sampling probe with adapter for sampling and evacuation of the probe after it is driven into the ground.

**Figure 5-30. Hydraulic Soil Gas Sampling Apparatus**

0392-078-38

2. A vacuum pump is hooked onto the adaptor.

3. The vacuum pump is turned on and used to evacuate soil gas.

4. Evacuation takes at least 30 seconds, but never more than 5 minutes, for samples having evacuation pressures less than 15 inches of Hg. Evacuation times are at least 1 minute, but no more than 5 minutes, for samples having evacuation pressures greater than 15 inches of Hg.

5. Gages on the vacuum pump are checked for excessive soil gas flow resistance by monitoring pressure reading (inches of Hg). A gage reading of at least 2 inches of Hg less than the maximum observed value is required to extract sufficient soil gas to collect a valid sample.

C. Sample Collection

1. With the vacuum pump running, a hypodermic syringe needle is inserted through the silicone rubber and into the stainless steel tubing of the adaptor (Figure 5-30).

2. Gas samples only come into contact with steel surfaces, never with potentially sorbing materials (i.e., tubing, hose, pump diaphragm).

3. The syringe is purged with soil gas. Without removing the syringe needle from the adaptor, a 10-cc soil gas sample is collected.

4. A second 10-cc sample is collected using the same procedure.

D. Demobilization

1. Following analysis, the vacuum pump is turned off and disconnected from the adaptor.

2. The adaptor is removed and stored with equipment to be cleaned.

3. The probe is removed from the ground and stored in the "used" probe tube.

4. The probe hole is backfilled with native soil or grout, as required in the SAP.

5. Complete all field data sheets and chain-of-custody forms.

E. Decontamination

1. Decontamination of probes, tubing, fittings, and syringes follows the procedures described in Section 5.10.

2. Equipment blanks (syringe and probe) are collected daily prior to beginning sampling to ensure that there is no contamination in the sampling system.

### 5.10.3 Deep Soil Gas Sampling Methods

The soil gas sampling methods that may be used at McClellan AFB for deep soil gas sampling includes the following:

- Downhole method;
- Split-spoon method; and
- Soil gas monitoring well method.

Downhole and split-spoon methods are used to locate areas of contamination and choose soil sampling locations during drilling. Soil gas monitoring wells are used for collecting soil gas samples similar to groundwater wells. The wells are typically installed to monitor the effectiveness of soil gas extraction systems.

**Downhole Method**

Downhole soil gas sampling is used in conjunction with hollow-stem augering and sonication drilling methods to help determine the extent of subsurface contamination and select soil samples for analysis. The probes consist of a hollow 3/4-inch outside diameter chrome/molybdenum steel alloy tube attached to a perforated, retractable, stainless steel tip. The probes are driven 2 to 3 feet beyond the auger head or drill bit using the downhole hammer or sonic unit on the rig (Figure 5-31). The probe is then retracted a minimum of 2 inches to expose the perforations on the sampling tip. The tubing is then purged using a vacuum pump by withdrawing three tubing volumes of gas. The exhaust from the vacuum pump is monitored with a PID and the negative pressure in the tubing is monitored by a vacuum gauge.

A sample is collected using a glass 50-cc syringe equipped with a Mininert® valve and hypodermic needle. The syringe is inserted through a septum attached to a manifold, which consists of Teflon® tubing and a stainless steel tee. The gas samples are then brought to the field laboratory for analysis. A canister sample can also be collected for analysis off-site by connecting a sampling line from the canister to the sampling manifold.

A gas chromatograph equipped with FID, PID, or ECD packed with capillary columns will be used. The soil gas samples are injected through a Teflon® septum into the GC using an analytical syringe. The 50-cc samples may be subsampled according to analytical requirements, and replicates are injected into the GC for documentation of reproducibility.

Below is a description of the Standard Operating Procedure.

A. Probe Placement

1. Initiate field data sheet/chain-of-custody form following the procedures described in Section 6.0 and the field task instructions.

2. Retrieve a decontaminated downhole soil gas probe, sampling tip, and Teflon® tubing; assemble probe.

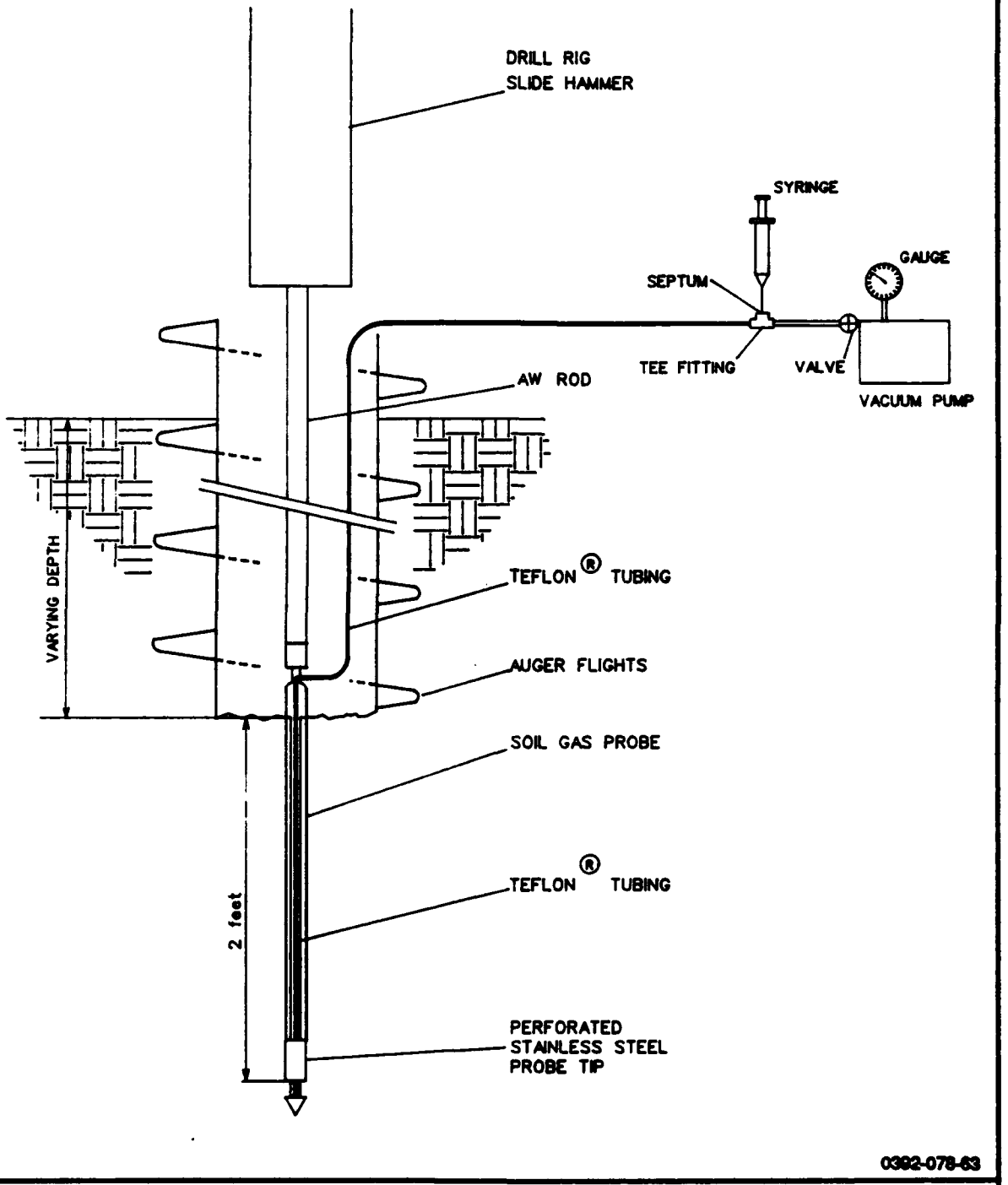



**Figure 5-31. Downhole Soil Gas Sampling Apparatus**

3. Purge Teflon® tubing with vacuum pump for 3 to 5 minutes.

4. Use downhole hammer to drive probe from 2 to 3 feet below the lead auger. If an obstruction is encountered, contact the Field Coordinator immediately.

B. Sample Extraction

1. Attach Teflon® tubing coming out of probe to sampling manifold and vacuum pump intake (connect canister sampling line to manifold).

2. Retract probe 2 to 3 inches to expose perforations on sampling tip.

3. Turn on vacuum pump, measure pressure (should be -10 to -29 inches of Hg). If the pressure is greater than -10 inches of Hg, check for leaks or improper seal around the probe. If the pressure is less than -29 inches of Hg, retract probe 2 to 3 inches while pulling a vacuum until greater than -29 inches of Hg is obtained. If these conditions are not met relocate probe.

4. Purge 3 to 5 tubing volumes of soil gas. Use the graph showing purge times for tubing length and vacuum readings provided in the task instructions to determine purge requirements. Monitor flow meter to determine when completed purging.

5. Close the valve and turn off the vacuum pump.

C. Sample Collection

1. To collect a syringe sample:

   a. A hypodermic syringe equipped with a Mininert® valve and hypodermic needle is inserted through a Teflon®

septum and into the Teflon® tubing connected to the probe.

b. The syringe is purged a minimum of three times by extracting 40 to 50 cc of soil gas into the syringe, closing the Mininert® valve, removing the syringe form the septum, and injecting the gas into the air (the gas can also be injected into an organic vapor monitor for real-time readings of the soil gas concentration).

c. Collect sample by withdrawing 10 to 50 cc of soil gas into the syringe, close Mininert® valve, and remove from septum. Collect second syringe sample, if required.

d. Cover syringe with foam insolation; label sample with sample control number and complete chain-of-custody form following the procedures in the task instructions and Section 6.0. Sign sample data sheet/chain-of-custody form over to soil gas runner or deliver sample to field lab for analysis.

2. To collect a canister sample:

a. Open vacuum gauge valve on top of canister to observe initial pressure. Record initial pressure.

b. Slightly open side valve on canister to draw sample into canister. Soil gas should be slowly drawn into the canister. Close valve slightly if a hissing sound is heard.

c. Watch canister vacuum gauge. When pressure is between negative 7 and 5 inches of Hg, close both valves. Cap sampling port on canister and record final pressure.

D. Demobilization and Decontamination

1. Disconnect vacuum pump from Teflon® tubing.

2. Extract probe, place probe in used probe bin.

3. Purge Teflon® tubing with vacuum pump for 10 to 15 minutes. If OVM readings exceed 1 ppmv, replace tubing.

4. Decontaminate all sampling equipment following the procedures described in Section 5.10.

5. Backfill the borehole with native material or grout, as specified in the SAP.

**Split-Spoon Sampling**

Soil gas concentrations may be measured and sampled during drilling operations by using split-spoon soil samples. Soil gas is measured with portable field instruments by splitting apart adjacent split-spoon samples and inserting the instrument probe between the sampling sleeves. This method yields approximate values only, but is useful as an indicator of the presence of gases. Dispersion of the gases is minimized during sampling by using a cap with an opening for the sampling instrument probe. The data are recorded on the drilling log form for the borehole.

**Soil Gas Monitoring Well Method**

Soil gas monitoring wells are used to provide long-term monitoring of soil gas concentrations in much the same manner that groundwater monitoring wells are used. The wells are usually used to monitor the effectiveness and aid in the operation of soil gas recovery systems. Monitoring well screens are installed at varying depths to provide a three-dimensional understanding of soil gas distribution.

A soil gas well differs from a groundwater well in two ways; the soil gas well is installed in the vadose zone, and the screened interval is isolated from the rest of the well

casing by a bulkhead. A schematic representation of the typical gas monitoring well is shown in Figure 5-32.

Soil gas monitoring wells are installed in a borehole drilled to the proper depth using a hollow-stem auger. The wells are fabricated using 2-inch diameter PVC casing fitted with a 3-foot screen at the bottom of the casing. The well is constructed with a sealed bulkhead positioned at the top of the screened interval to provide an enclosed interval for gas sampling. Stainless steel sample lines (1/4-inch outside diameter) extend through the bulkhead into the screened area, and are used to transport gas samples to the surface for analysis. Soil gas sampling from the well may be accomplished using canisters, syringes, real-time instruments, and sorbent tubes depending upon the level of analysis required in the SAP. Additionally, if two gas lines are installed in a well, they can be used to monitor emission rates.

Sampling of soil gas monitoring wells will be conducted as follows:

1. Initiate chain-of-custody. Record the location, number, date, time, and barometric pressure on the field data sheet.

2. Prior to each use of the sampling system (Figure 5-33), purge it with ambient air (or UHP air) and ensure that the background level as measured with the field instrument is at ambient concentration. Do not sample the well unless the ambient level is acceptable. Record the sampling system ambient concentration.

3. Check the sampling manifold for leaks by plugging the inlet and observing an attached rotameter; the flow should slowly drop to zero indicating that the system is airtight.

4. Set a Magnahelic® (not part of sampling manifold) up in a level position so it can be connected to the well. Zero the Magnahelic® gage. This gage is used to measure pressure differentials in the well that are used to calculate airflow and determine sample volumes.

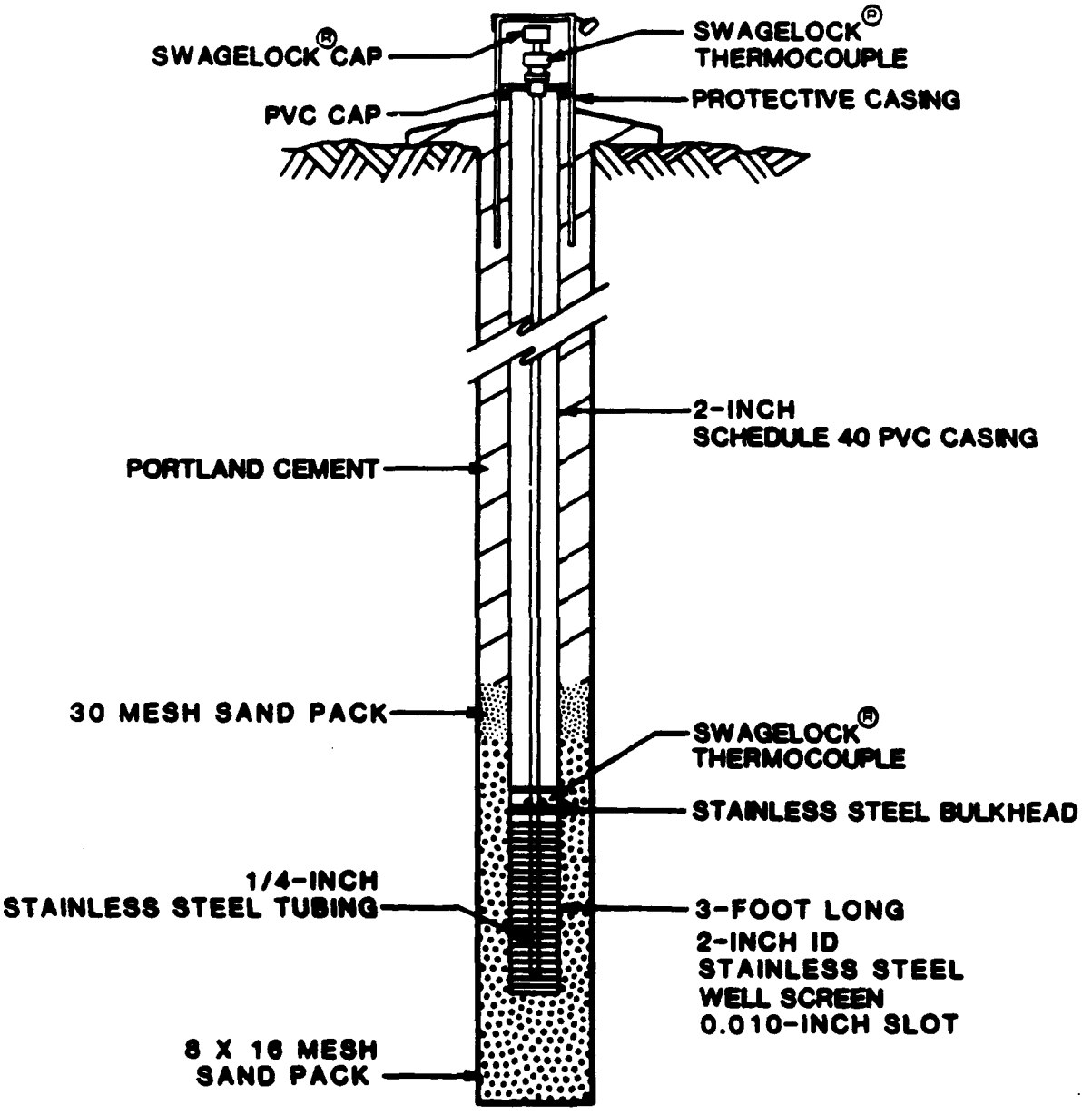



Figure 5-32. Soil Gas Monitoring Well

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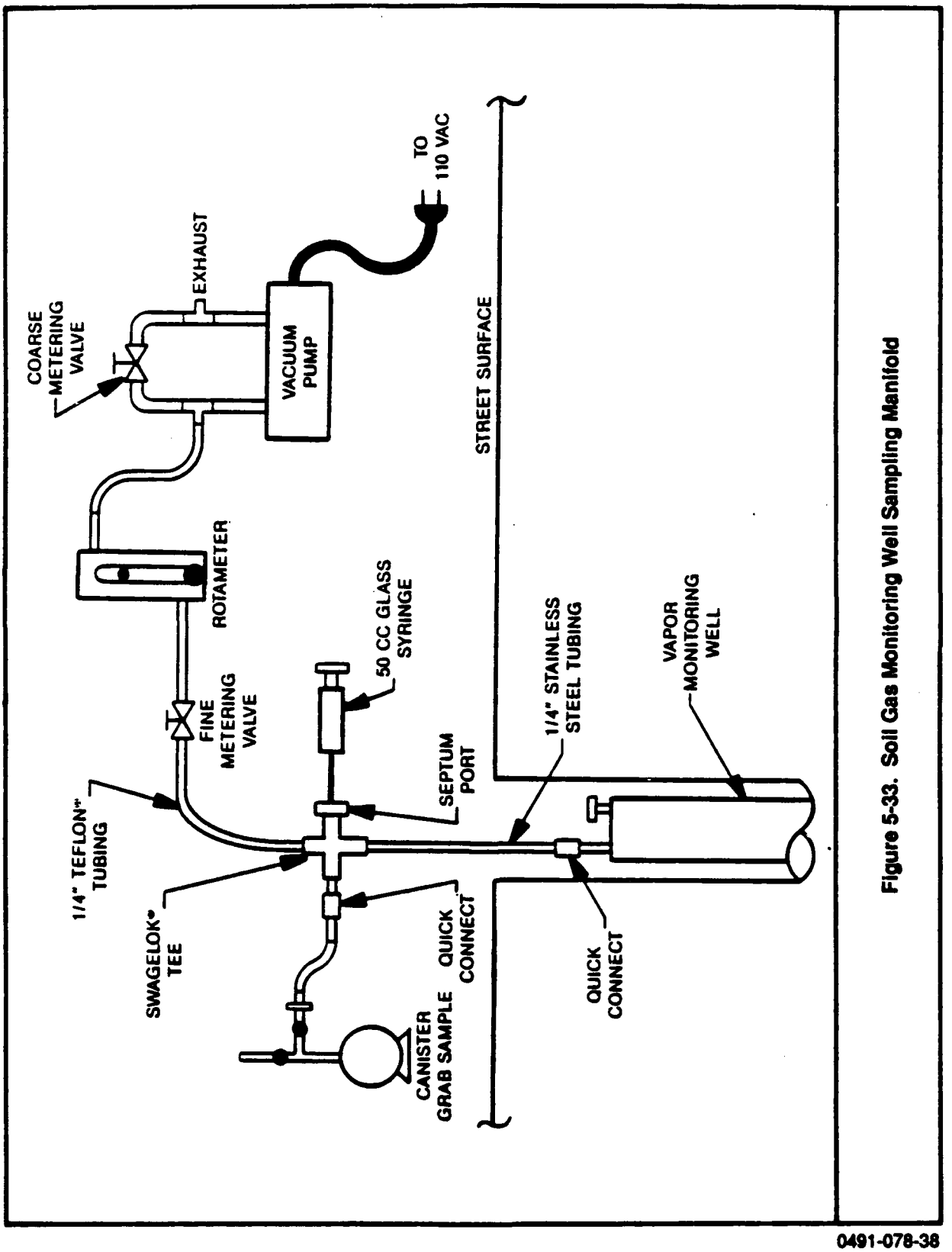


Figure 5-33. Soil Gas Monitoring Well Sampling Manifold

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5. Attach the Magnahelic® gage to the well and record the differential pressure of the well.

6. Attach the sampling system to the well. Ensure that the sampling system is not open to the atmosphere.

7. Turn on the sampling pump and adjust the well purge rate to 100 cc/min. Record the purge rate and begin an elapsed timer.

**Syringe Sampling**

8. Purge the syringe three times beginning at 8-1/2 minutes. To accomplish this, pierce the septum with the syringe and withdraw sample slowly (i.e., a 50-cc syringe should be filled every 1/2-minute period). Filling and emptying the syringe constitutes one purge cycle.

9. Purge the well of at least three well volumes at 100 cc/min purge rate and withdraw a syringe sample. The syringe sample should be withdrawn slowly as described above. When a full syringe has been collected, remove it from the sampling septum. Record the elapsed purge time at which the sample was obtained.

10. If a duplicate syringe sample is to be collected, collect both syringes simultaneously. Clearly indicate on the chain-of-custody forms for both samples that these are duplicates, so the analyst will know to perform duplicate analyses of these samples (nested duplicates: laboratory duplicate analysis of field duplicate samples).

11. Turn the sample pump off and shut the manifold valve.

**Canister Sampling**

12. If a canister sample is to be collected, connect the canister and open the valve on the canister and allow sufficient time for the sample to be collected. The vacuum flow regulator for the canister should be set nominally at $<500$ cc/min. Therefore, approximately 15 minutes will

be required to collect the sample if a 6-liter canister is used. If duplicate canisters are to be collected, they should be collected simultaneously. To accomplish this, both canisters must be connected to the sampling system with a tee, with the flow controller connected upstream of the tee. If a field blank is required, a clean canister will be filled with UHP air in the field.

13. Complete the canister sample chain-of-custody form. If duplicate canister samples are required, complete a chain-of-custody form for each canister. If the samples are duplicates, clearly indicate this on the chain-of-custody forms, so the analyst may select these samples for nested analysis.

**Sorbent Sampling**

14. If sorbent samples are to be collected, attach the sorbent sampling train and collect the sample with a sampling pump.

15. After the samples are collected, connect the field instrument to the sampling system. The instrument is connected in place of the canister for shallow wells and to the exhaust port of the sampling pump for deep wells. Open the manifold valve and, for deep wells, turn the sampling pump on to assist the instrument pump.

16. Monitor the readings from the field instrument and record the average value. If the readings vary more than 50 percent, record the range of the readings in the comments portion of the sampling data sheet.

17. Disconnect the sampling system from the well. Purge ambient air through the sampling system.

18. Complete the syringe sample(s) and canister sample(s) chain-of-custody forms.

### 5.10.4 Emission Flux Measurements

The emission flux (flow per unit area) sampling methods are direct gas emissions measurement techniques for determining the emission flux of surface and subsurface gases. The technique utilizes an isolation flux chamber of known volume to isolate a known surface area. Depending on the type of chamber used, the technique is applicable for measuring emissions at the ground surface, shallow, or deeper subsurface, or in trenches. In the chamber, ultra-pure hydrocarbon free air (sweep air) is mixed with emitted soil gases. The sweep air flow rate is controlled to prevent inducement or suppression of gas emissions. The gas/air mixture can then either be measured by a field instrument or collected in an evacuated canister, syringe, or sorbent for laboratory analysis. An emission rate is calculated from the measured concentrations of contaminants in the chamber exhaust gas, the sweep air flow rate, and the surface area of the exposed soil.

#### Surface Emission Flux Measurement

The surface emission flux sampling method is a direct surface gas emission measurement technique for determining gas emission rates to the atmosphere. The chamber can also be used in trenches or on liquid surfaces to make emission measurements. The technique utilizes the surface isolation flux chamber shown in Figure 5-34. The chamber is fabricated from a 9-inch-high by 16-inch-diameter Plexiglas® dome and a 6-inch-high by 16-inch-diameter stainless steel ring. The resulting exposed surface area is 0.13 square meters ($m^2$) and the resulting volume is approximately 31 liters. The clear Plexiglas® top allows solar heating at the soil surface, and minimizes disruption of natural surface conditions. Sweep air is introduced from a supply bottle, regulator, and rotameter through 1/4-inch Teflon® tubing leading to the instrument. The sweep air flow rate used is approximately 5 liters per minute, resulting in a 6-minute residence time within the chamber. Residence time is determined by dividing the chamber volume (v) by the sweep air flow rate (Q).

The emission flux is calculated as:

$$E_x = \frac{C_x Q}{A}$$

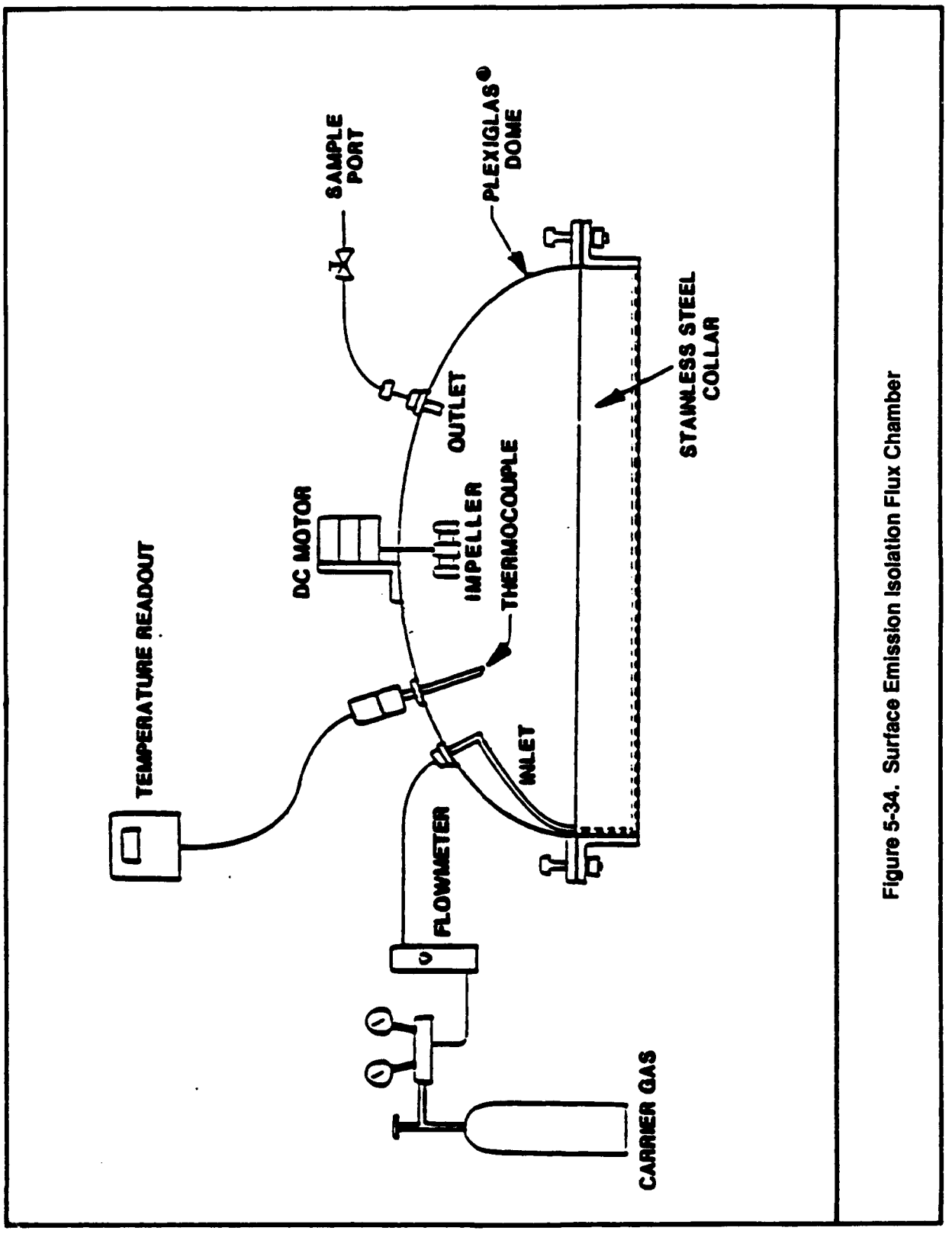


Figure 5-34. Surface Emission Isolation Flux Chamber

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where:

$E_x$ = Emission flux of species X ($\mu g/m^2$ -min)
$C_x$ = Measured concentration of species X (ppmv converted to $\mu g/m^3$)
Q = Sweep air flow rate ($m^3$/min)
A = Surface area exposed ($m^2$)

The field operation procedure for gas emissions sampling is as follows:

1. Initiate field data sheet.

2. The chamber is placed over the target surface. No attempt is made to force seal the chamber.

3. The sweep air flow and impeller are started, and the time is recorded. Check the rotameter to ensure correct sweep air flow rate (5 liters per minute).

4. Field information (location, date, site description, etc.), and the soil gas concentration resulting from peak, as well as steady-state emission are recorded on an emission measurement field worksheet (Figure 5-35). If more than one field instrument is used, each instrument should be connected to the output manifold individually, so that the instruments do not compete for flow or affect conditions within the chamber. Connect each instrument for one minute to take readings. Then remove and connect next instrument.

5. Samples are collected for analysis, if required, when steady-state conditions are reached (typically 18 to 24 minutes or 3 to 4 residence time units). Steady state is determined by measuring the temperature and organic vapor in the outlet air after each residence time. When temperature varies less than 2 degrees and the vapor measurements vary less than 1 ppmv, steady state is considered to be achieved. Event-specific criteria provided in the SAP may be used as an alternative. Remove instrument from outlet manifold and connect inlet of sampling device manifold. For syringe sampling, a septum should

ISOLATION FLUX CHAMBER

Date: _____ Time: _____ Sampler's Initials: _____

Location: _____ Well #: _____ Depth: _____

Concurrent Activity: _____

Surface Description: _____ OVA ID#: _____

| Time | Purge Air or Flowrate | Residence Time Number(T) | Temp. °C Surface Air | Gas Data ppmv Peak (Time) | Steady State | Air Sample Number |
|---|---|---|---|---|---|---|
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Comments:

Ambient: _____
Maximum Peak (Time): _____
Steady State: _____

**Figure 5-35. Emission Measurement Field Worksheet**

be installed in the sampling line upstream of the instrument, and the instrument left connected during sampling.

6. After sampling, the chamber is removed, wiped clean, and decontaminated by washing with laboratory-grade detergent and rinsing with potable and distilled water. Purge the outlet manifold by pulling ambient air through the manifold for 5 minutes or until readings are equal to ambient air, whichever is longer. Leave the chamber bottom exposed to ambient air while purging.

**Downhole Emission Flux Measurement**

The downhole emission flux (DHEF) sampling method is a direct soil gas emissions measurement technique that uses the downhole isolation flux chamber. The chamber isolates a known soil surface area at a desired depth within the hollow-stem auger. The DHEF chamber is fabricated from a 2 1/2-inch inside diameter (I.D.), 3-inch outside diameter (O.D.) Plexiglas® cylinder with a 1/4-inch thick, 3-inch diameter Plexiglas® plate cemented on the top. The total volume of the chamber is 0.643 liters. The chamber input and output lines are assembled in sections allowing the use of the shortest line needed for any individual measurement. Sweep air is introduced from a supply bottle to the bottom of the chamber in close proximity to the exposed surface. The sweep air flow rate should be set to approximately match the instrument flow rate (typically 0.6 to 1.0 liter per minute) as monitored by a calibrated rotameter, resulting in an approximately 1-minute retention time in the chamber. The calculation of emission flux for the DHEF measurement is identical to that for the surface emission flux measurement.

The field procedure for DHEF gas measurement and sampling is as follows:

1. Initiate a field data sheet. Initiate a chain-of-custody form if sampling is desired. The hole is drilled to the desired depth and the auger flight is disconnected from the drive shaft. The tip of the drive bit is removed, exposing soil at the bottom of the borehole;

2. The downhole flux chamber is lowered inside the hollow-stem auger with the sweep air flowing. After the chamber reaches the bottom of the borehole, no attempt will be made to force seal the chamber on the

exposed bottom surface. The time is recorded when the chamber is seated at the core depth and the field instrument is connected;

3. The soil gas concentration is monitored over a 15- to 20-minute period, and the peak as well as the steady-state values are recorded on a downhole emission measurement worksheet (Figure 5-35);

4. Samples will be collected for analysis, if required, when steady-state conditions are reached (typically after 15 to 20 minutes);

5. Samples are collected by disconnecting the field instrument and connecting the output manifold to the sampling device. Samples should be collected slowly at a rate of 0.64 liters per minute (1-minute retention time), except for sorbent sampling where the analytical method may specify a sampling rate. For syringe samples, a septum must be installed upstream of the instrument, and the instrument left in place during sample collection.

6. Remove the DHEF chamber and clean it by wiping with a clean towel. Decontaminate the chamber by washing with laboratory grade detergent and rinsing with potable water and distilled water. Purge the chamber and outlet line with ambient air until the next measurement using the instrument. At a minimum, purge with ambient air for 5 minutes or until the instrument reading is at ambient level, whichever is longer.

7. Complete the field data sheet, and chain-of-custody, if applicable.

### 5.10.5 Recordkeeping

Field personnel will be responsible for all recordkeeping associated with soil gas sampling. The following forms or equivalent shall be completed for each soil gas sample collected:

- Field Data Sheet/Chain-of-Custody Form (Figure 5-36);
- Direct Reading Indicator Tube Field Data Sheet (for sorbent tubes) (Figure 5-9);
- Emission Measurement Field Worksheet (Figure 5-35); and
- Daily Field Report of Subcontractor Activities (see Figure 5-6).

In addition to filling out these forms, field personnel will be responsible for keeping a daily log of events and observations in a field notebook. The following information will be recorded for soil gas samples:

- Time (military notation);
- Site number;
- Location ID;
- Vacuum pressure (negative inches of Hg);
- Evacuation time (seconds);
- Location description;
- Date;
- Samplers initials;
- Rainfall amount in last 24 hours;
- Surface temperature;
- Sampling method;
- Sample control number;

**OU B SOIL GAS SAMPLE DATA SHEET**
**SURFACE DOWNHOLE PROBE SAMPLES**

Boring ID_____ Date____/____/____

IC ID_____ Samplers____/____/____

Site ID_____ Cooler ID_____
(field cooler)

_____
(shipping cooler)

**Location Measurements:**

Reference Point_____ Surface Condition: Asphalt, Concrete, Soil, Other
(MW Number or Bldg. Number & Corner) (circle one)

_____Ft. N,S,E, or W _____Ft. N,S,E, or W Rainfall in last 24 hours? N_____ Y_____

_____Ft. N,S,E, or W _____Ft. N,S,E, or W (Approximate amount_____)
(circle direction)

Sample Method (circle one): Downhole, slide hammer hole, hand auger hole, hand driven, pneumatic, hydraulic, well

PID Readings (ppmv): Pre_____ Max_____ Post_____

Apparent Moisture (circle one): dry, moist, saturated Detector Tube N_____ Y_____

Backfill material (circle one): soil, concrete, bentonite, other_____

ORIGINAL SAMPLE Blind ID:_____

Field Sample ID:_____ Syringe Number:_____

Sampling Probe Number:_____ Sampling Time:_____

Depth of Sample:_____feet Attempts to sample:_____

Vacuum:_____(-) inch Hg Evacuation Time:_____sec.

Purge Volume:_____liter Sample Volume:_____cc

FIELD DUPLICATE SAMPLE Blind ID:_____

Field Sample ID:_____ Syringe Number:_____

Sample Volume:_____cc Sampling Time:_____

CANISTER SAMPLE Blind ID:_____

Field Sample ID:_____ Canister Number:_____

Initial Canister Vacuum:_____ Final Canister Vacuum:_____

CANISTER DUPLICATE SAMPLE Blind ID:_____

Field Sample ID:_____ Canister Number:_____

Initial Canister Vacuum:_____ Final Canister Vacuum:_____

**Figure 5-36. Soil Gas Sample Data Sheet**

- Syringe control number;
- Canister number;
- Initial canister pressure;
- Final canister pressure;
- Canister number;
- Probe number;
- Sample depth;
- Attempts to sample;
- Sampling time;
- Sample volume;
- Organic vapor analyzer readings (prior, post, maximum);
- Ground surface observation (paved/unpaved);
- Apparent moisture content (dry, moist, saturated) of the sampled zone;
- Backfill procedure and materials, if used; and
- Site map.

Only the appropriate information will be filled out for each sampling method. Additional observations should be noted in a field notebook.

# 6.0 SAMPLE CUSTODY

Sample possession during all sampling efforts must be traceable from the time of collection until the results are verified and reported. The sample custody procedures provide a mechanism for documentation of all information related to sample collection and handling to achieve this objective.

The field sampling task leader will be responsible for ensuring that the field team adheres to proper custody and documentation procedures for all sampling operations. At least one task member and a backup will be trained to perform sample control duties for each sample collection activity. Preformatted field data sheets and chain-of-custody forms will be used as the primary documentation mechanism to ensure that information pertaining to each sample is recorded. In addition, field notebooks and a master sample logbook (Master Log) will be maintained for all samples collected during each sample collection activity. An electronic version of the Master Log may also be generated.

Daily completion of chain-of-custody forms and sample packing for shipment will be performed in a designated sample control portion of the contractors' staging area. The samples will be released to the designated sample control staff member, and accompanying field data sheets and sample identification information will be submitted for entry into the electronic master log system. Samples and the completed chain-of-custody forms will be placed into coolers and sealed with custody tape. The sample control staff will release the sealed coolers to the overnight shipping courier or laboratory courier, or will deliver samples and custody documentation to on-site laboratories for immediate analysis. If immediate shipment is not planned, samples will be kept refrigerated or packed on ice in coolers in the sample control area. The security is maintained by having limited access by non-project staff to the contractors' fenced staging area located at McClellan Air Force Base (AFB).

Examples of documentation forms are presented in this section. Copies of the chain-of-custody forms and the master logs will be retained in the project files.

## 6.1 Documentation Procedures

### Field Records

Field personnel will be required to keep accurate written records of their daily activities in a bound logbook. All entries will be legible, written in waterproof ink, and contain accurate and inclusive documentation of an individual's field activities, including field data and observations, any problems encountered, and actions taken to solve the problem. The type of data recorded in the field logbook includes field measurements (pH, conductivity), ambient conditions, and any other information pertinent to sample collection.

The information to be recorded for specific sample collection activities will be designated on the data sheets or bound logbooks provided to the field personnel. Specific instructions are provided in field task instructions (including sample locations, type and frequency of quality control [QC] samples, examples of completed sample labels and data sheets, schedules, and maps) to ensure that complete, consistent, and accurate documentation will be maintained. Figure 6-1 is an example of field data sheet for groundwater sample collection. Similar data sheets are used for sampling other media.

Entry errors or changes will be crossed out with a single line, dated, and initialed by the person making the correction. Entries made by individuals other than the person to whom the logbook was assigned will be dated and initialed by the individual making the entry. Field logbooks will be available for review by the quality assurance (QA) representatives during systems audits or at any other time for QC checks. This documentation provides verification of sampling procedures. Sample documentation is maintained in files at the staging area by the sampling control staff until the field activity has been completed. The files are then transferred to the office for use, or as reference for data interpretation and reporting, and ultimately are archived in the project files.

### Sample Labels

Each sample collected may receive a sample label (Figure 6-2). Sample labels identify the sample by documenting the unique sample identification (ID) number, the sample type, analytical method, the sampler's name(s), date collected, and the preservation method used. These labels are completed with a ballpoint pen and affixed to the sample container. The labels used will be waterproof pressure-sensitive labels, or adhesive paper labels. Samples are placed in Ziploc® bags for shipment and storage to prevent loss of labels or information from wetting caused by breakage of liquid samples and melting ice.

WELL SAMPLING DATA SHEETS, PRELIMINARY GROUNDWATER OPERABLE UNIT REMEDIAL INVESTIGATION, McCLELLAN AFB

Well Number: _____

Samplers: _____/_____ Date: _____ Arrived at Well: _____
Sample Time : _____
Weather: _____ Left Well : _____
Total Time : _____

_____ Historic Information _____

Total depth: _____ Screen interval: _____

_____ Sample Specific Information _____

Depth to Water measured from: _____
Depth Below Measuring Point (feet):

Depth to Water (feet): -(_____)

( )
X _____

Well Volume (gal): ( )
X 3

Min. Purge Vol. (gal): _____

Pump Intake Setting (feet): _____

Temperature ( C): _____

pH: _____

Conductivity (umhos): _____

Purge Pump Controller Reading (psi): _____

Total Volume Purged (gal): _____

Parameters after Purging (circle): 8010 8020 8240 8070 9010 6010 7060 7421 7470 7740

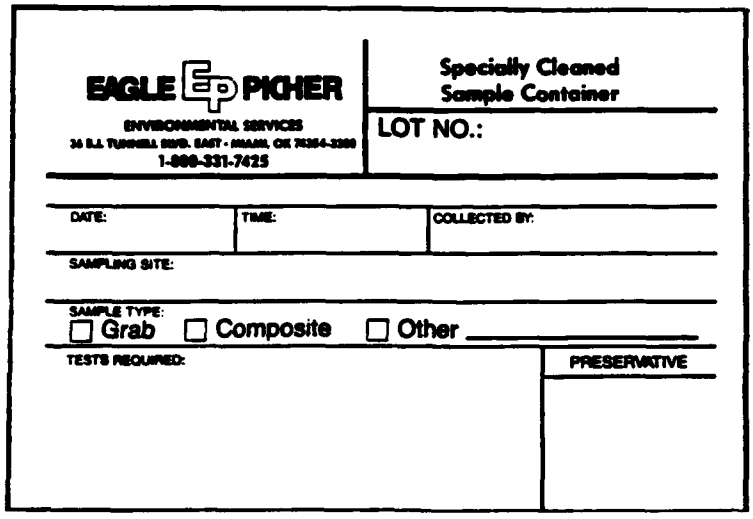
_____ QA/QC _____

| ( )Field Duplicate | | | ( )Split Samples | | | ( )Trip Blanks in custody | ( )Ambient Blanks<br>( )Equipment Blanks | | |
|---|---|---|---|---|---|---|---|---|---|
| 8010 | 8020 | 8240 | 8010 | 8020 | 8240 | | 8010 | 8020 | 8240 |
| 8070 | 9010 | 6010 | 8070 | 9010 | 6010 | | 8070 | 9010 | 6010 |
| 7060 | 7421 | 7470 | 7060 | 7421 | 7470 | | 7060 | 7421 | 7470 |
| 7740 | | | 7740 | | | | 7740 | | |
| I.D. _____ | | | I.D. _____ | | | I.D. _____ | I.D. _____ | | |

(check appropriate QA/QC and circle parameters sampled)

Comments/Field Notes: _____

**Figure 6-1. Example of Field Data Sheet**



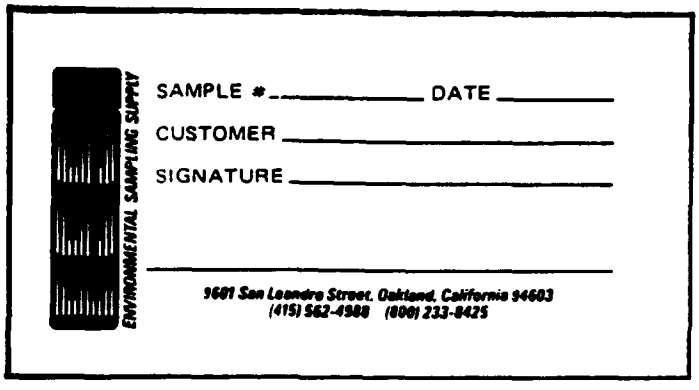




**Figure 6-2. Sample Labels**

The sample identification scheme and labeling procedures will be established for each sample collection activity. If the electronic masterlog will be used to produce pre-printed sample labels with "blind" ID numbers, the sample control staff will distribute the requested number of labels to the sampling teams daily. If labels will be manually completed, the sample identification information will be written on the labels at the sampling location. In all cases, the sample identification scheme will be documented in the field task instructions. The instructions include examples of properly completed labels to ensure that sample labeling and identification are accurate.

## 6.2 Electronically Generated Chain-of-Custody and Master Log

A field computer system may be used for efficient and consistent entry of field sample data. This system produces two necessary pieces of documentation; chain-of-custody forms and a Master Log, in a legible and uniform manner. The system also provides the McClellan AFB database direct access to field information concurrent with a sampling event by allowing the data files (e.g., for Groundwater Sampling and Analysis Program [GSAP] quarters, Operable Unit [OU] B) to be transferred electronically via modem or floppy disk. Error checking features have been built into the program (i.e., only sample locations that have been entered into the system's database will be accepted by the program). A hard copy of the Master Log may be produced at any point during the sampling event. Backup or archive copies of the files are maintained at the office. Security of the field computer is maintained using passwords, and the computer equipment is located in the staging area, which has limited access to non-project personnel.

Data for each sampling effort are entered into the system Master Log (see Figure 6-3) by the designated sample control task member to keep operator errors to a minimum. Another individual will be trained as a backup to enter data for the field program if necessary. In addition to error checking routines programmed into the electronic masterlog system, such as allowing only designated locations and checking for duplicate locations and sample IDs, the daily entries are verified manually by another task member. The printed chain-of-custody forms are verified by comparison with the field data sheets, sampling schedule, and sample containers. Audits of the sample custody procedures will be performed as part of field audits. A Standard Operating Procedure (SOP) and user's manual for operation of the system are also used for training and reference during daily operations.

MASTER LOG - 03/15/91

| Date Sampled | Location/ Sample id | Initials | Parameters Collected | # Dup | Date Shipped | Lab Name | Date Extract | Date Analyzed | Final Report | # Comp | Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 02/05/91 | MW-212 / MW-212 | SAF/MAE/ | 7470 | | | RAS-SAC | | | | | |
| 02/05/91 | MW-66 / MW-66 | MAT/RCM/ | 8010 | | | RAS-SAC | | | | | |
| 02/05/91 | MW-66 / MW-66 | MAT/RCM/ | 6010 | | | RAS-SAC | | | | | |
| 02/05/91 | MW-66 / MW-66 | MAT/RCM/ | 7060 | | | RAS-SAC | | | | | |
| 02/05/91 | MW-66 / MW-66 | MAT/RCM/ | 7421 | | | RAS-SAC | | | | | |
| 02/05/91 | MW-66 / MW-66 | MAT/RCM/ | 7740 | | | RAS-SAC | | | | | |
| 02/05/91 | MW-66 / MW-66 | MAT/RCM/ | 7470 | | | RAS-SAC | | | | | |
| 02/11/91 | MW-1045 / EB-1045 | SAF/MAT/ | 8010 | | | RAS-SAC | | | | | |
| 02/11/91 | MW-1045 / MW-1045 | MAT/SAF/ | 8010 | | | RAS-SAC | | | | | |
| 02/11/91 | MW-1045 / MW-1045 | MAT/SAF/ | 8020 | | | RAS-SAC | | | | | |

**Figure 6-3. Example of Electronic Master Log**

0491-078-45

**Generation of Chain-of-Custody Forms**

A chain-of-custody form will be generated at the end of each sampling day and will include information for all samples shipped in the same container to the laboratory. Data are entered into the database and checked against the sample labels and field documentation to verify sample identification, type of analysis, number of containers, and sample volume (i.e., 40 milliliter [mL] volatile organic analysis [VOA]). Additional information included are the date, time of collection, sampler's initials, preservative (HCl), matrix code (groundwater), and QC code that signifies to the laboratory that a QC analysis is required (i.e., M = matrix spike/matrix spike duplicate). An example of chain-of-custody form is shown in Figure 6-4. The chain-of-custody documents sample possession from the point of collection to the time of receipt by the laboratory sample control center.

**Generation of the Master Log**

The second piece of documentation created by the system is a Master Log, which contains information about sample collection, sample location, date, and analyses required, compiled in chronological order. The Master Log also contains field data for measurements taken at the sample location (purge volume, pH, etc.), laboratory custody and analytical tracking information, including sample collection conditions and parameters such as date of extraction, date analyzed, and comments which are added as the analytical results are received. A final Master Log is usually generated at the completion of the field event and used to support quality control tasks such as confirming number and location of wells sampled and analyses performed, cross checking the chain of custody information, and as a general reference. Figure 6-3 showed an example of the Master Log format.

**Manual Documentation**

The electronic Master Log system may not be available for use for every sampling event. If this occurs, the chain-of-custody forms and Master Log will be completed manually. The same information will be recorded and verified with the sample container labels and field data sheets.

## CHAIN OF CUSTODY RECORD

USE A BALLPOINT PEN AND PRESS FIRMLY
THE INSTRUCTIONS FOR FILLING OUT
THIS FORM ARE ON THE BACK

1170

| TASK OR SUB TASK (one per form): | LABORATORY NAME & ADDRESS: |
|---|---|
| CONTRACT NAME: | |
| CHARGE NUMBER: | |

| SAMPLE NUMBER | COLLECTION DATE | COLLECTION TIME | SAMPLER'S INITIALS | NUMBER OF UNITS | UNIT QUANTITY | MATRIX CODE | PRESERVATIVE | TYPE OF ANALYSIS | QC |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
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| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| | | | | | | | | | |

DO NOT WRITE IN THIS AREA

THE INFORMATION IN THIS SECTION WILL NOT BE AVAILABLE TO THE LABORATORY

| LOCATION | DEPTH | QC CODE |
|---|---|---|
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

| RELEASED BY | DATE | TIME |
|---|---|---|
| | / / | : |

COMMENTS:

SAMPLING COMMENTS:

| RECEIVED BY | DATE | TIME | RELINQUISHED BY | DATE | TIME |
|---|---|---|---|---|---|
| | / / | : | | / / | : |
| | / / | : | | / / | : |
| | / / | : | | / / | : |
| | / / | : | | / / | : |
| | / / | : | | / / | : |

| DISPOSAL CONFIRMED BY | DATE | TIME | CHAIN-OF-CUSTODY RETURNED BY | DATE | TIME |
|---|---|---|---|---|---|
| | / / | : | | / / | : |

WHITE - COORDINATOR / GOLDENROD - PROJECT DIRECTOR / PINK - SAMPLE CONTROL / YELLOW - LABORATORY / BLUE - LABORATORY RECEIPT

6/90-1

**Figure 6-4. Chain of Custody Form**

**Photographs**

When photographs, slides, or movies are taken for the purpose of visual documentation of a site or procedure, they will be numbered to correspond to the field logbook entries. If possible, a reference point (building, sign, etc.) will be included to assist in verifying the location of the photograph and provide an approximate scale. The name of the photographer, date, time, site location, and site description will be entered sequentially in the logbook as photos are taken. Photographs will be coordinated with McClellan AFB Environmental Management (EM) to ensure adherence to United States Air Force (USAF) procedures.

**Shipment**

All sample shipments will be accompanied by the chain-of-custody record, which identifies its contents. The original record will accompany the shipment, and a copy will be retained in the project file. The laboratory (or laboratories) designated to perform specific analyses will be designated in the Sampling and Analysis Plan (SAP) or Scope of Work (SOW) for the project, and shipping instructions and addresses will be documented in the field tasks instructions and sampling schedule for the sample collection activity. Sample shipment is the responsibility of the designated sample control task member.

When samples are split for duplicate analysis, a separate chain-of-custody record will be prepared. The person relinquishing the samples to the facility or agency will request the signature of a representative to acknowledge receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Received By" space. When appropriate, as in the case of overnight shipment, the custody record should contain a statement that the samples were delivered to the designated location and the date and time noted. Sample collection and shipment will be coordinated to ensure that the receiving laboratory has staff available to process the samples according to method specifications.

All shipping containers will be secured with chain-of-custody seals for transportation to the laboratory. The method of shipment, courier name(s), and other pertinent information is entered in the "Remarks" section when the samples are to be shipped (i.e., Federal Express, Express Mail, etc.) instead of hand-delivered.

## 6.3 Sample Handling Procedures

The objective of sample handling procedures is to ensure that samples arrive at the laboratory intact, at the proper temperature, and free of external contamination. All samples will be shipped to the designated laboratory via overnight carriers, according to Department of Transportation standards. Chain-of-custody procedures will be followed during transport.

Sample packaging requirements for hazardous materials requiring interstate transport are defined in the Code of Federal Regulations 40 (CFR) 49, Chapter 1, Part 171. These requirements outline in detail the proper classification and procedures for transportation of hazardous materials that will be used for transport of the samples.

When samples are required to be stored at 4°C or less, generous amounts of ice or blue ice will be packed with the samples. The ice or blue ice will be of sufficient volume and will be distributed in the coolers so that the proper storage temperature will be maintained until the samples reach the laboratory. When the samples are delivered to the laboratory they will be immediately placed in the sample control refrigerator.

The following procedures will be used to prevent bottle breakage and cross-contamination:

- All samples will be transported inside hard plastic coolers;
- All 40-mL VOA bottles will be placed in blocks of foam;
- All other glass bottles will be placed in plastic mesh sleeves to prevent glass-to-glass contact;
- All samples will be sealed in individual Ziploc® bags to prevent cross-contamination in case of breakage and prevent the labels from becoming wet;
- Plastic sample bottles and foam blocks will be used to separate glass bottles;
- Vermiculite will also be used to isolate the bottles from each other;

- The coolers will be taped shut and sealed with chain-of-custody tape to prevent accidental opening;

- Samples that are known or suspected to be highly contaminated (based on field screening data or observation) will be packaged and shipped separately from other samples;

- Ice will be contained in plastic bags to minimize leakage; and

- If samples are known or suspected to be highly contaminated, laboratory sample control will be notified, so those samples can be stored separately from less contaminated samples, minimizing the potential for contamination. Additionally, all samples are to be kept in the individually sealed inside plastic bags for storage in the laboratory sample control refrigerators.

**Laboratory Sample Control Procedures**

Each laboratory will follow sample control and login procedures. Standard operating procedures will be documented and reviewed during audits. Specific procedures/responsibilities for laboratory sample control include:

- Initial sample login and verification of samples received with the chain-of-custody form;

- Initiation of internal laboratory custody procedures;

- Verifying preservation requirements, such as temperature and pH of samples;

- Notification of project personnel if any problems or discrepancies occur;

- Proper storage procedures, including daily refrigerator temperature checks and sample security, segregation of highly contaminated samples, separate storage of samples for volatile compound analysis,

and collection of refrigerator blanks as designated in the laboratory QA manual;

- Distribution of samples to laboratory personnel; and

- Return shipment of coolers.

These procedures are referenced in the laboratory QA manuals, and are detailed in sample control SOPs.

# 7.0 CALIBRATION PROCEDURES AND FREQUENCY

Calibration procedures for laboratory and field instrumentation are performed to ensure that the instruments are operating properly, and can produce data that satisfy the objectives of the sampling program.

## 7.1 Laboratory Instrument Calibration

Laboratory instruments are calibrated according to manufacturers' specifications, and in compliance with the analytical method requirements. Detailed calibration procedures and recommended frequencies are included in the analytical standard operating procedures (SOPs). Brief descriptions of calibration procedures are included with the analytical method descriptions in Section 8.0, and the specific method requirements are also presented in Section 10.0.

## 7.2 Field Instrument Calibration

### Water Sampling Field Instruments

Field pH and conductivity meters will be routinely used for surface water and groundwater sampling. The pH meters will be calibrated daily, prior to sample analysis with a multi-point calibration and with a single-point check at each well/location. If the drift exceeds 0.1 pH units, a new multi-point calibration is necessary. The conductivity meters will also be calibrated twice daily using two potassium chloride (KCl) solutions of known conductivity. Temperature will be recorded with the conductivity and thermometers will undergo a multipoint calibration to a NIST reference thermometer on a monthly basis. Water-level meters are calibrated quarterly. One meter is verified against a known length or depth and then all meters are taken to the field and compared by measuring the depth to water for each instrument in the same well. If differences in measurements greater than 0.01 foot from the standard meter occur, the offset for each meter is noted in the calibration log and is taped to the meter for use in correcting field measurements.

### Real-Time Portable Organic Vapor Analyzers

Two types of portable field instruments, flame ionization detectors (FIDs) and photoionization detectors (PIDs), will be used to measure organic vapors in ambient air and

soil gas. Details on the use and operation of these instruments can be found under Analytical Procedures, located in Section 8.4. Instrument maintenance or backups will be available on-site or by the next sampling day if damage or failure occurs. Standard operating procedures or written instructions on instrument calibration and use are developed and provided to field personnel.

A factory calibration of the Organic Vapor Analyzer (OVA) will be conducted on a quarterly basis. Each month, a multipoint calibration check will be performed using a zero and two different concentrations of certified methane-in-air gas standards. The concentration of the standards will range from 100 to 10,000 parts per million by volume (ppmv) methane (or 100,000 ppmv, depending on the range of the instrument). The standard gas will enter the normal sampling port of the probe at atmospheric pressure by using a "tee" to vent excess pressure from the delivery line. The observed responses will be recorded on a calibration and daily quality control (QC) data form, and a plot of known concentration versus instrument response will be prepared to visually check the linearity of instrument response. The linearity of the calibration curve will be evaluated using linear regression analysis. If the correlation coefficient (r) is $\geq$ 0.9950, the instrument response will be considered linear, and the instrument ready for field use. If this criterion is not met, the calibration will be repeated (after instrument maintenance, if necessary) until r $\geq$ 0.9950. Once linearity is considered acceptable, an average response factor (RF) will be calculated based on the multipoint data.

Prior to sampling each day, the electronic calibration of the instrument will be checked and adjusted, if necessary. Hydrocarbon-free air will then be analyzed to check the zero, and the mid- and high-level calibration standards will be analyzed using methane-in-air at concentrations of 100 and 10,000 ppmv. The response factors obtained for the calibration standards analyzed before and after daily sampling must be within $\pm$20 percent of the monthly multipoint response factor; if greater deviation occurs, the data collected that day will be flagged to indicate excessive instrument drift. If the following day's calibration cannot meet the acceptance criteria, a backup instrument will be available for use. When sampling activities have been completed, another multipoint calibration test will be performed to note if any deviation exists. If the correlation coefficient at the end of sampling is less than 0.9950, the field data will be noted accordingly.

Calibration procedures for the HNu® tracer gas analyzer (one kind of PID instrumentation) will be the same as for the calibration for the OVA, except that certified

trichloroethene-in-air gas standards will be used in the range of 10 to 2,000 ppmv. The 10.2 electron volt lamp is used for calibration because it covers a wide range of detectable species.

A second variety of PID instrument that may be used in the field is the Organic Vapor Meter Model 580A and 580B (OVM). The OVM contains an internal microprocessor which performs the necessary calculations for the calibration process. The manufacturer recommends using a span gas and a zeroing gas to calibrate the instruments. The recommended span gas is isobutylene at 100 ppmv in air, and clean ambient air is the zeroing gas (a commercial source of zeroing gas should be used if any doubts about the purity of the ambient air exist). After the instrument has been exposed to the zeroing gas and the span gas, the microprocessor computes and displays a message of either successful calibration or error in calibrating; if it is the latter, the instrument is recalibrated. If the error message continues the instrument is sent for repairs and a backup instrument is used.

Another type of PID used in field events is the Photovac MicroTip™ (Total Ionizables Present) Detector®. This instrument, like the OVM, has built in microprocessing capabilities and is calibrated much the same way. A number of gases can be used to calibrate the MicroTip™, however, the manufacturer recommends isobutylene because of its medium response as a reliable means of reporting an average concentration of total ionizables present. Like the OVM, the MicroTip™ will inform the operator if the calibration has been completed successfully or if an error has been registered that invalidates the calibration. If the calibration is not successfully completed, an attempt to recalibrate the instrument will be made. If calibration is still unsuccessful, the instrument is sent out for repairs and a backup instrument is used.

**Radioactivity Meters**

If radiant energy must be measured in the field, radioactive spectrum detectors will be used. The calibration of radioactivity detectors is exacting and can pose a health hazard; for this reason, detectors are sent annually to the manufacturers to be calibrated.

**Flow Controller Calibration**

The flow controllers used with the evacuated stainless steel canisters will be calibrated prior to each sampling event using The mini-Buck® calibrator primary gas flow calibrator. This calibration is performed so that the flow into the evacuated canister is constant, as required in the Compendium of Methods for the Determination of Toxic Compounds in Ambient Air (U.S. EPA 600/4-84-041, April 1984) for whole air sampling. The calibrator is attached to the intake port of the controller by Teflon® tubing and Swagelok® connecting hardware. A charcoal tube and a bubble trap are included to ensure that soap bubbles do not enter the flow controller (Figure 7-1). The bubble trap consists of a 500-ml Erlenmeyer vacuum flask with a one-holed rubber stopper. A stainless steel outlet tube positioned 1 inch from the bottom of the flask, passes through the rubber stopper, and is attached to the Teflon® tubing. The side port of the flask is attached to The mini-Buck® calibrator by a 3-inch piece of Tygon® tubing. Hash marks are etched into the valve adjustment screw and the housing of the flow controller, so the screw adjustment can be determined and calibration settings can be reset in the field.

Ultrapure air is used throughout the calibration procedure to avoid contamination of the flow controllers. Pre-sampling calibration (i.e., in the laboratory or field preparation area) consists of three replicate measurements that provides a flow rate within 6.6 to 8.3 ml/min. The flow setting position will initially be determined by calibrating the flow controller at several settings to determine the appropriate setting for 8.3 ml/min. The mini-Buck® calibrator displays the flow in cc (ml) per minute. Recorded values are rounded to the nearest tenth of a ml/min.

Field calibration will consist of a single flow measurement; a value within $\pm 5$ percent of the initial value will be required. In-field calibration will be performed at the beginning of sampling, after 1 hour of sampling, after 20-22 hours of sampling (for 24-hour integrated samples), and at the completion of sampling. Additional measurements may be performed during sampling if the sampling stations are accessible for the full sampling interval.

Field pump calibration procedures consist of The mini-Buck® calibrator calibration to the appropriate flow rate. Field calibration is identical to the flow controller field check procedure.

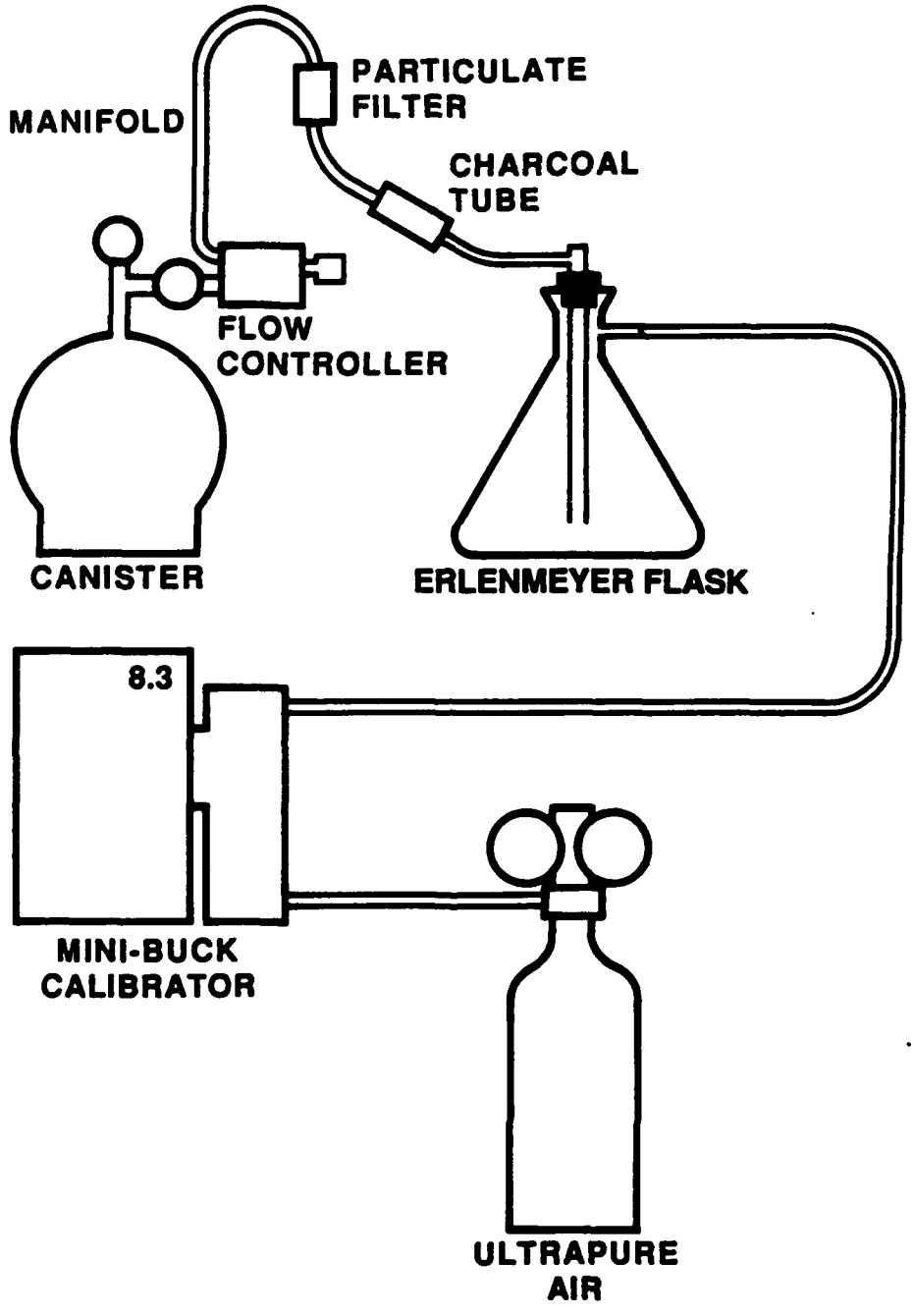


**Figure 7-1. Flow Controller Calibration Schematic.**

0288-018-6

**Hydrologic Equipment Calibration**

Transducers used for performing aquifer tests are calibrated by the vendor, and the equipment accuracy or tolerance is provided. Transducer operation is verified in the field by comparison with calibrated water-level meters at the beginning periodically and throughout the test.

Flow meters and pumps are calibrated using a container (bucket) of known volume and a stopwatch. The pump is turned on, and flow rate is measured in gallons per minute. Accuracy to $\pm 1$ gallon per minute is considered sufficient for measuring purge volumes and pumping rates.

# 8.0 ANALYTICAL PROCEDURES AND CALIBRATION

This section contains brief descriptions of calibration procedures and analytical methodology for the analysis of water, soil, and air samples that will be collected during various phases of remedial investigation (RI) and monitoring programs. The analogous water and soil methods are described together and Air Force required reporting or quantitation limits are tabulated for each method. The laboratory methods identified in this document were published by United States Environmental Protection Agency (U.S. EPA) in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW846," Third Edition, November 1986. Additional methods identified were published in "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater" (EPA-600/4-85 054), U.S. EPA Methods for Analysis of Water and Wastes (EPA-600/4-79-020, 1979), Methods for Determination of Organic Compounds in Drinking Water (EPA-600/4-88/039) in Title 22, Article 11 of the California Administrative Code, "Criteria for Identification of Hazardous and Extremely Hazardous Wastes," "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act," 40 CFR, Part 136, Federal Register 49 (209), 26 October 1984, Annual Book of ASTM Standards, Volume 4.08, the Luft Field Manual, October 1989, California, and the Compendium of Methods for the Determination of Toxic Compounds in Ambient Air (EPA-600/4-84-041, April 1984).

## Method Detection Limits and Quantitation Limits

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The laboratory performs MDL studies on an annual or quarterly basis (depending on the method) to demonstrate that it can meet or exceed the method recommended MDLs and project requirements. The U.S. EPA procedure used for establishing MDLs is described in Appendix A to Part 136 "Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.1," 40 CFR 136, 1984. This procedure consists of analyzing seven aliquots of a standard spiked at three to five times the expected MDL, which is taken through all the sample processing steps of the analytical method. The MDL is defined as three times the standard deviation of the mean value for the seven analyses.

The Air Force has established quantitation limits for IRP sites. These IRP limits will be used to assess Level 3 analytical results (off-site laboratory results), and are presented as the project quantitation or reporting limits in Tables 8-3 through 8-15 at the end of this section. In this manner, consistency in data assessment and interpretation can be achieved, and analytical results produced by different laboratories will be comparable within these limits. For some methods, IRP quantitation limits have not been established and the SW846 recommended detection limits from the reference method will be used as project quantitation limits. The project limits for each method and analytical level are evaluated to ensure they will satisfy the DQOs prior to selecting methods for a site or project.

## 8.1 Laboratory Standards and Reagents

All standards and laboratory reagents, with the exception of common laboratory solvents, must be dated upon receipt. The preparation and use of all standards must be recorded in bound laboratory notebooks that document standard traceability to U.S. EPA or NBS standards. Additional information recorded includes date of preparation, concentration, name of the preparer, lot or cylinder number, and expiration date, if applicable.

## 8.2 Extraction Methods

Extraction methods for liquid and solid matrices are briefly described in this section.

### SW1310
### Extraction Procedure Toxicity Test Method

This method is employed to determine whether a waste exhibits the characteristic of Extraction Procedure (EP) Toxicity. If a representative sample of the waste contains $>0.5$ percent solids, the solid phase of the sample is ground to pass a 9.5 mm sieve and extracted with deionized water that is maintained at a pH of $5.0 \pm 0.2$, with acetic acid. Wastes that contain $<0.5$ percent solids are not subject to extraction, but are directly analyzed. Monolithic wastes, which can be formed into a cylinder, may be evaluated using the Structural Integrity Procedure instead of being ground to pass a 9.5 mm sieve. Method detection limits for metals are presented in Table 8-15 (see end of Section 8.0).

**Waste Extraction Test - California Administrative Code**

The Waste Extraction Test (WET), described in the California Administrative Code, Title 22, Article 11, Section 66700, can be used to determine the amount of extractable substance in a waste or other material.

**Method 1311 - Toxicity Characteristic Leaching Procedure**

Method 1311 is used to determine the concentration of organic (semivolatile and volatile) and inorganic constituents that are extractable from waste or other material.

Quality control is accomplished by preparing a TCLP blank at a rate of one blank per batch or 10 percent, whichever is greater. Additional extract is prepared so that matrix spikes/matrix spike duplicates may be run at a rate of 5 percent of samples or one per waste type, whichever is greater. These quality assurance measures are in accordance with requirements set forth in EPA Method 1311, Section 8.0.

Digestion, extraction and analysis of extracts are carried out as per SW-846 protocol.

**Method SW3005**
**Acid Digestion of Aqueous Samples**

This method is an acid digestion procedure used to prepare water samples for metals analysis. The digested samples can be analyzed for total recoverable and dissolved metals determination by either flame (FLAA) or graphite furnace atomic absorption (GFAA) spectrophotometry or inductively coupled plasma emission spectroscopy (ICPES). Samples may be analyzed for the following metals:

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

For analysis of total recoverable metals, the entire sample is acidified at collection time with nitric ($HNO_3$) acid. At the time of analysis, the sample is heated with acid and reduced to a specific volume. The sample must not be boiled because antimony is volatile and easily lost. The digestate is then filtered and diluted to the desired concentration for analysis.

For analysis of dissolved metals, the samples are filtered through a 0.5um filter immediately upon collection, and acidified with nitric ($HNO_3$) acid. For analysis, the sample is heated with acid and reduced in volume. The digestate is again filtered (if necessary) and diluted to volume.

**Method SW3010**
**Acid Digestion for Metals**

Method 3010 prepares waste samples for total metals determination by flame AA and ICPES. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid.

**Method SW3020**
**Acid Digestion for Metals**

Method 3020 prepares waste samples for total metals determination by GFAA spectroscopy. The samples are vigorously digested with nitric acid followed by dilution with nitric acid.

**Method SW3050**
**Acid Digestion for Solids, Sediments, and Sludges for Metals Determinations**

Method SW3050 is applicable to the preparation of sediment, sludge, and soil samples for metals analysis by FLAA or GFAA or ICPES.

A 1 g (wet weight) sample is treated and digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with nitric or hydrochloric acid, depending on the type of analysis to be performed. When using HCl as the final refluxing acid, do not boil

because antimony is volatile and easily lost. A separate sample is dried for a total solids and/or percent moisture determination.

Some sludge samples can contain diverse matrix types, which may present specific analytical problems. Spiked samples and any relevant standard reference material should be processed to aid in determining whether Method SW3050 is applicable to a given waste.

**Method SW3060**
**Alkaline Digestion**

This method is a basic digestion used to solubilize the total concentration of hexavalent chromium in solid samples.

Method 3060 is taken from SW846, 2nd edition, and is used only as a guideline for the extraction of hexavalent chromium in a solid matrix. The digestion approach may vary according to the matrix, and sample conditions may significantly influence the recovery of hexavalent chromium.

A specific weight of sample is extracted with hot, 3 percent sodium carbonate -- 2 percent sodium hydroxide solution. The digestion solution is then heated to near boiling with constant mixing for 30 to 45 min, cooled, and filtered and diluted with deionized distilled water to a one-liter volume.

**SW3500 Series Methods**
**Organic Extraction and Sample Preparation**

The SW3500 series methods are used to quantitatively extract nonvolatile and semivolatile organic compounds from various sample matrices. Prior to analysis, a sample of a known volume or weight is solvent extracted, then dried and concentrated in a Kuderna-Danish apparatus.

**Method SW3510**
**Separatory Extraction**

Method SW3510 is designed to quantitatively extract nonvolatile and semi-volatile organic compounds from liquid samples using standard separatory funnel techniques. The sample and extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup and detection methods are described in the organic analytical method that will be used to analyze the extract.

Samples are adjusted to a specified extraction pH and extracted with the appropriate solvent for the analytical method. Methylene chloride should be employed when a solvent is not specified. Samples are extracted three times, and the combined extracts are dried with anhydrous sodium sulfate and concentrated in a Kuderna-Danish apparatus.

**Method SW3520**
**Continuous Extraction**

Method SW3520 is designed to quantitatively extract nonpurgeable organic compounds from liquid samples using a continuous extraction apparatus. The method minimizes emulsion formation, and thus improves recovery of target compounds. The sample and extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup methods and detection are described in the analytical methods.

The method is designed for extraction solvents with greater density than the sample, although continuous extraction devices are available for extraction solvents that are less dense than the sample. The analyst must demonstrate the effectiveness of any such automatic extraction device before employing it in sample extraction.

Each sample is placed into a continuous extraction apparatus adjusted to the specified extraction pH, and extracted with the appropriate solvent. Methylene chloride should be employed when a solvent is not specified. The extraction pH and solvent to be used are listed in the quantification method. Samples are extracted for 18 hours; the extract is collected, dried with anhydrous sodium sulfate, and concentrated with a Kuderna-Danish apparatus. In some cases, the sample pH is adjusted after the first extraction, and continuous extraction is carried out for an additional 18 hours to recover another class of compounds.

**Method SW3540**
**Soxhlet Extraction**

Method SW3540 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent. Extraction is accomplished by mixing the solid sample with anhydrous sodium sulfate, placing it in an extraction thimble or between two plugs of glass wool, and extracting it for 16 to 24 hours with an appropriate solvent in the Soxhlet extractor. Methylene chloride should be employed when a solvent is not specified. The extract is dried and concentrated, and then treated using a clean up method, or analyzed directly by the appropriate measurement technique.

**Method SW3550**
**Sonication Extraction**

Method SW3550 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. The sonication process ensures intimate contact of the sample matrix with the extraction solvent.

A weighed sample of the solid waste is ground, mixed with the extraction media, then dispersed into the solvent using sonication. The extract is then dried with anhydrous sodium sulfate and concentrated with a Kuderna-Danish apparatus. The resulting solution may then be cleaned up or analyzed directly using the appropriate technique. Methylene chloride is typically used as the solvent, although other solvents may be used for specific analytical applications.

**Method SW3650**
**Acid-Base Cleanup**

The Method SW3650 cleanup procedure is employed to remove interferences that prevent direct chromatographic measurement, and is based on the differential solubility between the compounds of interest and the interfering species. The method allows for a choice of solvents to optimize extraction of specific species.

An aliquot of the sample is mixed in a separatory funnel with solvent and distilled water, and the pH adjusted to 12-13 with sodium hydroxide. After extraction, the aqueous phase is collected. The solvent phase is extracted twice more with distilled water and the aqueous extracts are combined. The analyte of interest will reside in either the solvent or aqueous phase. If it is in the aqueous phase, that phase is then solvent extracted at pH 2 and the extract is concentrated in a Kuderna-Danish apparatus using a three-ball Snyder column. If the solvent phase contains the analyte of interest, only the concentration step is required.

**Method SW5030**
**Purge-and-Trap Method**

Method SW5030 is used to determine the concentration of volatile organic compounds (VOCs) in a variety of liquid and solid matrices. It is based upon a purge-and-trap gas chromatographic procedure. The method is applicable to nearly all types of samples, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, water, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complexity of matrices of solid waste samples.

A direct purge-and-trap can be performed for low concentration solid samples. If higher concentrations are expected, a portion of the solid sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the PEG, tetraglyme, or methanol solution is combined with water in a purging chamber. An inert gas is then bubbled through the solution at ambient temperature to transfer the volatile components to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. For SW8020 and SW8030, drying of the trap for four minutes under helium flow is required. The gas chromatographic column is heated to elute the components that are detected by the appropriate detector (SW8010, SW8020, SW8030).

**Percent Moisture**

Percent moisture is determined for solid samples undergoing analysis for organic analytes. The percent moisture must be known so that the analytical results can be reported on a dry weight basis (i.e., $\mu$g/kg or mg/kg). The sample is weighed, dried, and then re-weighed. Percent moisture is calculated as:

$$\frac{\text{Initial Weight} - \text{Dried Weight}}{\text{Initial Weight}} \times 100$$

The moisture content is used to calculate results for dry weight concentrations by:

$$\frac{\text{Result}}{1 - \%\ \text{Moisture}}$$

## 8.3 Organic and Inorganic Analytical Methods for Water and Soil Samples

These subsections contain brief descriptions of the currently anticipated analytical methods that will be used for samples collected during McClellan AFB sampling and analysis programs. The Project Reporting Limits are based on the reference methods and/or project requirements (DQOs) and are presented in tables at the end of this section.

**U.S. EPA Method 120.1**
**Conductance**

Sample conductance is measured on site according to U.S. EPA Method 120.1. Standard field meters are used, and the electrode is rinsed with sample prior to measuring conductance; temperature is also reported. The meters are standardized daily using KCl solutions of known conductance with an allowance of $\pm 5$ percent of true value. Reporting limits are presented in Table 8-3.

**U.S. EPA Method 150.1/SW9045**
**pH**

Field and laboratory pH measurements may be taken for water samples; the pH of soil samples is measured in the laboratory. All measurements are determined electrometrically using either a glass electrode in combination with a reference potential, or a combination electrode. The meters are calibrated daily using a minimum of two buffer solutions. The calibration readings must be within 0.05 units of the known buffer pH.

**U.S. EPA Method 160.1**
**Total Dissolved Solids**

Total dissolved solids in water are determined using U.S. EPA Method 160.1. In this gravimetric method, the sample is filtered, transferred to a pre-weighed evaporating dish, and evaporated to dryness at 180°C. The sample is cooled, and then weighed; the drying cycle is repeated until a constant weight is obtained. Reporting limits are presented in Table 8-3.

**U.S. EPA Method 170.1**
**Temperature**

On-site water temperature is measured using U.S. EPA Method 170.1. A standard mercury thermometer is rinsed twice with sample prior to recording the temperature. Thermometer calibration is checked against an NBS traceable standard thermometer.

**U.S. EPA Method 300.0**
**Anions by Ion Chromatography**

Water samples are analyzed for fluoride, chloride, nitrate, and sulfate anions by ion chromatography using U.S. EPA Method 300.0. Ion chromatography is a rapid method for separating and analyzing complex solutions of ionic species. The technique employs a carbonate/bicarbonate eluent and ion exchange resins to separate individual ions, and a suppressor column to remove the eluent ions. The detection and quantitation of the

anions is performed conductimetrically. Reporting limits for this method are presented in Table 8-3.

A multipoint calibration curve (minimum of five points) is prepared daily by analyzing standard solutions containing the anions of interest. The calibration curve is verified by analyzing quality control check samples. A calibration curve is acceptable if the correlation coefficient for the anion is greater than, or equal to, 0.995 and recoveries for a QC check samples are in the range of 90 to 110 percent recovery.

**U.S. EPA Method 310.1**
**Alkalinity**

Water samples are analyzed for alkalinity using U.S. EPA Method 310.1. An unaltered sample is titrated to an end point of pH 4.5 using a standard (0.02N) hydrochloric or sulfuric acid. This method is applicable for all concentration ranges; however, the sample aliquot should be adjusted so that the final titrated volume does not exceed 50 ml. Reporting limits are presented in Table 8-3.

Calibration of the pH meter consists of three point calibration using 10.0, 7.0, and 4.0 pH buffers. The acid titrant is standardized against reagent sodium carbonate standard solution. Duplicate determinations should agree within 20 percent. One QC check sample from an independent source is analyzed per batch and at a frequency of 10 percent. Acceptable error is $\pm 20$ percent.

**U.S. EPA Method 353.2**
**Nitrate-Nitrite**

Water samples are analyzed for nitrate and nitrite using Method EPA 353.2. A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye, which is measured colorimetrically. The typical minimum detection limit is 0.01 $\mu$g/L. Separate, rather than combined nitrate-nitrite, values can be obtained by running the procedure first with, and then without, the reduction step. Reporting limits are presented in Table 8-3.

Analysis should be made as soon as possible. When analysis can be done within 24 hours, samples should be preserved by refrigeration at 4°C. When samples must be stored for more than 24 hours, they should be preserved with 2 ml of sulfuric acid ($H_2SO_4$) per liter and refrigerated.

Calibration--A calibration curve will be generated daily using a reagent blank and five or more standards. The correlation coefficient must exceed 0.995 for the calibration equation. A quality control check sample will be analyzed for every 10 samples; recovery must be within ±15 percent of the expected value.

**Method SW6010**
**Trace Elements (Metals) by ICPES for Water and Soil**

Samples are analyzed for trace elements or metals, using SW6010 for water and soils. Analysis for most metals requires digestion of the sample by nitric acid. This digestion is performed as U.S. EPA Method 3005 for water or U.S. EPA Method 3050 for soil. Following digestion, the trace elements are simultaneously or sequentially determined using ICPES. The elements and corresponding reporting limits for this method are listed in Table 8-4.

Calibration--Detailed calibration procedures for ICPES systems are described in SW846, Third Edition. A response factor is calculated daily for each metal based on three exposures of a calibration standard and calibration blank. The RF is calculated and stored in the ICPES computer. Following calibration, a mid-level calibration check sample is analyzed; agreement between the measured value and the expected value must be within 5 percent for analyses to proceed. Calibration is verified by analyzing a QC check standard (prepared independently of calibration standards) every 10 samples; agreement within ±5 percent of the expected value is required for all metals analyzed by ICPES.

**Graphite Furnace Atomic Absorption Metals Analyses, SW7060 for Arsenic, SW7421 for Lead, and SW7740 for Selenium**

Graphite furnace AA spectrometry is used to measure concentrations of Arsenic (As), Lead (Pb), and Selenium (Se) in water and soil samples. The samples are extracted using SW3005 or SW3050 as appropriate. Discrete aliquots of sample extract are

deposited in a graphite tube furnace in microliter amounts. The graphite tube is resistively heated by an electrical current. The sample solution is dried and charred to remove sample matrix components, and then atomized at temperatures sufficient to vaporize the element of interest. Matrix modification is used to eliminate interference effects, and may also enhance the vaporization efficiency and allow lower detection limits. This method usually has a linear analysis range at the ppb or sub-ppb level. Reporting limits for these analyses are presented in Table 8-4.

Calibration--The calibration procedures for the graphite furnace and cold vapor AAS systems are described in the respective method in SW846, Third Edition. A multi-point calibration curve is generated daily for each element using a calibration blank and at least three upscale standards. The correlation coefficient for the linear regression equation must exceed 0.995 to be acceptable. Calibration will be verified every 10 samples by analyzing a QC check sample and calibration blank. Agreement within $\pm 10$ percent of the expected value is required; otherwise, a new calibration curve must be generated.

**Method SW7470-7471**
**Mercury - Manual Cold-Vapor Technique**

Water and soil samples are analyzed for mercury using SW7470 and SW7471, respectively. This method is a cold-vapor flameless AA technique based on the absorption of radiation by mercury vapor. Mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an AA spectrophotometer. Mercury concentration is measured as a function of absorbance. The reporting limit for this method is included in Table 8-4.

**Method SW7196**
**Hexavalent Chromium (Colorimetric)**

Dissolved hexavalent chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, may be determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet color of unknown composition is produced. The reaction is very sensitive, the absorbency index per gram atom of chromium being about 40,000 at 540nm. Addition of an excess of diphenylcarbazide yields the red-violet product, and its absorbance is measured

photometrically at 540 nm. The reporting limit for this method is presented in Table 8-3. A calibration curve is generated daily, using a blank and five concentrations of standards. The correlation coefficient must be 0.995 or greater to be acceptable. Quality control check samples are analyzed daily or after every 15 samples, whichever is greater. The response must be within ±15 percent of the predicted value or laboratory-established criteria may be used.

**Method SW9010 and SW9012**
**Total and Amenable Cyanide**

Water and waste samples will be analyzed for total cyanide using SW9010 or SW9012. These methods are equivalent in principle of analysis; SW9010 is a manual procedure and SW9012 is an automated procedure.

Both methods are used to determine the concentration of inorganic cyanide in an aqueous waste, leachate, or soils. The methods detect inorganic cyanides that are present as either sample soluble salts or complex radicals. The procedure is sensitive to approximately 0.02 mg/L and is used when cyanide concentrations are below 1 mg/L. It is used to determine values for both total cyanide and cyanide amendable to chlorination. The cyanide, as hydrocyanic acid (HCN), is released by refluxing the sample with a strong acid and catalyst and distillation of the HCN into an absorber-scrubber containing sodium hydroxide solution. Total cyanide in soils is determined after acidification of the soil with sulfuric acid, distillation, and mixing in sodium hydroxide scrubber. The cyanide ion in the absorbing solution is then determined by spectrophotometry for Method SW9010 and by automated colorimetry for Method SW9012. Reporting limits for cyanide are presented in Table 8-3.

Calibration--A daily calibration curve is prepared using a blank and a minimum of six standards, with a correlation coefficient greater than 0.995. A high and a low standard are distilled and compared with similar values on the curve to ensure that the distillation technique is reliable. The distilled standards must agree within ±15 percent of the undistilled standards. Quality control check samples are analyzed daily or every 15 samples (whichever is greater), and the response must be within ±15 percent of the predicted response or within laboratory-established limits.

**CADHS/HML Method 338**
**Organic Lead Compounds**

Organic lead compounds in soil and water samples are analyzed using the California Department of Health Services Hazardous Materials Laboratory (CADHS/HML) Method 338. The typical minimum detection limit is 0.8 milligrams per kilogram (mg/kg) for soil samples and 0.2 milligrams per liter (mg/L) for water samples. Organic lead compounds are extracted from the water or soil samples with xylene. The organic lead in the extract is reacted with iodine and tri-capryl methyl ammonium chloride. Methylisobutylketone is then added. The concentration of lead in the solution is determined by atomic absorption spectrometry.

Calibration--A multipoint calibration curve is generated using a blank and at least three calibration standards of organic lead containing xylene, iodine, tri-capryl methyl ammonium chloride, and methylisobutylketone. The correlation coefficient for the linear regression equation must exceed 0.995 to be acceptable. A quality control check sample of leaded gasoline is analyzed before and after samples are run. Agreement within $\pm 20$ percent of the expected value is required; otherwise, a new calibration curve must be generated.

**U.S. EPA Method 504**
**1,2-Dibromoethane**

This method is applicable to the determination of 1,2-dibromoethane (EDB) and 1,2-dibromo-3-chloropropane (DBCP) in finished drinking water and unfinished groundwater. Thirty-five milliliters of sample is extracted with 2 milliliters of hexane. Two microliters of the extract is then injected into a GC equipped with a linearized ECD for separation and analysis. Aqueous calibration standards are extracted and analyzed in an identical manner as the samples in order to compensate for possible extraction losses. The reporting limits for EDB are presented in Table 8-15.

Calibration--At least three calibration standards are needed. One should contain EDB and DBCP at a concentration near but greater than, the MDL for each compound; the other two should be concentrations that bracket the range expected in samples.

**U.S. EPA Method 607**
**Nitrosamines**

Nitrosamines are measured in soil and water using a modified U.S. EPA Method 607. The sample is extracted with methylene chloride using either a sonication extraction method for soils or a separator burner extraction for water samples. The methylene chloride extract is washed with dilute hydrochloric acid to remove free amines, dried, and concentrated to a volume of 2.0 milliliters. The extract is transferred to a pre-rinsed florisil and anhydrous sodium sulfate column. Diphenylamine is removed by eluting the column with an ethyl ether/pentane mixture. Nitrosamines are then eluted with an acetone/ethyl ether mixture and methanol is added to the eluate. Following concentration, the extract is analyzed by GC with a nitrogen-phosphorous detector. The expected method detection limit for n-nitrosodiphenylamine in water is 0.8 micrograms per liter ($\mu$g/L).

Calibration--A calibration curve is prepared with three calibration standards. The RSD between the instrument response for the standards must be less than 10 percent to be acceptable. A mid-level standard is run daily. If the response factor for that daily standard differs from the initial calibration response factor by more than $\pm$15 percent, a new calibration curve must be prepared.

**Method SW8010**
**Halogenated Volatile Organics**

Halogenated volatile organics in water and soil samples are analyzed using Method SW8010. This method is a purge-and-trap (SW5030) gas chromatographic method. An inert gas is bubbled through a water matrix to transfer the volatile halocarbons from the liquid to the vapor phase. The volatile compounds are removed from the inert gas by passing it through a sorbent trap, which is then backflushed onto a gas chromatographic column with an electrolytic conductivity detector to separate and quantify the compounds of interest. Soil samples are analyzed by direct purge-and-trap (for low-level samples) or by extraction of the sample with 5 mls of methanol and diluting a minimum of 1:50 in reagent water. The reporting limits for the organic analytes are presented in Table 8-5.

This method provides for the use of a second gas chromatographic column of dissimilar phase to resolve compounds of interest from interferences that may occur. When

second column analysis is performed, retention times for the analyte must match those established for each column or the chromatographic peaks are considered interferences and the analyte is not considered to be present in the sample. Specifications require confirmation for detected analytes, as described in Section 10.0.

Calibration--Calibration standards at five concentration levels are prepared in reagent water by dilution of stock standards. The average calibration factor is acceptable if the RSD for the calibration factors at each level does not exceed 20 percent, or a linear regression correlation coefficient is 0.995 or greater, otherwise a new calibration is performed. Daily calibration checks are conducted and must be within $\pm 15$ percent, or a new calibration curve must be prepared for analytes that exceeded that criterion.

**SW8015 Modified**
**Volatile and Extractable Total Petroleum Hydrocarbons (TPH)**

Volatile petroleum hydrocarbon components, gasoline, jet fuel, and other low molecular weight petroleum products, and volatile aromatics, benzene, toluene, xylenes, and ethyl benzene (BTXE), are analyzed by the direct purge-and-trap technique described in Method SW5030, followed by a modified approach to SW8020 and SW8015. Extractable TPH components are analyzed by extraction by Method SW3520 or SW3550, followed by GC with an FID. Additional information regarding analysis of TPH components is provided in the California LUFT Field Manual, 10/89.

For volatile TPH, either 5 mL of water or 5 grams of soil/sludge is placed in the purge-and-trap sparge vessel. In the case of soil/sludge, 5 mLs of reagent grade organic-free water is also added to the sparger. Analysis is conducted using a gas chromatograph equipped with an inlet splitter, two fused silica megabore columns, and FID and PID detectors. The BTXE components are confirmed on a second GC column of dissimilar phase and retention characteristics if there is no petroleum pattern present. Reporting limits for BTXE, volatile TPH, and extractable TPH are provided in Table 8-6.

Extractable TPH components, kerosene, diesel, motor oil, and other high molecular weight extractable petroleum products, are analyzed via Method SW3520 (continuous liquid/liquid extraction) for water based matrices or SW3550 (sonication extraction) for soil/sludge matrices. Hexane is used as the extracting solvent. One liter of

water or 10 grams of soil/sludge are extracted and concentrated to a volume of 5 mls. Analysis is accomplished on a gas chromatograph equipped with a capillary or megabore column and FID.

Identification and quantitation of TPH components (except BTXE) is based on pattern recognition techniques and requires a greater degree of analytical judgement than other GC methods. The TPH chromatograms consist of groups of peaks that have a general shape or pattern and fall within noted a carbon range (i.e., number of carbon atoms in the molecule). Selected TPH components are used to calibrate the instruments, and the resulting patterns and carbon ranges are used to compare the sample results. These components are usually reported as a TPH range, with components that matched the calibrated patterns identified. Often, unknown/uncalibrated hydrocarbons are encountered.

Calibration--Calibration of the gas chromatograph is achieved via the external standard technique using a minimum of five concentration levels. The average calibration factor is used if the RSD for the calibration factors at each level does not exceed 20 percent, otherwise linear regression is used if the correlation coefficient is 0.995 or greater. The working calibration factor is verified on each working day by analyzing a single-point calibration standard. If the response for the single-point calibration standard varies from the predicted response by more than $\pm 15$ percent, a new multipoint calibration factor will be generated.

**Method SW8020**
**Aromatic Volatile Organics**

Aromatic volatile organics in water and soil samples are analyzed using Method SW8020. This method, also known as BTX since the compounds of interest include benzene, toluene, and xylene, is a purge-and-trap gas chromatographic method. An inert gas is bubbled through a water matrix to transfer the volatile aromatic hydrocarbons from the liquid to the vapor phase. The aromatics are removed from the inert gas by passing it through a sorbent trap, which is then backflushed onto a gas chromatographic column with a photoionization detector to separate and quantify the compounds of interest. Soil samples are analyzed via extraction with methanol and diluted a minimum of 1:50 in reagent water. Reporting limits for Method SW8020 are presented in Table 8-7.

This method provides for a second chromatographic column of dissimilar phase to resolve compounds of interest from interferences that may occur. When second column analysis is performed for confirmation, retention times for the analyte must match those established for each column, or the chromatographic peaks are considered interferences and the analyte is not considered to be present in the sample. Air Force specifications require confirmation for detected analytes, as described in Section 10.0.

Calibration--Calibration standards at five concentration levels are prepared in reagent water by dilution of stock standards. The average calibration factor is used if the RSD for the calibration factors at each level does not exceed 20 percent otherwise, linear regression is used if the correlation co-efficient is 0.995 or greater. Daily calibration checks are conducted, and the instrument performance is considered acceptable if the daily response falls within ±15 percent of the predicted response.

**Method SW8080**
**Organochlorine Pesticides and PCBs**

Organochlorine pesticides and polychlorinated biphenyls (PCBs) in water and soil samples are analyzed using Method SW8080. This analytical method involves extraction of the sample with methylene chloride, followed by exchange to hexane and concentration of the extract. The pesticides and PCBs are separated and quantified by gas chromatography using electron capture detection. Both neat and diluted liquids may be analyzed by direct injection on to the chromatographic column. Reporting limits for this method are presented in Table 8-8.

Calibration--The external standard quantitation discussed in the method is used to quantitate all pesticides/PCBs. The retention time window is calculated for each pesticide/ PCB after adjusting the GC operating conditions for the routine retention times of 4,4'-DDT. The GC/ECD is initially calibrated at a minimum of five concentrations. The average calibration factor is acceptable if the RSD for the calibration factors at each level does not exceed 20 percent or linear regression may be used if the correlation coefficient is 0.995 or greater. Concentration of the components in a standard varies depending on the response of the compounds in the analytical system. Breakdown of 4,4'-DDT and endrin is also monitored. Breakdown may not exceed 20 percent. A daily single-point calibration check must agree within ±15 percent of the multipoint response or the instrument is recalibrated.

**Method SW8140**
**Organophosphorus Pesticides**

Method 8140 is a gas chromatographic (GC) method used to determine the concentrations of various organophosphorus pesticides. Prior to analysis, appropriate sample extraction techniques are used. Both neat and diluted organic liquids are analyzed by direct injection. A 2- to 5-$\mu$L aliquot of the extract is injected into a gas chromatograph, and compounds in the GC effluent are detected with a flame photometric detector. Any compounds tentatively identified in the primary analysis are confirmed on a second GC column. Reporting limits for these pesticides are presented in Table 8-9.

Calibration--The external standard quantitation method is used to quantitate all pesticides. The retention time window is calculated for each pesticide after adjusting the GC operating conditions for the routine retention times of each parameter of interest. The instrument is calibrated at a minimum of five concentrations. The average calibration factor is acceptable if the RSD for the calibration factors at each level does not exceed 20 percent or linear regression may be used if the correlation coefficient is 0.995 or greater. Concentration of the components in a standard varies depending on the response of the compounds in the analytical system. A daily single-point calibration check must agree with $\pm$15 percent of the initial multipoint response or the instrument is recalibrated.

**Method SW8150**
**Chlorinated Herbicides**

Method 8150 is a gas chromatographic (GC) method for determining selected chlorinated acid herbicides. Spiked samples are used to verify the applicability of the chosen extraction technique to each new sample type. The esters are hydrolyzed with potassium hydroxide, and extraneous organic material is removed by a solvent wash. After acidification, the acids are extracted with solvent and converted to their methyl esters using diazomethane as the derivatizing agent. After excess reagent is removed, the esters are determined by gas chromatography employing an electron capture detector. The results are reported as the acid equivalents. Any compounds tentatively identified in the primary analysis are confirmed on a second GC column. Reporting limits for herbicides are presented in Tables 8-10.

Calibration--The external standard quantitation method is used to quantitate all herbicides. The retention time window is calculated for each herbicide after adjusting the GC operating conditions for the routine retention times of each parameter of interest. The GC/ECD is initially calibrated at a minimum of five concentrations. The average calibration factor is acceptable if the RSD for the calibration factors at each level does not exceed 20 percent or linear regression may be used if the correlation coefficient is 0.995 or greater. Concentration of the components in a standard will vary depending on the response of the compounds in the analytical system. A daily single-point calibration check must agree within ±15 percent of the multipoint response or the instrument is recalibrated.

**Method SW8240**
**Volatile Organics**

Volatile, or purgeable, organics in water and soil samples are analyzed using Method SW8240. This method uses a purge-and-trap GC/MS technique. An inert gas is bubbled through the water samples, or a soil-water slurry for soil samples, to transfer the purgeable organic compounds from the liquid to vapor phase. Soil samples with higher contaminant levels are extracted using methanol before purging. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is backflushed and heated to desorb the purgeable organics onto a gas chromatographic column where they are separated and then detected with a mass spectrometer. The species detected and reporting limits for this method are listed in Table 8-11.

Alternate methods for determination of these species are the GC methods SW8010 and SW8020, which generally yield slightly lower detection limits and have lower costs. Nonspecified compounds cannot be determined using GC. Acrolein and acrylonitrile can be determined using SW8240 if specifically requested.

Calibration--The mass spectrometer is tuned daily to give an acceptable spectrum for bromofluorobenzene (BFB). Relative ion abundance criteria for BFB are given in SW846, see Table 8-1. The internal standard method is used for quantitation of analytes of interest. For quantitation, response factors are calculated from the base ion peak of a specific internal standard that is added to each calibration standard, blank, QC sample, and sample.

**TABLE 8-1. BFB KEY ION ABUNDANCE CRITERIA**

| Mass | Ion Abundance Criteria |
|---|---|
| 50 | 15 to 40% of mass 95 |
| 75 | 30 to 60% of mass 95 |
| 95 | Base peak, 100% relative abundance |
| 96 | 5 to 9% of mass 95 |
| 173 | Less than 2% of mass 174 |
| 174 | Greater than 50% of mass 95 |
| 175 | 5 to 9% of mass 174 |
| 176 | Greater than 95%, but less than 101% of mass 174 |
| 177 | 5 to 9% of mass 176 |

System performance is verified initially and after every 12 hours to ensure a minimum average response factor of 0.3 (0.25 for bromoform) for the following system performance check compounds (SPCCs):

- Chloromethane;
- 1,1-Dichloroethane;
- Bromoform;
- 1,1,2,2-Tetrachloroethane; and
- Chlorobenzene.

A five-point calibration, used for generating response factors, is performed initially using 10, 20, 50, 100, and 200 µg/L standards. The relative standard deviation (RSD) must be less than 30 percent for the five response factors calculated for each of the following calibration check compounds (CCCs):

- 1,1-Dichloroethene;
- Chloroform;
- 1,2-Dichloropropane;
- Toluene;
- Ethylbenzene; and
- Vinyl chloride.

The continuing (every 12 hours) calibration check is performed, following the system performance check, using the CCCs listed above. A single concentration of each CCC is analyzed and a response factor calculated. The single-point RF for each CCC must be within 25 percent of the average five-point RF; otherwise, a new five-point calibration must be generated.

**Method SW8270**
**Semivolatile Organics Analysis**

Semivolatile organics, also known as base/neutral and acid extractables (BNA), in water and soil samples are analyzed using Method SW8270. These techniques quantitatively determine the concentration of a number of semivolatile organic compounds. Organic compounds are extracted from the sample with methylene chloride at pH greater than 12 to

obtain base/neutral extractables. Acid extractable compounds are obtained from the sample by extraction with methylene chloride at pH 2 or less. Both base/neutral and acid extracts are then concentrated by removal of the methylene chloride through evaporation. Compounds of interest are separated and quantified using a GC/MS. The compounds that can be detected using Method SW8270 and the reporting limits are listed in Table 8-12.

Alternate methods for determination of these species are the GC methods, SW8010, SW8040, and SW8080. Many of the base/neutral extractable species have no corresponding GC method, and nonspecified compounds cannot be determined using GC.

Calibration--The mass spectrometer is tuned daily to give an acceptable spectrum for decafluorotriphenyl phosphene (DFTPP). Decafluorotriphenyl phosphene ion abundance criteria are given in SW846, see Table 8-2. The internal standard method is used for quantitation of analytes of interest. For quantitation, response factors are calculated from the base ion peak of a specific internal standard that is added to each calibration standard, blank, QC sample, and sample.

System performance is verified initially and after every 12 hours to ensure a minimum average response factor of 0.050 for the following system performance check compounds (SPCCs):

- N-nitroso-di-n-propylamine;
- Hexachlorocyclopentadiene;
- 2,4-Dichlorophenol; and
- 4-Nitrophenol.

A five-point calibration, used for generating response factors, is performed initially using 10, 20, 50, 80, and 160 $\mu$g/L standards. The variability for specific ion response factors for the SW8270 calibration check compounds must be less than 30 percent RSD over the range calibrated.
The CCCs are:

- Phenol;
- 1,4-Dichlorobenzene;
- 2-Nitrophenol;

**TABLE 8-2. DFTPP KEY ION ABUNDANCE CRITERIA**

| Mass | Ion Abundance Criteria |
|---|---|
| 51 | 30 to 60% of mass 198 |
| 68 | Less than 2% of mass 69 |
| 70 | Less than 2% of mass 69 |
| 127 | 40 to 60% of mass 198 |
| 197 | Less than 1% of mass 198 |
| 198 | Base peak, 100% relative abundance |
| 199 | 5 to 9% of mass 198 |
| 275 | 10 to 30% of mass 198 |
| 365 | Greater than 1% of mass 198 |
| 441 | Present, but less than mass 443 |
| 442 | Greater than 40% of mass 198 |
| 443 | 17 to 23% of mass 442 |

- 2,4-Dichlorophenol;
- Hexachlorobutadiene;
- 4-Chloro-3-methylphenol;
- Acenaphthene;
- 2,4,6-Trichlorophenol;
- N-nitroso-di-n-phenylamine;
- Pentachlorophenol;
- Fluoranthene;
- Di-n-octylphthalate; and
- Benzo(a)pyrene.

A continuing (every 12 hours) calibration check is performed, following the system performance check, using the CCCs listed above. A single concentration of each CCC is analyzed and a response factor calculated. The single-point RF for each CCC must be within 30 percent of the average five-point RF; otherwise, a new five-point calibration must be generated.

**Method SW8280**
**Polychlorinated Dibenzo-p-Dioxins (PCDDs) and**
**Polychlorinated Dibenzofurans (PCDFs)**

Method SW8280 is used to analyze for PCDDs and PCDFs in water, soil, and waste. This is a GC/MS Method that uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column GC/low resolution MS techniques to separate and identify the analytes of interest. The sensitivity of the method is dependent on the level of matrix interference; selected cleanup methods may be used to reduce or eliminate interferences. Target analytes include all congener classes, tetra- through octa-dioxins and furans. Achieved detection limits vary according to matrix and analyte. Because of the extreme toxicity of these compounds, the analyst must take appropriate precautions during preparation and analysis to prevent accidental exposure. Reporting limits are presented in Table 8-13.

Calibration--PCDDs/PCDFs are analyzed according to the procedures outlined in Method 8280, SW-846, 3rd ed. Mass calibration of the instrument are performed daily

with PFTBA and verified using DFTPP ion abundance criteria (as specified in SW-846 Method 8270) for mass 198 and greater. Adherence to Method 8280 criteria for isotopic ratio measurements for PCDDs and PCDFs is then verified.

Since selected-ion-monitoring mass spectrometry is used for PCDD/PCDF analysis, it is first necessary to determine the retention time windows for each isomer group. This is accomplished by injection of a PCDD/PCDF retention time standard which contains the first and last eluter from each isomer group. The isomer group retention time analysis is performed once at the beginning of each project and again if retention times shift more than 0.2 minutes. Adequate sensitivity is also verified from the retention time standard which contains 2,3,7,8-TCDD at a concentration of 0.2 $\mu$g/mL.

A TCDD chromatographic test mixture is analyzed daily to verify that there is at least 25 percent valley resolution between 2,3,7,8-TCDD and 1,2,3,4-TCDD.

Response factors for each PCDD/PCDF isomer class are determined from an initial five point calibration at 0.1, 0.2, 0.5, 1.0, and 2.5 $\mu$g/mL concentrations. This five-point calibration is performed in triplicate and the average response factors are calculated. The response factors determined for each isomer class must not have a percent RSD greater than 30 percent. Where more than one isomer per isomer group is present, the average response factor is used.

For quantitation, the following mixture of internal standards is contained in the calibration standard solutions. This mixture is also added to each sample before extraction:

- $^{13}C_{12}$-2,3,7,8-TCDD;
- $^{13}C_{12}$-2,3,7,8-TCDF;
- $^{13}C_{12}$-1,2,3,7,8-PeCDD;
- $^{13}C_{12}$-1,2,3,7,8-PeCDF;
- $^{13}C_{12}$-1,2,3,6,7,8-HxCDD;
- $^{13}C_{12}$-1,2,3,4,7,8-HxCDF;
- $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD;

- $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF;
- $^{13}C_{12}$-OCDD and;
- $^{13}C_{12}$-OCDF.

Each isomer class is quantitated using a C-13 internal standard from that class.

The 1.0 μg/mL PCDD/PCDF standard is analyzed at the beginning of each working day to verify that the response factors obtained for that day agree to within ±30 percent of those obtained from the initial five-point calibration in triplicate. Also, isotope ratios for each PCDD/PCDF isomer class are verified to be within ±15 percent of their theoretical values.

**Method SW8310**
**Polynuclear Aromatic Hydrocarbons (PAHs)**

SW8310 is used to determine the concentration of selected PAHs in groundwater and wastes. SW8310 provides high performance liquid chromatographic (HPLC) conditions for the detection of ppb levels of PAHs. Samples are analyzed by direct injection. Detection is by ultraviolet (UV) and fluorescence detectors. Reporting Limits are listed in Table 8-14.

Calibration--For initial calibration, standards at five concentration levels are prepared by dilution of stock standards with acetonitrile. The average calibration factor is acceptable if the relative standard deviation (RSD) does not exceed 20 percent or linear regression may be used if the correlation coefficient is 0.995 or greater. Daily calibration checks are acceptable if the difference between the daily response versus the initial (multipoint) response does not exceed ±15 percent.

**Method SW9060**
**Total Organic Carbon**

Total organic carbon is measured using a carbonaceous analyzer by quantitatively converting the organic carbon in a sample to carbon dioxide, which is measured by an infrared detector. Reporting limits are presented in Table 8-15.

Calibration--A multipoint calibration curve is generated daily, prior to sample analysis, using a blank and a minimum of three standards. Potassium hydrogen phthalate is used for total carbon measurements and sodium carbonate/bicarbonate is used for inorganic carbon measurements. The correlation coefficient for the calibration equation must exceed 0.995. A QC check is analyzed after every 10 samples; recovery must be within ±10 percent of the expected value.

**U.S. EPA Method 600/M4-82-20**
**Polarized Light Microscopy (PLM) Asbestos**

Asbestos in soil is analyzed by U.S. EPA Method 600/M4-82-020 using PLM. The soil sample is dried and coarsely ground, then viewed through a stereomicroscope to assure the soil has been ground to a uniform size. The individual fibers are identified with a polarized light microscope. The concentration of asbestos in the soil sample is determined by comparison with three different standard concentrations.

Calibration--The instrument is calibrated with three different concentrations of asbestos standards--0.1 percent, 0.25 percent, and 0.5 percent by weight. The method detection limit for this method is 0.1 percent by weight.

**U.S. EPA Method 901.1**
**Gamma Emitting Radionuclides in Water**

This method measures gamma photons in water samples using gamma spectroscopy. The sample is put into a standard geometry and counted using a lithium drifted germanium detector, Ge(Li). The counting efficiency for the geometry is compared to the efficiency determined for a standard (known) radionuclide activity. The sample aliquot is counted long enough to meet the required sensitivity of the measurement, specified in the SOP.

Calibration--A standard of radium-226 in a barium sulfate precipitate is counted and must meet an acceptance criterion of 30 percent of the known value or a blank check and reanalysis must be done.

**Method SW9310**
**Gross Alpha and Gross Beta**

An aliquot of sample is evaporated to a small volume and transferred quantitatively to a tared 2-inch stainless steel counting planchet. The residue is dried to a constant weight, reweighed to determine dry residue weight, and then counted for alpha and/or beta radioactivity. Reporting limits are presented in Table 8-15.

Calibration--For gross alpha and gross beta measurement, the detectors must be calibrated to obtain the ratio of count rate to disintegration rate. Standards using americium-24 (for alpha activity) and strontium-90 (for beta activity) are used. Separate alpha and beta particle self-absorption graphs showing water sample residue weight (mg) vs. efficiency factor (cpm/dpm) using standard alpha and beta emitter solutions and tap water should be prepared for each counting instrument.

## 8.4 Real-Time Portable Field Meters

Real-time portable field meters will be used to provide screening-level information for organic vapors and radioactivity during site investigations, sampling, and health and safety monitoring. The types of meters used are described in the following subsections.

### 8.4.1 Real-Time Portable Organic Vapor Analyzers

Two types of portable analyzers will be used to perform real-time non-specific analyses of hydrocarbon vapors. The instruments include a Foxboro Century Organic Vapor Analyzer (OVA), which utilizes the technique of flame ionization detection (FID), and an HNu® Systems (HNu®) trace gas analyzer, OVM, and MicroTip™, which use the technique of photoionization detection (PID). One or more of these instruments may be used at a specific site, depending upon the contaminant species of interest. When used together, the instruments provide complementary information because they are sensitive to different types of hydrocarbon vapors.

The portable analyzers will be used as a screening tool to help determine the optimum locations for the collection of samples. Field data recorded on the Chain-of-

Custody forms will give the laboratory analysts an indication of the approximate concentration of contaminants and will aid in calculating dilution factors prior to analysis. Additionally, the real-time instruments will be used to aid in the selection of the proper level of personal protective equipment and for monitoring air emissions during sampling activities. The comparability of results obtained from the PID instruments can be considered only to be within the variability of this type of screening instrument. Comparability is greatest when the instruments are calibrated with the same standards and operated within similar concentration ranges.

**Organic Vapor Analyzer**

The Foxboro Century Organic Vapor Analyzer (OVA) uses the principle of hydrogen flame ionization for the detection and measurement of total hydrocarbon vapors. The OVA has a dynamic operating range from 1 to 10,000 ppmv or 100,000 ppmv, depending on the instrument, and provides a non-specific response to total hydrocarbons. If concentrations in excess of the range of the instrument are encountered, a dilution probe will be attached to the OVA to allow measurement of elevated vapor concentrations. The instrument is highly sensitive to compounds such as methane, benzene, and acetone, but is less sensitive to alcohols and halogenated compounds.

During operation, a sample is drawn into the probe and transmitted to the detection chamber by an internal pumping system. Inside the chamber, the sample is exposed to a hydrogen flame that ionizes the organic vapors. As the organic vapors burn, the ions produced are collected on an electrode in the chamber, and a current proportional to the hydrocarbon concentration is generated. This current is measured and displayed on the meter.

**HNu®**

The HNu® Systems trace gas analyzer, OVM, and/or Photovac MicroTIP® uses the principle of photoionization for the detection and measurement of non-methane hydrocarbon vapors. The HNu® has a dynamic operating range from 0.5 to 2000 ppmv. The HNu® is highly sensitive to aromatic compounds such as benzene and toluene; moderately sensitive to unsaturated chlorinated hydrocarbons such as trichloroethene and

dichloroethene and unsaturated hydrocarbons; and less sensitive to aliphatic hydrocarbons with 1 to 7 carbons and ammonia.

During operation, a gas sample is drawn into the probe and past an ultraviolet (UV) light source by an internal pumping system. Contaminants in the sample are ionized, producing an instrument response if their ionization potential is equal to or less than the ionizing energy supplied by the UV lamp being used. The radiation produces a free electron for each molecule of ionized contaminant, which generates a current directly proportional to the number of ions produced. This current is measured and displayed on the meter.

The HNu® can be used with one of three different probes, each with a different ionizing energy: 9.5, 10.2, or 11.7 electronic volts (eV). Normally, the HNu® is used with the 10.2 eV lamp, which ionizes many of the common air contaminants. Species that have a very high ionization potential, greater than the lamp rating, will display a poor instrument response, or no response at all. Using the 11.7 eV lamp will ensure the greatest range of detectable species; however, it requires constant maintenance and frequent replacement.

The HNu® is an excellent survey tool, but there are some limitations: the response to a single compound may radically change if it is mixed with other compounds, indicating considerably more or less than the true total, depending upon calibration; radio frequency interference may produce an error in response; the lamp window must be periodically cleaned to ensure ionization of the air contaminants; and the response to compounds may not be linear over the full range of the instrument.

**Organic Vapor Meter (OVM)**

The Thermo Environmental Instruments Inc. Model 580B is a portable Organic Vapor Meter (OVM) that detects and quantifies most organic vapors using a highly sensitive photoionization detector (PID). The instrument has an operating range of 0 - 2000 parts per million (ppm) with a minimum detection limit of 0.1 ppm.

Air samples are drawn into the OVM with a positive displacement pumping system. The samples are passed through the detector which uses a 10.0 electron volt (eV) lamp as its ionization source. Organic compounds with ionization potentials of 10.0 eV or

less will be detected with the OVM. Compounds that will not be detected include freons, methane, ethane, and propane. It is important to note that the OVM does not characterize the particular organic vapor species (unless the source being sampled is known), but measures the total value for all species present with ionization potentials of 10.0 eV or less.

**MicroTip™**

The Photovac Inc. MicroTip™ monitor is another type of hand-held portable photoionization detector (PID). The MicroTip™ has an operating range of 0 - 200 ppm with a minimum detection limit of 0.1 ppm.

Operation of the MicroTip™ is similar to the HNu®. Air samples are drawn into the detector with a built in pump. The instrument is equipped with a 10.6 eV ionization lamp, but can be fitted with 9.5 eV or 11.7 eV lamps. The user must keep in mind that the ionization potential of the compound(s) of interest must be considered when selecting the voltage of the lamp. As with the other PID instruments, the measured values reported by the MicroTip™ are quantitative but not species specific (unless the source is already known).

### 8.4.2 Real-Time Radioactivity Meter

A surface monitoring system will be used as a screening tool to help determine the location for the collection of samples that will be analyzed for gross alpha and beta radioactivity or gamma-emitting radionuclides. The radioactive spectrum detector is a pulse-count ratemeter with a power supply. With the appropriate detector probes, it acts as a survey meter for alpha, beta, and gamma radiation. Its range of operation is 0 to 60,000 counts per minute or 0 to 15 milliroentgen per hour.

## 8.5 Air Analysis

**Compendium Method TO-14**
**Multiple Detector Gas Chromatography of Vapors By Stainless Steel Canister**

This subsection briefly describes the canister analysis procedure for analyzing air samples. The canister method has been accepted by the U.S. EPA and the California

Department of Health Services (DHS), and follows the guidelines established in U.S. EPA's Compendium of Methods for the Determination of Toxic Compounds in Ambient Air (EPA-600/4-84-041, April 1984).

Ambient air samples for detailed speciation of volatile organics will be collected in evacuated stainless steel canisters.

When a sample is received from the field, it is first assigned a code number and logged into a master log notebook. The final pressure is then measured and logged into a separate sample analysis notebook before pressurizing the canister with ultra high purity nitrogen (UHP $N_2$) to 10-15 psig. The final pressure is then measured and recorded. Nitrogen is added to the canisters to provide positive pressure for removing the sample and to dilute oxygen and moisture in the sample and minimize sample component reactions.

To achieve the desired detection levels, volatile organic species are separated from the ambient air matrix and concentrated. The analytical procedure consists of the following operations:

- Collection of VOCs through a Permapure® drier onto a cryogenic trap;
- Flash thermal desorption onto a fused silica capillary GC column;
- Detection of the VOCs by flame ionization detector (FID), photoionization detector (PID), and Hall electrolytic conductivity detector (HSD); and
- Computer-assisted data reduction.

Calibration--The canister analysis calibration is performed by a monthly generation of a calibration curve using various standards, depending upon the detector. The FID and PID detectors are calibrated using a multipoint propane/hexane concentration curve plus an ultra high purity air blank. The correlation coefficient must be greater than or equal to 0.9950. A daily response factor check and monthly retention time check ensures continuing performance.

The halide specific detector (HSD) is calibrated with a multipoint halogenated standard, with the curve having a correlation coefficient of greater than or equal to 0.9950. Daily response factor checks ensure continuing performance.

## 8.6 Analytical Methods for Physical Properties

Samples will be collected from selected sites for physical properties analyzed to supply information for engineering design, modeling studies, or other types of projects that require these parameters to fulfill the DQOs. Quality control procedures for these analyses are specified in the reference methods.

**ASTM D422-68 (Reapproved 1972)**
**Standard Method for Particle-Size Analysis of Soils**

This method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 $\mu$m (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 $\mu$m is determined by a sedimentation process using a hydrometer.

**ASTM D854-83**
**Standard Test Method for Specific Gravity of Soils**

This test method covers the determination of the specific gravity of soils by means of a pycnometer. When the soil is composed of particles larger than the No. 4 (4.75 mm) sieve, the method outlined in Test Method C127 shall be followed. When the soil is composed of particles both larger and smaller than the No. 4 sieve, the sample shall be separated on the No. 4 sieve and the appropriate test method used on each portion. The specific gravity value for the soil shall be the weighted average of the two values. When the specific gravity value is to be used in calculations in connection with the hydrometer portion of Method D422, it is intended that the specific gravity test be made on that portion of the soil which passes the No. 10 (2.00 mm) sieve.

Porosity of a soil sample will be determined from the relation:

$$\text{Total porosity} = 1 - \frac{\text{dry bulk density}}{\text{particle density}}$$

A particle density of 2.65 g/cm$^2$ will be assumed for the soils beneath McClellan AFB. This particle density is a reasonable value for the quartz-feldspar-clay-mica soils typically encountered.

Volumetric water content of a soil sample will be determined from the relation:

$$\text{Volumetric fraction } (cm^3/cm^3) = \frac{\text{dry bulk density soil}}{\text{bulk density water}} \times \text{weight fraction water } (g/g)$$

The bulk density of water will be assumed to equal 1 g/cm$^3$. The weight fraction of water will be determined by ASTM Method 2216-80. The dry bulk density of the soil is also obtained from this method.

**ASTM 2216-80**
**Standard Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures**

This method covers the laboratory determination of the water (moisture) content of soil, rock, and soil-aggregate mixtures by weight. The practical application in determining the water content of a material is to determine the mass of water removed by drying the moist material to a constant mass in a drying oven controlled at 110 $\pm$5°C and to use this value as the mass of water in the test specimen. The mass of material remaining after oven-drying is used as the mass of solid particles.

**ASTM 2434-68 (Reapproved 1974)**
**Standard Method for Permeability of Granular Soils (Constant Head)**

This method covers the determination of the coefficient of permeability by a constant-head method for the laminar flow of water through granular soils. The procedure is to establish representative values of the coefficient of permeability of granular soils that may occur in natural deposits as placed in embankments, or when used as base courses under pavements. In order to limit consolidation influences during testing, this procedure is limited to disturbed granular soils containing not more than 10 percent soil passing the 75 $\mu$m (No. 200) sieve ASTM D2435-80.

The method requires that an element of soil be restrained laterally and axially loaded in increments with a constant stress being applied until all of the excess port water pressures have dissipated for each increment. During the compression process, measurements of decreases in the sample height are made and these data are used to compute the parameters that describe the relationship between effective stress and void ratio or strain and describe the rate at which compression can occur.

**ASTM D2435-80**
**Standard Method for One-Dimensional Consolidation Properties**

The method requires that an element of soil be restrained laterally and axially loaded in increments with a constant stress being applied until all of the excess pore water pressures have dissipated for each increment. During the compression process, measurements of decreases in the sample height are made and these data are used to compute the parameters that describe the relationship between effective stress and void ratio or strain and describe the rate at which compression can occur.

## 8.7 Analytical Methods for Field Analyses

The field analyses describe here are Level 2 analyses and are modifications of standard methods. The calibration and QC acceptance criteria are less stringent to reflect the objectives of the screening level investigation. Standard operating procedures are included in Appendix A. The analytical procedure appropriate for the field program objectives is selected.

**Soil Gas - Subsurface Probe Method**

A modified Geo-Probe Model 8M or transportable Xitech® soil gas sampling system will be used to install stainless steel soil gas probes into the soil to approximately 6 feet in depth. The sample is withdrawn from the bottom of the probe through a Teflon® insert and a glass bulb at the upper end of the probe, by a battery powered vacuum pump. Samples will be analyzed in an on-site laboratory stationed near the collection site. A known volume of soil gas will be injected, and the analysis will be performed using a Gas Chromatograph for target compounds by a modification of Method 8240. Low

concentrations of target chlorinated compounds will be analyzed by a GC with dual electron capture detectors (ECD).

Calibration (GC/MS)--A minimum of a four-point initial calibration is performed using gas standards. The relative standard deviation (RSD) must be less than 25 percent for all target compounds. An initial daily calibration check must agree within 30 percent and a QC check standard analyzed as the last sample of the day must be within 70 to 130 percent of the predicted value.

Calibration (GC)--An initial four-point calibration (blank and 3 upscale standards) is performed using gas standards. A response factor (R.F.) will be determined for each of the compounds using a linear regression. The correlation coefficient for each of the compounds must be $>0.990$ or the calibration is repeated. An initial daily calibration check must agree within 30 percent and a QC check standard analyzed as the last sample of the day must be within 70 to 130 percent of the predicted value.

**Soil Gas - Downhole and Shallow Subsurface Probe**

Soil gas samples will be collected by drawing a known amount of soil gas through a teflon line and probe at a specified depth at a drilling site. All downhole soil gas samples will be analyzed using HP 5890 II GCs equipped with a DB-624 capillary column and a photoionization detector/electron capture detector (PID/ECD). Reporting limits are presented in Table 8-16.

Calibration--An initial calibration is performed using three concentrations of gas standards and a blank. A linear regression for each analyte is done and a correlation coefficient of $>0.990$ must be achieved, or the calibration is repeated. The slope for the linear regression is used as the response factor. Daily calibration check standards should meet recoveries within $\pm 30$ percent of the calculated value, or the standard must be reinjected. If the second daily calibration check standard does not meet the criteria, corrective action and a new calibration must be performed. A calibration check will also be performed at the end of the day, and if the result is not within $\pm 30$ percent of the expected value, results for analytes not meeting the criteria will be flagged to indicate uncertainty in quantitation. Corrective action, up to and including a new multipoint calibration will be required the next day of operation.

**Polychlorinated Biphenyls (PCB) in Soils**

Soil samples will be collected during drilling, and analyzed by a modification of Method 8080, and the data will be used for screening levels of PCBs (7 different Aroclors) in the soil. A mini acid extraction of the soil samples will be performed, and the extracts will be injected into a GC equipped with a packed or equivalent column, and detected using either an ECD or other halogen specific detector. The reporting limits for PCBs will be 200 $\mu$g/kg.

Calibration--The GC is initially calibrated at a minimum of three concentrations. The average calibration factor is acceptable if the RSD for the calibration factors at each level does not exceed 30 percent or linear regression may be used if the correlation coefficient is 0.990 or greater. A daily single-point calibration check must agree within $\pm$30 percent of the predicted value. A calibration check at the end of each analysis day (or approximately 1 every 20 samples) must be within $\pm$30 percent of the predicted value.

**Volatile Organic Compounds (VOCs) in Soils**

Soil samples will be screened for VOCs using a modification of EPA Methods SW8010 and SW8020. Each GC will be equipped with a megabore column, purge and trap extraction apparatus, and both a PID and Hall-type detectors. The samples will be analyzed for the target analytes vinyl chloride, 1,2-DCE, chloroform, 1,1,1-TCA, TCE, PCE, benzene, toluene, and xylenes. The reporting limits are presented in Table 8-17. . Unidentified peaks greater than approximately 100 $\mu$g/kg will be quantitated using the response factor for the nearest calibrated analyte calibration. The GCs will be initially calibrated using a blank and 3 upscale standards. The calibration will be acceptable if the average response factor relative standard deviation is $\geq$ 20 percent or the correlation coefficient is $\geq$ 0.995. An initial daily single point calibration check using a mid-range standard must be $\pm$20 percent of the expected value to proceed, and continuing checks analyzed after every 10 samples must be $\pm$30 percent. Corrective actions for not meeting these criteria will include reanalysis of the standard, or recalibration for non-complaint initial daily checks, and flagging data for analytes not meeting continuing checks.

## TABLE 8-3. REPORTING LIMITS FOR INORGANIC ANALYTICAL METHODS

| Method | Parameter | Analytes | Reporting Limits | |
|---|---|---|---|---|
| | | | Soil | Water |
| E120.1 | Conductivity | -- | 100 umhos/cm | 1 umhos/cm |
| E160.1 | Total dissolved solids | -- | -- | 10 mg/L |
| E300.0 | Anions | Chloride | 2.00 mg/kg | 0.5 mg/L (0.2)* |
| | | Fluoride | | 0.5 mg/L (0.2)* |
| | | Sulfate | 5.00 mg/kg | 1 mg/L (0.2)* |
| | | Nitrite | -- | 1 mg/L |
| | | Nitrate | -- | 0.1 mg/L |
| E310.1 | Alkalinity | Bicarbonate | 100 mg/kg | 10 mg/L |
| | | Carbonate | 100 mg/kg | 10 mg/L |
| | | Total alkalinity | 100 mg/kg | 10 mg/L |
| | | Hydroxide | -- | 10 mg/L |
| E353.2 | Nitrate-nitrite | Nitrate-nitrite | 2.00 mg/kg | 0.5 mg/L (0.05)* |
| SW7196 | Chromium VI | Chromium VI | 0.500 mg/kg | 0.005 mg/L |
| SW9010/ SW9012 | Cyanide | Cyanide | 1.00 mg/kg | 0.02 mg/L |

* Exceeds 5/91 IRP limit, shown in parentheses.

HML = Hazardous Materials Laboratory (Cal/EPA).
E = Methods from Unites States Environmental Protection Agency, 1983.
SW = Methods from SW846, Third Edition.

## TABLE 8-4. REPORTING LIMITS FOR TRACE ELEMENTS (METALS) FOR ICPES AND GRAPHITE FURNACE METHODS

| Method | Parameter | Analytes | Reporting Limits Soil (mg/kg) | Reporting Limits Water (mg/L) |
|---|---|---|---|---|
| SW6010 | ICPES | Aluminum | 50 | 0.5 |
| | | Antimony | 20 (15)* | 0.5 |
| | | Arsenic | 30 | 0.3 |
| | | Barium | 10 | 0.1 |
| | | Beryllium | 1 | 0.01 |
| | | Cadmium | 0.400 | 0.005 |
| | | Calcium | 100 | 1.0 |
| | | Chromium | 5 | 0.05 |
| | | Cobalt | 5 | 0.05 |
| | | Copper | 5 | 0.05 |
| | | Iron | 5 | 0.05 |
| | | Lead | 10 (5)* | 0.2 |
| | | Magnesium | 100 | 1 |
| | | Manganese | 2 | 0.02 |
| | | Molybdenum | 4 | 0.10 |
| | | Nickel | 15 | 0.15 |
| | | Potassium | 100 | 5 |
| | | Selenium | 50 | 1 |
| | | Silver | 1 | 0.05 |
| | | Sodium | 100 | 1 |
| | | Thallium | 10 (7)* | 0.4 |
| | | Vanadium | 10 | 0.1 |
| | | Zinc | 2 | 0.02 |
| SW7060 | Graphite furnace | Arsenic | 0.5 | 0.005 |
| SW7421 | Graphite furnace | Lead | 0.5 | 0.005 |
| SW7470/ SW7471 | Cold vapor | Mercury | 0.1 | 0.001 |
| SW7740 | Graphite furnace | Selenium | 0.50 | 0.005 |

* Exceeds the 5/91 IRP limit, shown in parentheses.

SW = Methods for SW846, Third Edition.

**TABLE 8-5. REPORTING LIMITS FOR METHOD SW8010 HALOGENATED VOLATILE ORGANICS**

| Method | Parameter | Analytes | Reporting Limits Soil[a] (mg/kg) | Reporting Limits Water (µg/L) |
|---|---|---|---|---|
| SW8010 | Halogenated volatile organics | Bromobenzene | 0.16 (0.05)* | 5.0 |
| | | Bromodichloromethane | 0.05 | 1.0 |
| | | Bromoform | 0.05 | 2.0 |
| | | Bromomethane | 0.035 (0.010)* | 10.0 |
| | | Carbon tetrachloride | 0.035 (0.005)* | 1.0 |
| | | Chlorobenzene | 0.03 (0.005)* | 2.5 |
| | | Chloroethane | 0.07 (0.005)* | 5.0 |
| | | 2-Chloroethylvinyl ether | 0.06 (0.010)* | 10 |
| | | Chloroform | 0.015 (0.005)* | 0.5 |
| | | 1-Chlorohexane | 0.34 (0.005)* | 5.0 |
| | | Chloromethane | 0.05 (0.005)* | 1.0 |
| | | Dibromochloromethane | 0.02 (0.005)* | 1.0 |
| | | Dibromomethane | 0.016 (0.005)* | 5.0 |
| | | 1,2-Dichlorobenzene | 0.025 (0.005)* | 2.0 |
| | | 1,3-Dichlorobenzene | 0.032 (0.005)* | 3.0 |
| | | 1,4-Dichlorobenzene | 0.025 (0.005)* | 2.0 |
| | | 1,1-Dichloroethane | 0.05 (0.005)* | 1.0 |
| | | 1,2-Dichloroethane | 0.015 (0.005)* | 1.0 |
| | | 1,1-Dichloroethene | 0.07 (0.005)* | 1.0 |
| | | trans-1,2-Dichloroethene | 0.025 (0.005)* | 1.0 |
| | | 1,2-Dichloropropane | 0.015 (0.005)* | 1.0 |
| | | cis-1,3-Dichloropropene | 0.02 (0.005)* | 5.0 |
| | | trans-1,3-Dichloropropene | 0.015 (0.005)* | 3.0 |
| | | Methylene chloride | 0.04 (0.005)* | 2.0 |
| | | 1,1,1,2-Tetrachloroethane | .25 (0.005)* | 5.0 |
| | | 1,1,2,2-Tetrachloroethane | 0.03 (0.005)* | 1.0 |
| | | Tetrachloroethene | 0.01 (0.005)* | 1.0 |
| | | 1,1,1-Trichloroethane | 0.055 (0.005)* | 1.0 |
| | | 1,1,2-Trichloroethane | 0.02 (0.005)* | 1.0 |
| | | Trichloroethene | 0.02 (0.005)* | 1.0 |
| | | Trichlorofluoromethane | 0.055 (0.005)* | 1.0 |
| | | 1,2,3-Trichloropropane | 0.16 (0.010)* | 10 |
| | | Vinyl chloride | 0.025 (0.005)* | 2 |

[a] Soil limits are based on 2.0 g methanol extraction followed by a 1:50 dilution prior to analysis by the purge-and-trap method.

* Exceeds the 5/91 IRP limit, shown in parentheses.

SW = Methods from SW846, Third Edition.

**TABLE 8-6. REPORTING LIMITS FOR MODIFIED METHOD SW8015 TOTAL PETROLEUM HYDROCARBONS (TPH)**

| Method | Parameter | Analytes | Reporting Limits Soil[a] (mg/kg) | Reporting Limits Water (µg/L) |
|---|---|---|---|---|
| SW8015-Volatile | TPH | Benzene | 10 | 0.5 |
| | | Toluene | 10 | 0.5 |
| | | Total xylenes | 10 | 1.0 |
| | | Ethylbenzene | 10 | 0.5 |
| | | Volatile TPH[b] (jet fuel, gasoline) | 10 | 1,000 |
| SW8015-Extractable | TPH | Extractable TPH[c] (diesel, oils to C32) | 10 | 1,000 |

[a] Volatile TPHs are analyzed via direct soil sparging of 5 g sample.

[b] Volatile TPH components used for calibration are jet fuel and gasoline; other peak patterns may occur and be narrated on the analytical reports. A range of TPH concentrations is usually reported; the reporting limit represents the lowest concentration in that range.

[c] Extractable TPH components used for calibration are diesel and oils up to C32. Representative peak patterns are noted on the analytical report. A range of TPH concentrations is usually reported; the reporting limit represents the lowest concentration in that range.

SW = Methods from SW846, Third Edition.

**TABLE 8-7. METHOD REPORTING LIMITS FOR METHOD SW8020 AROMATIC VOLATILE ORGANICS**

| Method | Parameter | Analytes | Reporting Limits Soil[a] (mg/kg) | Reporting Limits Water (µg/L) |
|---|---|---|---|---|
| SW8020 | Aromatic volatile organics | Benzene | 0.03 (0.01)* | 1.0 |
| | | Toluene | 0.02 | 1.0 |
| | | Ethylbenzene | 0.04 | 4.0 |
| | | Chlorobenzene | 0.02 | 1.0 |
| | | Total xylenes | 0.03 (0.02)* | 2.0 |
| | | 1,4-Dichlorobenzene | 0.04 (0.01)* | 0.5 |
| | | 1,3-Dichlorobenzene | 0.04 | 4.0 |
| | | 1,2-Dichlorobenzene | 0.04 | 4.0 |

[a] Soil limits are based on 2.0 g methanol extraction followed by a 1:50 dilution prior to analysis by the purge-and-trap method.

* Exceeds the 5/91 IRP limit, shown in parentheses.

SW = Methods from SW846, Third Edition.

**TABLE 8-8. REPORTING LIMITS FOR METHOD SW8080 ORGANOCHLORINE PESTICIDES AND PCBs**

| Method | Parameter | Analytes | Reporting Limits[a] Soil[b] (mg/kg) | Reporting Limits[a] Water (µg/L) |
|---|---|---|---|---|
| SW8080 | Organochlorine pesticides and PCBs | alpha-BHC | 0.0015 | 0.03 |
| | | gamma-BHC (Lindane) | 0.015 | 0.04 |
| | | beta-BHC | 0.015 | 0.05 |
| | | Heptachlor | 0.002 | 0.03 |
| | | delta-BHC | 0.015 | 0.05 |
| | | Aldrin | 0.015 | 0.04 |
| | | Heptachlor epoxide | 0.002 | 0.05 |
| | | Endosulfan I | 0.015 | 0.05 |
| | | 4,4'-DDT | 0.003 | 0.10 |
| | | Dieldrin | 0.003 | 0.05 |
| | | Endrin | 0.003 | 0.06 |
| | | 4,4'-DDD | 0.003 | 0.11 |
| | | Endosulfan II | 0.004 (0.003)* | 0.10 |
| | | 4,4'-DDE | 0.003 | 0.04 |
| | | Endrin aldehyde | 0.003 | 0.10 |
| | | Endosulfan sulfate | 0.003 | 0.10 |
| | | Methoxychlor | 0.0015 | 0.50 |
| | | Chlordane | 0.015 | 0.05 |
| | | Toxaphene | 0.160 | 2.5 |
| | | PCB-1016 | 0.030 | 1.0 |
| | | PCB-1221 | 0.030 | 1.0 |
| | | PCB-1232 | 0.030 | 1.0 |
| | | PCB-1242 | 0.030 | 1.0 |
| | | PCB-1248 | 0.030 | 1.0 |
| | | PCB-1254 | 0.030 | 1.0 |
| | | PCB-1260 | 0.030 | 1.0 |

[a] Sensitivity of the method depends on the level of interferences rather than instrumental limitations. Typical waste samples may have higher reporting limits and may require additional cleanup techniques.

[b] Detection limits for soil are based on the extraction of 10 g of soil and are approximately 100 times those for water. In some cases, lower reporting limits may be achieved by extracting 30 g of sample.

* Exceeds the 5/91 IRP limit, shown in parentheses.

SW = Methods from SW846, Third Edition.

## TABLE 8-9. REPORTING LIMITS FOR METHOD SW8140 PESTICIDES

| Method | Parameter | Analytes | Reporting Limits Soil (mg/kg) | Reporting Limits Water (µg/L) |
|---|---|---|---|---|
| SW8140 | Pesticides | Azinphos methyl | 1.0 | 15 |
| | | Bolstar | 0.1 | 2 (1.5)* |
| | | Chlorpyrfos (Dursban) | 0.2 | 3 |
| | | Coumaphos | 1.0 | 15 |
| | | Demeton - O | 0.2 | 2.5 |
| | | Demeton - S | 0.2 | 2.5 |
| | | Diazinon | 0.4 | 6 |
| | | Dichlorvos | 0.7 | 10 |
| | | Disulfoton | 0.1 | 2 |
| | | Ethoprop | 0.2 | 3 (2.5)* |
| | | Fensulfothion | 10 | 15 |
| | | Fenthion | 0.1 | 1 |
| | | Merphos | 0.2 | 2.5 |
| | | Mevinphos | 0.2 | 3 |
| | | Naled | 0.1 | 1 |
| | | Parathion methyl | 0.030 (0.020)* | 1 (0.3)* |
| | | Phorate | 0.1 | 1.5 |
| | | Ronnel | 0.2 | 3 |
| | | Stirophos | 3.4 | 50 |
| | | Tokuthion | 0.4 | 5 |
| | | Trichloronate | 0.1 | 1.5 |

* Exceeds the 5/91 IRP limit, shown in parentheses.

SW = Methods from SW846, Third Edition.

**TABLE 8-10. REPORTING LIMITS FOR METHOD SW8150 HERBICIDES**

| Method | Parameter | Analytes | Reporting Limits Soil (mg/kg) | Reporting Limits Water (µg/L) |
|---|---|---|---|---|
| SW8150 | Herbicides | 2,4-D | 0.8 | 12 |
| | | 2,4-DB | 0.6 | 9 |
| | | Dalapon | 4.0 | 60 |
| | | Dicamba | 0.2 | 2.7 |
| | | Dichloroprop | 0.5 | 6.5 |
| | | Dinoseb | 0.05 | 0.7 |
| | | MCPA | 170 | 2,500 |
| | | MCPP | 130 | 1,900 |
| | | 2,4,5-T | 0.1 | 2 |
| | | 2,4,5-TP (Silvex) | 0.1 | 1.7 |

SW = Methods for SW846, Third Edition.

**TABLE 8-11. REPORTING LIMITS FOR METHOD SW8240 VOLATILE ORGANIC COMPOUNDS**

| Method | Parameter | Analytes | Reporting Limits Soil* (mg/kg) | Reporting Limits Water (µg/L) |
|---|---|---|---|---|
| SW8240 | Volatile organic compounds | Acetone | 2.0 (0.010)* | 100 (10)* |
| | | Benzene | 0.1 (0.005)* | 5 |
| | | Bromodichloromethane | 0.1 (0.005)* | 5 |
| | | Bromoform | 0.1 (0.005)* | 5 |
| | | Bromomethane | 0.2 (0.010)* | 10 |
| | | 2-Butanone | 0.2 (0.010)* | 100 (10)* |
| | | Carbon disulfide | 0.1 (0.005)* | 5 |
| | | Carbon tetrachloride | 0.1 (0.005)* | 5 |
| | | Chlorobenzene | 0.1 (0.005)* | 5 |
| | | Chloroethane | 0.2 (0.010)* | 10 |
| | | 2-Chloroethylvinylether | 0.2 (0.010)* | 10 |
| | | Chloroform | 0.1 (0.005)* | 5 |
| | | Chloromethane | 0.2 (0.010)* | 10 |
| | | Dibromochloromethane | 0.1 (0.005)* | 5 |
| | | 1,1-Dichloroethane | 0.1 (0.005)* | 5 |
| | | 1,2-Dichloroethane | 0.1 (0.005)* | 5 |
| | | 1,1-Dichloroethene | 0.1 (0.005)* | 5 |
| | | trans-1,2-Dichloroethene | 0.1 (0.005)* | 5 |
| | | 1,2-Dichloropropane | 0.1 (0.005)* | 5 |
| | | trans-1,3-Dichloropropene | 0.1 (0.005)* | 5 |
| | | cis-1,3-Dichloropropene | 0.1 (0.005)* | 5 |
| | | Ethylbenzene | 0.1 (0.005)* | 5 |
| | | 2-Hexanone | 1.0 (0.010)* | 50 (10)* |
| | | Methylene chloride | 0.1 (0.005)* | 5 |
| | | 4-Methyl-2-pentanone | 1.0 (0.010)* | 50 (10)* |
| | | Styrene | 0.1 (0.005)* | 5 |
| | | Tetrachloroethene | 0.1 (0.005)* | 5 |
| | | 1,1,2,2-Tetrachloroethane | 0.1 (0.005)* | 5 |
| | | Toluene | 0.1 (0.005)* | 5 |
| | | 1,1,1-Trichloroethane | 0.1 (0.005)* | 5 |
| | | 1,1,2-Trichloroethane | 0.1 (0.005)* | 5 |
| | | Trichloroethene | 0.1 (0.005)* | 5 |
| | | Vinyl acetate | 0.1 (0.010)* | 10 |
| | | Vinyl chloride | 0.2 (0.010)* | 10 |
| | | Total xylenes | 0.1 (0.005)* | 10 |

* Soil limits are by methanol extraction and are 20 times the water values, in µg/kg. Based on 10 g sample plus 10 mL methanol with 250 µL aliquot spiked in 5 mL and purged. Lower limits are available by direct sparge. The limits would be the same as the water, except in µg/kg.

* Exceeds 5/91 IRP limits, shown in parentheses.

SW = Methods from SW846, Third Edition.

**TABLE 8-12. REPORTING LIMITS FOR METHOD SW8270 SEMIVOLATILE ORGANIC COMPOUNDS**

| Method | Parameter | Analytes | Reporting Limits Soil[a] (mg/kg) | Reporting Limits Water (µg/L) |
|---|---|---|---|---|
| **Base/Neutral Extractables** | | | | |
| SW8270 | Semivolatile organic compounds | Acenaphthene | 0.33 (0.3)* | 10 |
| | | Acenaphthylene | 0.33 (0.3)* | 10 |
| | | Anthracene | 0.33 (0.3)* | 10 |
| | | Benzo(a)anthracene | 0.33 (0.3)* | 10 |
| | | Benzo(b)fluoranthene | 0.81 (0.3)* | 10 |
| | | Benzo(k)fluoranthene | 0.82 (0.3)* | 10 |
| | | Benzo(ghi)perylene | 0.33 (0.3)* | 10 |
| | | Benzo(a)pyrene | 0.33 (0.3)* | 10 |
| | | Benzyl alcohol | 0.33 (0.3)* | 10 |
| | | bis(2-Chloroethoxy)methane | 0.40 (0.3)* | 10 |
| | | bis(2-Chloroethyl)ether | 0.40 (0.3)* | 10 |
| | | bis(2-Chloroisopropyl)ether | 0.40 (0.3)* | 10 |
| | | bis(2-Ethylhexyl)phthalate | 0.40 (0.3)* | 10 |
| | | 4-Bromophenyl phenyl ether | 0.33 (0.3)* | 10 |
| | | Butyl benzyl phthalate | 0.33 (0.3)* | 10 |
| | | 4-Chloroaniline | 0.40 (0.3)* | 10 |
| | | 2-Chloronaphthalene | 0.70 (0.3)* | 20 (10)* |
| | | 4-Chlorophenyl phenyl ether | 0.33 (0.3)* | 10 |
| | | Chrysene | 0.33 (0.3)* | 10 |
| | | Dibenz(a,h)anthracene | 0.33 (0.3)* | 10 |
| | | Dibenzofuran | 0.33 (0.3)* | 10 |
| | | Di-n-butylphthalate | 0.40 (0.3)* | 10 |
| | | 1,2-Dichlorobenzene | 0.20 (0.3)* | 10 |
| | | 1,3-Dichlorobenzene | 0.20 (0.3)* | 10 |
| | | 1,4-Dichlorobenzene | 0.20 (0.3)* | 10 |
| | | 3,3'-Dichlorobenzidine | 1.6 (0.6)* | 20 |
| | | Diethyl phthalate | 0.33 (0.3)* | 10 |
| | | Dimethyl phthalate | 0.33 (0.3)* | 10 |
| | | 2,4-Dinitrotoluene | 0.33 (0.3)* | 10 |
| | | 2,6-Dinitrotoluene | 0.33 (0.3)* | 10 |
| | | Di-n-octylphthalate | 0.33 (0.3)* | 10 |
| | | Fluoranthene | 0.33 (0.3)* | 10 |
| | | Fluorene | 0.33 (0.3)* | 10 |
| | | Hexachlorobenzene | 0.33 (0.3)* | 10 |
| | | Hexachlorobutadiene | 0.33 (0.3)* | 10 |
| | | Hexachlorocyclopentadiene | 0.33 (0.3)* | 10 |
| | | Hexachloroethane | 0.33 (0.3)* | 10 |

(Continued)

TABLE 8-12. (Continued)

| Method | Parameter | Analytes | Reporting Limits Soil[a] (mg/kg) | Reporting Limits Water (µg/L) |
|---|---|---|---|---|
| **Base/Neutral Extractables (Continued)** | | | | |
| | | Indeno(1,2,3-cd)pyrene | 0.40 (0.3)* | 10 |
| | | Isophorone | 0.33 (0.3)* | 10 |
| | | 2-Methylnaphthalene | 0.70 (0.3)* | 20 (10)* |
| | | Naphthalene | 0.33 (0.3)* | 10 |
| | | 2-Nitroaniline | 1.7 (1.6)* | 50 |
| | | 3-Nitroaniline | 1.7 (1.6)* | 50 |
| | | 4-Nitroaniline | 1.7 (1.6)* | 50 |
| | | Nitrobenzene | 0.33 (0.3)* | 10 |
| | | n-Nitrosodiphenylamine | 0.89 (0.3)* | 10 |
| | | n-Nitrosodipropylamine | 0.70 (0.3)* | 20 (10)* |
| | | Phenanthrene | 0.33 (0.3)* | 10 |
| | | Pyrene | 0.40 (0.3)* | 10 |
| | | 1,2,4-Trichlorobenzene | 0.33 (0.3)* | 10 |
| **Acid Extractables** | | | | |
| | | Benzoic acid | 2.0 (1.6)* | 50 |
| | | 4-Chloro-3-methylphenol | 0.33 (0.3)* | 10 |
| | | 2-Chlorophenol | 0.40 (0.3)* | 10 |
| | | 2,4-Dichlorophenol | 0.40 (0.3)* | 10 |
| | | 2,4-Dimethylphenol | 0.40 (0.3)* | 10 |
| | | 4,6-Dinitro-2-methylphenol | 2.0 (1.6)* | 50 |
| | | 2,4-Dinitrophenol | 1.7 (1.6)* | 50 |
| | | 2-Methylphenol | 0.33 (0.3)* | 10 |
| | | 4-Methylphenol | 0.33 (0.3)* | 10 |
| | | 2-Nitrophenol | 0.40 (0.3)* | 14 (10)* |
| | | 4-Nitrophenol | 1.7 (1.6)* | 50 |
| | | Pentachlorophenol | 1.0 | 30 |
| | | Phenol | 0.40 (0.3)* | 10 |
| | | 2,4,5-Trichlorophenol | 1.6 | 50 |
| | | 2,4,6-Trichlorophenol | 0.33 (0.3)* | 10 |

[a] Reporting limits for soil are based on the extraction of 10 g of sample and are approximately 100 times those for water. In some cases, lower detection limits may be achieved by extracting 30 g of sample. Gel permeation chromatograph (GPC) cleanup of samples, if necessary, raises detection limits twofold.

* Exceeds 5/91 IRP limit, shown in parentheses.

SW = Methods from SW846, Third Edition.

**TABLE 8-13. REPORTING LIMITS FOR METHOD SW8280 DIOXINS/FURANS**

| Method | Parameter | Analytes | Reporting Limits[a] Soil (ng/g)[b] | Reporting Limits[a] Water (ng/L)[c] |
|---|---|---|---|---|
| SW8280 | Dioxins/Furans | HpCDD | 1.87 (.01)* | 10 |
| | | HpCDF | -- (.01)* | 10 |
| | | HxCDD | 1.25 (.01)* | 10 |
| | | HxCDF | 0.83 (.01)* | 10 |
| | | OCDD | 2.35 (.01)* | 10 |
| | | OCDF | -- (.01)* | 10 |
| | | PeCDD | 0.70 (.01)* | 10 |
| | | PeCDF | 0.33 (.01)* | 10 |
| | | TCDD | 0.17 (.01)* | 10 |
| | | 2,3,7,8-TCDD | 0.17 (.01)* | 10 |
| | | TCDF | 0.11 (.01)* | 10 |

[a] Assuming 100% surrogate recovery.
[b] Using 25 g sample.
[c] Using one-liter sample.

* Exceeds 5/91 IRP limit, shown in parentheses.

SW = Methods from SW846, Third Edition.

**TABLE 8-14. REPORTING LIMITS FOR METHOD SW8310 POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC**

| Method | Parameter | Analytes | Reporting Limits Soil (mg/kg) | Reporting Limits Water (µg/L) |
|---|---|---|---|---|
| SW8310 | Polynuclear aromatic hydrocarbons | Acenaphthene | 1.0 | 18 |
| | | Acenaphthylene | 1.5 | 23 |
| | | Anthracene | 0.5 | 7 |
| | | Benzo(a)anthracene | 0.01 | 1 |
| | | Benzo(a)pyrene | 0.01 | 1 |
| | | Benzo(b)fluoranthene | 0.01 | 1 |
| | | Benzo(g,h,i)perylene | 0.01 | 1 |
| | | Benzo(k)fluoranthene | 0.01 | 1 |
| | | Chrysene | 0.1 | 1.5 |
| | | Dibenzo(a,h)anthracene | 0.01 | 1 |
| | | Fluoranthrene | 0.1 | 2 |
| | | Fluorene | 0.1 | 2 |
| | | Indeno(1,2,3-cd)pyrene | 0.01 | 1 |
| | | Naphthalene | 1.0 | 18 |
| | | Phenanthrene | 0.4 | 6 |
| | | Pyrene | 0.2 | 3 |

SW = Methods from SW846, Third Edition.

## TABLE 8-15. REPORTING LIMITS FOR OTHER METHODS

| Method | Parameter | Analytes | Reporting Limits | |
|---|---|---|---|---|
| | | | Soil | Water |
| E504 | 1,2-Dibromomethane (EDB) | EDB | -- | 0.05 µg/L |
| E607 | Nitrosamines | n-Nitrosodiphenylamine | -- | 0.8 µg/L |
| HML338 | Organic lead compounds | | 0.8 mg/kg | 0.2 mg/L |
| SW1310 | Extraction procedure toxicity (metals) | Arsenic | -- | 5.0 mg/L |
| | | Barium | -- | 100.0 mg/L |
| | | Cadmium | -- | 1.0 mg/L |
| | | Chromium | -- | 5.0 mg/L |
| | | Lead | -- | 5.0 mg/L |
| | | Mercury | -- | 0.2 mg/L |
| | | Selenium | -- | 1.0 mg/L |
| | | Silver | -- | 5.0 mg/L |
| E901.1 | Radioactivity Gamma | | | a |
| SW9310 | Radioactivity Gross alpha and beta | | -- | 4 pCi/L[a] |
| SW9060 | Total organic carbon | | | 1.0 mg/L |

[a] Sample quantitation is based on the counting period and required sensitivity of the measurement.

E = Methods from United States Environmental Protection Agency, 1983.
SW = Methods from SW846, Third Edition.

**TABLE 8-16. REPORTING LIMITS FOR DOWNHOLE SOIL GAS**

| Compound | OU B RI RL (ppbv) |
|---|---|
| Vinyl chloride | 20 |
| trans-1,2-Dichloroethene | 10 |
| cis-1,2-Dichloroethene | 10 |
| Chloroform | 10 |
| 1,1,1-Trichloroethene | 10 |
| Trichloroethene | 10 |
| Tetrachloroethene | 10 |
| Benzene | 10 |
| Toluene | 10 |
| p & m-Xylenes | 20 |
| o-Xylene | 20 |

ppbv = Parts per billion by volume.
RL = Reporting limit.

## TABLE 8-17. REPORTING LIMITS FOR ON-SITE VOC FIELD SCREENING

| Compound | Reporting Limit (mg/kg) |
|---|---|
| Vinyl chloride | 0.015 |
| trans-1,2-Dichloroethene | 0.002 |
| cis-1,2-Dichloroethene | 0.002 |
| Chloroform | 0.002 |
| 1,1,1-Trichloroethene | 0.003 |
| Trichloroethene | 0.002 |
| Tetrachloroethene | 0.002 |
| Benzene | 0.002 |
| Toluene | 0.005 |
| p & m-Xylenes | 0.003 |
| o-Xylene | 0.002 |

VOC = Volatile organic compound.
mg/kg = Milligrams per kilogram of soil (wet weight).

# 9.0 DATA REDUCTION, VALIDATION, AND REPORTING

The data reduction, validation, and reporting procedures described in this section will ensure that complete documentation is maintained, that transcription and data reduction errors are minimized, the data are reviewed and documented, and the reported results are properly qualified. Laboratories are required to have established procedures to conduct data review, validation, and reporting. The specific procedures and assigned personnel vary; however, equivalent laboratory data reduction and verification procedures are required to ensure the overall objectives of analysis and reporting according to method and project specifications are achieved. The contracting laboratories will be required to provide documentation to support their procedures, which will be verified during technical systems audits.

## 9.1 Laboratory Data Reduction and Validation

The laboratory analyst for both on-site and off-site analyses is responsible for the reduction of raw data generated at the laboratory bench. The data interpretation that is required to calculate sample concentrations follows the methodology described in the specific analytical standard operating procedure (SOP). After all analyses have been completed and reported, the laboratory supervisor reviews the raw data and verifies that the analyses were properly performed and reported. These reviews are performed according to standardized procedures for each laboratory and type of analysis. Raw data, together with all supporting documentation, are stored permanently in confidential files maintained in the laboratory.

After all analyses in a reporting batch (often called a work order or log order) are complete, the data are entered into the laboratory reporting system and a preliminary report is generated for review by the laboratory supervisors. This review is followed by a quality check carried out by a designated quality control (QC) reviewer to verify that the QC meets the specifications of the method and project. At this point, the laboratory review and validation process is complete.

Identification of outliers is also a part of the data review. An outlier is an unusually large (or small) value in a set of observations. There are many possible reasons for outliers including:

- Faulty instruments or component parts;
- Inaccurate reading of a record, dialing error, etc.;
- Errors in transcribing data; or
- Calculation errors.

Sometimes analysts or operators can identify outliers by noting the above types of occurrences when they record observations. In these instances, the errors are corrected, or if correction is not possible, the suspect observations may be removed from the data before calculations are performed. If no such information exists, statistical tests may be used to test suspected outliers. Outliers identified by these tests method may be removed from the data before further processing. The presence of outliers may indicate a systematic problem that must be identified. This problem may require corrective action, which should be initiated by the quality assurance (QA) coordinator or laboratory supervisor.

## 9.2 Project Data Flow, Transfer, and Verification

Data flow from the laboratory and field to the project staff and data users follows established procedures to ensure that data are properly tracked, reviewed, and validated for use. The first step in the data flow is QC review. Laboratory results for Level 2 and Level 3 analyses are reviewed by designated QC task members to ensure accurate reporting and adherence to project and method requirements. For large projects (e.g., the OU B RI), a pre-designated percentage (e.g., 10%) of all reports will be reviewed for correct sample identification, dates, reporting limits, and flags. If systematic errors or frequent occurrence of random errors are observed, successively higher percentage of reports will be reviewed. The review percentage will be noted in the Scope of Work (SOW) and task instructions for the project. In all cases, holding time and batch QC results will be reviewed according to the procedure described in Section 9.3. After QC review and approval, the electronic data are uploaded to the project database. After the initial upload, reports are produced for QC review by comparison with the hard-copy results.

Corrections or edits may be handled by requesting reissuance of laboratory reports and/or data files, or by providing the data management staff with the required changes. The correction mechanism depends upon the types of corrections or changes needed. Analytical information request forms or database amendment forms are used to request these changes and track the status of each request. Field data are transferred to the

project database by downloading the electronic master log files on a weekly basis or by data entry of the field logbook information. The field data are verified after entry into the field computer by comparison with field data sheets and notebooks. After the field and laboratory results have been validated and loaded into the project database, they are accessible to data users via standard and custom reports. Electronic files may be generated for use in mapping or geographic information system (GIS) applications.

For Level 3 data, a Laboratory Information Management System (LIMS) is used to store, transfer, and report analytical data. A series of programs allows electronic transfer of data to the project database. The laboratory is responsible for generating hard copies and electronic files of the analytical results.

Both the analytical reports and electronic data files are transferred to designated project QC task members and/or data management staff. The laboratory provides additional documentation regarding chain-of-custody procedures, etc., that are not transmitted electronically. The information on the floppy disks is uploaded to the project database by using a set of programs to read, check, and append the analytical results to the database; data received via hard copy only are manually entered. The analytical reports are used to verify the electronic transfer process, and are then permanently stored in project files. Figure 9-1 is an example of the SAM® data sheet, one type of analytical report used for reporting analytical data. The specific information and electronic file formats are established and tested prior to analysis of any samples to ensure that the formats will be compatible with the existing project database, and that all required information is reported.

**Manual Data Entry Procedures**

Manual entry of field data and any data received from analytical laboratories that were not provided electronically will be required. Data entry programs for each type of data request the information for each record. A range check is conducted for numeric parameters, if appropriate, and the operator corrects any errors identified by the check. The records are printed, and must be verified by the person requesting the data entry before the new records are appended to the database.

Page 21 RAL-Sacramento REPORT Work Order # 90-05-008
Received: 05/02/90 Results by Sample

SAMPLE ID WELL-8A FRACTION 05A TEST CODE 8025W001 NAME Aromatics by SW8020 (1)
LIST Complete analyte list
Date & Time Collected 05/01/90 Category

Analyst BSJ Prepared File ID E805085
Instrument 8 Analyzed 05/08/90 Blank ID R805081
Matrix Water Factor 1 Report as received VER RY
Units ug/L

| Alt ID | ANALYTE | Result | Det Lim | Factor |
|---|---|---|---|---|
| 71-43-2 | Benzene | <0.20 | 0.20 | 1 |
| 108-90-7 | Chlorobenzene | <0.20 | 0.20 | 1 |
| 95-50-1 | 1,2-Dichlorobenzene | <0.40 | 0.40 | 1 |
| 541-73-1 | 1,3-Dichlorobenzene | <0.40 | 0.40 | 1 |
| 106-46-7 | 1,4-Dichlorobenzene | <0.30 | 0.30 | 1 |
| 100-41-4 | Ethylbenzene | <0.20 | 0.20 | 1 |
| 108-88-3 | Toluene | <0.20 | 0.20 | 1 |
| 1330-20-7 | Xylenes (total) | <0.20 | 0.20 | 1 |

| Surrogates | % Recovery | Sur Limits |
|---|---|---|
| 1-Bromo-4-fluorobenzene | 93 | 40- 140 |

(1) See Appendix A for glossary of report and data definitions

**Figure 9-1. Example of SAM® Data Sheet**

## 9.3 Data Validation

The project QC task members will review field documentation and measurement data for acceptable sample collection and analysis procedures, consistency with expected results or other results, adherence to prescribed QA procedures, and agreement with the acceptance criteria described in Section 4.0. A QA SOP for data validation has been developed to ensure that consistent review procedures are used (see Appendix A).

Initially, the reviewer will determine whether holding times were met, and that all required analytical QC checks were performed and reported with the data. Then all QC sample results will be reviewed to evaluate the sampling and analytical performance. Reagent and field blank results will be evaluated to identify any systematic contamination; spike and duplicate results will be compared to the QA objectives presented in Section 4.0, and the results used to calculate precision and accuracy for the data set. This process will identify analytical methods and compounds for which the QA objectives are not satisfied, and corresponding sample data will be evaluated and qualified with a "flag" indicating the problem, as appropriate. Samples collected on the same day, or analyzed in the same run or batch, or individual samples may be flagged, depending on the type of problem that has been identified. Re-analysis or resampling may be recommended as a corrective action at this time if data are determined to be unacceptable for the intended application. Corrective actions and data assessment procedures are described in the SOP, and in Sections 13.0 and 14.0.

Laboratory control charts may be reviewed for compliance with established criteria. Control chart review will also be requested periodically during site visits or audits. Control charts or control limits for field duplicate results can be developed by the project staff to document systematic precision for a method.

The qualifier codes, or "flags," will be stored with the data in the McClellan AFB database. The specific statistical procedures and qualifier codes used in the validation process are described in detail in Section 13.0. After data are received from the laboratory, entered, checked, and qualified, they are a permanent part of the database and cannot be altered without documenting the changes requested and the reason for the change.

## 9.4 Reporting

Data reporting for McClellan AFB projects will consist of Quality Assurance Reports (refer to Section 15.0), investigative data reporting, QC data reporting, Installation Restoration Program (IRP) reporting, and Installation Restoration Program Information Management System (IRPIMS) support.

General reporting practices for measurement data will include:

- Heading information identifying the sample ID and the analytical method;
- Unique sample identification number or code;
- Consistent units of measure;
- Consistent number of significant figures;
- No blank or dashed places reported; all spaces will contain a designation (i.e., not analyzed, not sampled, etc.);
- Explanation of outlier values or the cause for deviation from historical data;
- Comparison with regulatory threshold values if applicable;
- Quality assurance flags; and
- Quantification of accuracy and precision for analytical data.

### Investigative Data Reporting

Measurement data generated during the course of an investigation will be reported in tabular form from the computerized database. The formats of the reports will vary, depending on the objectives of the investigation. In general, data will be presented

according to sampling location, analytical method, parameter, and/or matrix, and the reporting units will be specified. Data qualifier flags can be reported on the tables, and qualified data tables are provided for reference by data users. Commonly used reporting formats will be catalogued and used repeatedly. Specialized formats will be developed as needed.

**Installation Restoration Program Information Management System (IRPIMS)**

The data collected during past and ongoing efforts for the McClellan AFB Remedial Investigation/Feasibility Study (RI/FS) will also be provided in a format compatible with the IRPIMS as requested. The IRPIMS is a data management system designed to accommodate all types of data collected for IRP RI/FS programs. Specific codes and data forms have been developed to allow consistent and efficient input of information to the system. The McClellan AFB database information will be provided to the U.S. Air Force via ASCII files in specified IRPIMS formats on floppy diskettes or 9-track tapes. The information transferred will include all required technical data such as site information, well characteristics, hydrogeologic, geologic, and physical and chemical analysis results. Submission of data to the IRPIMS will be specified in the SOW for each field activity. The database contains only validated data; therefore, the review and error checking procedures for production of IRPIMS files will consist of format and completeness checks.

**General Reporting Procedures**

The procedures employed to ensure general reporting quality are:

- Calculation checks for any field calculations and measurements are verified by recalculation by the person initially providing data. All calculations and measurements are then checked by another individual. The calculations are documented on a formatted calculation sheet that has headings for identifying the calculation activity, project, individual performing the work, and sign-off blocks for the reviewers. Any calculations and measurements that differ from the initial totals are resolved by both individuals. Once the calculations and measurements are included in an internal working copy of a document, they are rechecked during peer review. If a systematic calculation error is

identified, corrective action will be initiated by the reviewer, and will be documented using corrective action procedures presented in Section 15.0.

- Numerical values presented in reports and comparisons of numbers appearing in text, tables, and appendices are addressed in the manner discussed above. Technical editors check all reports for proper format, grammatical correctness, and correspondence between text references to figures, tables, sections, or appendix labels.

**QC Data Reporting**

QC results will be reported by sample matrix and analytical method in tabular form. How these QC results influence the measurement data will be delineated. For example, matrix spike interference will influence specific samples or matrices, while laboratory blank contamination will influence all samples extracted or analyzed on a specific day or during a specific analytical run.

In cases where there are a large number of QC analyses of one type, a second level, or summary, table may be constructed. The summary tables will typically report mean or pooled statistics to describe the overall performance of the method. For example, the summary table of duplicate sample results might report the average RPD for all duplicates measured for the compound, and indicate the number of individual RPDs that did not meet the acceptance criteria. This type of table can serve as an indication of the overall QC results. However, these applications will often have to be developed or modified from existing programs for individual investigations. A summary assessment of the data presented in these tables will be prepared for each phase of sampling, or specific RI/FS task, as appropriate.

Finally, custom table formats will be used as an aid to interpretation of the investigative data. The particular format will depend on how the QC results are expected to influence the investigative data and will be developed by data management staff through discussion with the users. This type of table might be used to identify corresponding investigative results (samples analyzed on corresponding dates), which may be inaccurate. Specialty tables will be generated automatically or manually, depending on the volume of data to be processed and the complexity of the calculations.

**Archiving**

Hard-copy and electronic data are archived in project files and on electronic archive tapes. Hard-copy data are filed by field event, site, and/or analytical batch, depending upon the type of project. Electronic backups of the computer system are performed regularly. Daily tape backups are automatically performed for changes made that day, and weekly and monthly tape backups of the entire system are performed. Data files provided by the laboratories are maintained in the project files.

# 10.0 INTERNAL QUALITY CONTROL

Internal quality control (QC) is achieved by collecting and/or analyzing a series of duplicate, replicate, blank, spike, and spike duplicate samples to ensure that the analytical results are within quality control limits specified for the program. Laboratory QC samples are documented at the bench, and reported with the analytical results. Field QC samples will be documented in field logbooks and submitted "blind" to the laboratory. The QC sample results are used to quantify precision and accuracy, and identify any problems or limitations in the associated sample results. These components of the sampling program will ensure that data of known quality are produced throughout sampling and analysis field programs.

## 10.1 Analytical Laboratory Quality Control Samples

Laboratory QC is necessary to control the analytical process, to assess the accuracy and precision of analytical results, and to identify assignable causes for atypical analytical results. The QC checks in the laboratory protocol are specific to the analytical method and generally include the use of the following QC samples as appropriate for the method. The specific analytical QC samples, acceptance criteria and corrective actions for each method are presented in Tables 10-1 through 10-18 (at the end of this section), and in method standard operating procedures (SOPs). All laboratories performing the method are required to adhere to these procedures.

### Calibration Standards

Initial calibration is performed as required for each analytical method, usually using a range of calibration standards with the low standard near the detection limit for the compound. These standards are used to determine the linear dynamic range for the initial instrument calibration. Calibration is discussed in more detail in Sections 7.0 and 8.0.

### Laboratory Control Samples

Laboratory Control Samples (LCS) are blank (reagent water) spikes containing analytes of interest at a specified concentration, usually in the mid-calibration range. This type of spike is sometimes referred to as a method spike. These samples are carried through

the entire sample preparation and analysis process and are used to demonstrate that the method/instrument is operating within acceptable accuracy limits. Laboratory Control Samples are required for all analytical methods performed in the laboratory, and their preparation and the required frequency of analysis is described in each analytical SOP.

**Reagent Blanks**

A reagent blank is a sample composed of all the reagents (in the same quantities) used in preparing a sample for analysis. It is carried through the same sample preparation (digestion, extraction) procedure as a sample. As stated in the SW846 Method 8000 procedures, reagent blanks are used to ensure that interferences from the analytical system, reagents, and glassware are under control. The required frequency for analyzing reagent blanks is specified in the analytical SOP for each method, and generally consists of one per day for each method/instrument and/or one per extraction batch.

For gas phase analyses reagent or method blanks are analyzed. This type of reagent blank is a sample of ultrapure nitrogen gas that is carried through the entire analytical scheme. The $N_2$ blank volume is roughly equal to 1 liter and is from the same nitrogen source that is used to dilute the samples. A reagent blank analysis is performed daily or immediately after analysis of any sample exhibiting a single peak area exceeding $10^6$ area counts or other criterion specified in the SOP. Reagent blanks are analyzed until the total nonmethane hydrocarbon (TNMHC) value is less than 25 ppbv-c.

**Matrix Spike/Matrix Spike Duplicates**

A matrix spike is a solution of target analytes at known concentrations that is spiked into a field sample before sample preparation and analysis. Two aliquots of the sample are spiked for the duplicate analysis. The results of the analysis of the duplicate spiked samples are used to measure the percent recovery of each spiked compound, and to compare the recovery between samples, which provides estimates of the bias due to matrix interferences (based on spike recoveries) and precision of the method. The calculations for spike recovery and precision are outlined in Section 13.0, and the quality assurance objectives for accuracy are listed in Tables 4-1 through 4-13. The frequency for matrix spike analysis is 5 percent of samples analyzed for each method where spikes are performed.

### Surrogate Spikes

Surrogate spikes are a group of compounds that are not otherwise found in nature but behave similarly to the target analytes that have been selected for each organic analytical method. A solution of known concentration is prepared and spiked into each sample prior to sample preparation and analysis. The recovery of the surrogate spike compounds is reported for each sample, and the results are compared to the recovery objectives established for the method. Surrogate spike results provide a measure of instrument performance method, and also an indication of the presence of matrix effects.

### Laboratory Duplicates (Duplicate Analyses)

Laboratory duplicates are repeated but independent determinations of the same sample, by the same analyst, at essentially the same time and under the same conditions. The sample is split in the laboratory and each fraction is carried through all stages of sample preparation and analysis. Duplicate analyses measure the precision of each analytical method. The method of calculation for precision is outlined in Section 13.0. Laboratory duplicate analyses are performed for 10 percent of samples analyzed, where required by the method.

Tables 10-1 through 10-18 summarize the specific internal QC checks performed as required for the analytical method. These tables also include information relating to the initial calibration and ongoing calibration checks.

## 10.2 Field Quality Control Samples

### Field Duplicate Samples

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate sample results are used to assess precision, including variability associated with both the laboratory analysis and the sample collection process. Duplicate samples will be collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis.

Precision of soil samples to be analyzed for volatile organic compounds (VOCs) will be assessed from co-located samples because the compositing process required to obtain uniform samples could result in loss of the compounds of interest. The co-located samples will otherwise be handled and analyzed in the same manner. Soil samples to be analyzed for nonvolatile compounds will be recovered by collecting a single sample, and dividing it into equal portions for laboratory analysis or by collecting co-located samples if there is a large volume of soil required for analysis.

Recovery and analysis of 10 percent of samples collected for analysis by each method will be performed. The sample containers will be assigned a control number such that they cannot be identified (blind duplicate) as duplicate samples by laboratory personnel performing the analysis. Specific locations will be designated for collection of field duplicate samples by QC staff prior to beginning sample collection.

**Ambient Blank**

Ambient blanks are samples of organic-free water that are collected and processed using the same sampling and handling procedures as other samples. Ambient blanks are used to assess the potential introduction of contaminants from ambient sources to the samples during sample collection, and are prepared only for VOC samples. The frequency requirements and locations for collecting ambient blanks will be specified in the scope of work and field task instructions for each sampling and analysis task. Organic-free water will be prepared with Type II water that has been filtered, deionized, and boiled to volatilize organic compounds. This water is then continuously purged with nitrogen to prevent reentry of volatile organic compounds. Water to be used for the sensitive gas chromatography analysis (SW8010 and SW8020) will also be boiled for at least 20 minutes and kept under positive pressure by purging with nitrogen. This water is tested by GC analysis prior to its use in the field blanks to ensure complete purity.

**Equipment Blanks**

Equipment blanks consist of a sample of Type II reagent water (for inorganic and semivolatile analyses) or organic-free water (for volatile organic analyses) poured into the groundwater sampling device, collected in the sample bottle, and transported to the laboratory for analysis. The frequency requirements for collecting equipment blanks will be

specified in the scope of work and field task instructions for each sampling and analysis task. Equipment blanks will be recommended for equipment that has been decontaminated after sampling a contaminated well or site, and periodically throughout the sampling effort.

**Trip Blanks**

A trip blank is a sample of organic-free water (prepared as for ambient blanks) that is placed in the sample bottle in an uncontaminated area in the laboratory prior to going in the field. Trip blanks are prepared only for VOC samples and are subjected to the same handling as other samples. Trip blanks serve to identify contamination from sample containers or transportation and storage procedures. Trip blanks consisting of unopened evacuated stainless steel canisters will be used during gas phase sampling. One trip blank per batch of samples will be collected for methods that analyze for the presence of VOCs.

**Field Blanks for Gas Phase Samples**

A field blank is a sample of UHP air collected in the field. Field blanks are collected in stainless steel canisters in association with gas phase sampling programs. The field blanks are collected and processed using the same sampling and handling procedures as field samples, and the results are used to identify sources of contamination in sample collection and handling procedures. Field blanks are collected at a frequency of 10 percent of the total number of field samples.

## TABLE 10-1. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8010

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8010 | Halogenated Volatile Organics | Five-point calibration (for all analytes) | Initial calibration prior to sample analysis | a) RSD <20% for RFs or r ≥ 0.995 for linear regression<br>b) For quantitation, use average RF if RSD <20%, otherwise use linear regression calibration factor (CF) | Repeat after corrective action |
| | | Second source calibration verification (for all analytes) | Once per five-point calibration | All analytes within ±15% of average RF or CF | 1) Reanalyze second source standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | Refer to:<br>SW846-3500[a]<br>SW846-5030[a]<br>SW846-8000[a]<br>IRP Handbook[b] | Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample | Once per analyst | Analyte-specific limits as per SW846, Table 6 | 1) Recalculate results<br>2) Locate and fix problem with system<br>3) Rerun demonstration for those analytes which did not meet criteria |

(Continued)

TABLE 10-1. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8010 (Cont'd) | | Initial daily calibration factor verification | Daily, before sample analysis | Response for any analyte within ±15% of the average RF or CF | 1) Perform system maintenance as per SW846-8000<br>2) Prepare new calibration curve for any analyte not meeting criteria<br>3) Reinject any samples analyzed after criteria were exceeded |
| | | Retention time (RT) windows calculated for each analyte | One 72-hour study performed on each GC column and whenever a new column is installed | 1.5% for Megabore, 2.0% for packed columns[d] | 1) Perform maintenance<br>2) Repeat test |
| | | Mid-level Laboratory Control Sample (LCS) (see Section 4.0 tables for list of analytes spiked) | Daily (as per instrument calibration requirements in IRP Handbook[b]). Must be run prior to samples | Response for any analyte within project limits | 1) Repeat LCS<br>2) Repeat daily calibration |
| | | Ongoing calibration check | 10% sample frequency, minimum two per set | a) RF within ±15% of daily calibration factor<br>b) Ongoing calibration analytes elute within daily RT windows | 1) Locate and correct source of problem--document actions taken<br>2) Repeat test only for analytes that failed to meet criteria |

(Continued)

TABLE 10-1. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8010 (Cont'd) | | | | | 3) After repeated failures, perform system maintenance as per SW846-8000<br>4) Repeat test for all compounds of interest |
| | | Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration (if applicable), or 2) concentration of the ongoing calibration check | 1 MS and 1 MSD per every 20 Air Force project samples | a) Laboratory-established criteria as per SW846-8000 (see tables in Section 4.0 of QAPP), or<br>b) QC Acceptance Criteria Table, SW846-8010 | 1) Flag data<br>2) Analyze a QC check standard containing each analyte that failed criteria<br>3) If recovery for QC check standard is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required<br>4) Reanalyze any samples affected by out-of-control condition |

(Continued)

TABLE 10-1. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8010 (Cont'd) | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[a] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | NA | NA |
| | | Surrogate standard spike | Every sample spike, standard, and reagent blank | Limits determined as per SW846-8000 (limits are to be updated annually on a matrix-by-matrix basis) | 1) Recalculate result, if still out:<br>2) Reanalyze sample, if still out:<br>3) Flag result and document in report that steps 1 and 2 were performed |
| | | Reagent blank | One per day/ instrument | No analytes detected at >3 times the detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br>3) All samples processed with a contaminated blank are to be repurged and reanalyzed at no cost to the Air Force |

(Continued)

TABLE 10-1. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8010 (Cont'd) | | | | | 4) Sample results associated with reagent blank contamination at $\leq$3 times the detection limit are to be flagged |
| | | Second-column confirmation, to meet IRP[b] requirements | 100% for all positive results except noted common laboratory contaminants[c] (also see footnote "f") | Analysis of standards and samples performed on a second GC column of dissimilar phase and retention characteristics within specified holding times | Resampling and reanalysis performed at no cost to government, even if first column analysis was conducted within holding time[b] |
| | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits that exceeded established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

(Footnotes on next page)

**TABLE 10-1. (Continued)**

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as is specified in the IRP Handbook.
[d] CLP SOW 3/90.
[e] Methylene chloride, acetone, toluene, and 2-butanone are considered to be common laboratory contaminants. Therefore, it is not required that second-column confirmation be performed for the sole purpose of confirming the presence of any one of these four analytes.
[f] For quarterly sampling, second-column confirmation is not required for sample if all detected compounds were confirmed at least once in previous two quarters.

TABLE 10-2. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8015M - VOLATILE

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8015M (Modified) | Total Petroleum Hydrocarbons - Volatile | Five-point calibration (for all analytes) | Initial calibration prior to sample analysis | a) RSD < %20 for RFs or r ≥ 0.995 for linear regres-sion<br>b) For quantitation, use average RF if RSD <20%, otherwise use linear regression calibration factor (CF) | Repeat after corrective action |
| | | Independently prepared calibration verification (for all analytes) | Once per five-point calibration | All analytes within ±15% of average RF or CF | 1) Reanalyze second source standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | Refer to: LUFT Field Manual (State of California, October 1989) SW846-3500[a] SW846-5030[a] SW846-8000[a] IRP Handbook[b] | Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample | Once per analyst | Laboratory established limits | 1) Recalculate results<br>2) Locate and fix problem with system<br>3) Rerun demonstra-tion for those analytes which did not meet criteria |

(Continued)

TABLE 10-2. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8015M (Cont'd) | | Initial daily calibration factor verification using one or more calibration standards | Daily, before sample analysis | Response for any analyte within ±15% of average RF or CF | 1) Perform system maintenance as per SW846-8000<br>2) Prepare new calibration curve for any analyte not meeting criteria<br>3) Reinject any samples analyzed after criteria were exceeded |
| | | Retention time (RT) windows calculated for each analyte (BTEX) | One 72-hour study performed on each GC column and whenever a new column is installed | 1.5% for Megabore, 2.0% for packed columns[d] | 1) Perform maintenance<br>2) Repeat test |
| | | Extracted mid-level Laboratory Control Sample (LCS) for BTEX | Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence | Response for any analyte within ±50% of predicted response until laboratory acceptance limits can be established | 1) Repeat LCS<br>2) Repeat daily calibration |

(Continued)

TABLE 10-2. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8015M (Cont'd) | | Ongoing calibration check | 10% sample frequency, minimum of two per set | a) RF within ±15% of daily calibration factor<br>b) Ongoing calibration analytes elute within daily RT windows | 1) Locate and correct source of problem-document actions taken<br>2) Repeat test only for analytes that failed to meet criteria<br>3) After repeated failures, perform system maintenance as per SW846-8000<br>4) Repeat test for all compounds of interest |
| | | Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit (if applicable), or 2) concentration of the ongoing calibration check | 1 MS and 1 MSD per every 20 Air Force project samples | Laboratory established criteria as per SW846-8000 | 1) Flag data<br>2) Analyze a QC check standard containing each analyte that failed criteria<br>3) If recovery for QC check standard is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required<br>4) Reanalyze any samples affected by out-of-control condition |

TABLE 10-2. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8015M (Cont'd) | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[a] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | N/A | N/A |
| | | Surrogate standard spike | Every sample, spike, standard, and reagent blank | Limits determined as per SW846-8000 (limits are to be updated annually on a matrix-by-matrix basis) | 1) Recalculate result, if still out:<br>2) Reanalyze sample, if still out:<br>3) Flag result and document in report that steps 1 and 2 were performed |
| | | Reagent blank | One per day/ instrument | No analytes detected at >3 times the detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br>3) All samples processed with a contaminated blank are to be re-extracted/ repurged and re-analyzed at no cost to the Air Force |

(Continued)

TABLE 10-2. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8015M (Cont'd) | | | | | 4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |
| | | Second-column confirmation, to meet IRP requirements[b] | For all detectable BTEX results in the absence of a gasoline pattern | Analysis of standards and samples performed on a second GC column of dissimilar phase and retention characteristics within specified holding times | Resampling and reanalysis performed at no cost to government, even if first column analysis was conducted within holding time |
| | | MDL study | Once per year | See Table 8-6 | Detection limits established shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as is specified in the IRP Handbook.
[d] CLP SOW 3/90.

## TABLE 10-3. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8015M - EXTRACTABLE

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8015M (Modified) | Total Petroleum Hydrocarbons - Extractable | Five-point calibration (for all analytes) | Initial calibration prior to sample analysis | a) RSD < %20 for RFs or r ≥ 0.995 for linear regression<br>b) For quantitation, use average RF if RSD <20%, otherwise use linear regression calibration factor (CF) | Repeat after corrective action |
| | | Independently prepared calibration verification (for all analytes) | Once per five-point calibration | All analytes within ±15% of average RF or CF | 1) Reanalyze second source standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | Refer to:<br>LUFT Field Manual (State of California, October 1989)<br>SW846-3500[a]<br>SW846-5030[a]<br>SW846-8000[a]<br>IRP Handbook[b] | Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample | Once per analyst | Laboratory established limits | 1) Recalculate results<br>2) Locate and fix problem with system<br>3) Rerun demonstration for those analytes which did not meet criteria |

(Continued)

TABLE 10-3. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8015M (Cont'd) | | Initial daily calibration factor verification using one or more calibration standards | Daily, before sample analysis | Response for any analyte within ±15% of average RF or CF | 1) Perform system maintenance as per SW846-8000<br>2) Prepare new calibration curve for any analyte not meeting criteria<br>3) Reinject any samples analyzed after criteria were exceeded |
| | | Retention time (RT) windows calculated for each analyte (BTEX) | One 72-hour study performed on each GC column and whenever a new column is installed | 1.5% for Megabore, 2.0% for packed columns[d] | 1) Perform maintenance<br>2) Repeat test |
| | | Extracted mid-level Laboratory Control Sample (LCS) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence | Response for any analyte within ±50% of predicted response until laboratory acceptance limits can be established | 1) Repeat LCS<br>2) Repeat daily calibration |

(Continued)

TABLE 10-3. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8015M (Cont'd) | | Ongoing calibration check | 10% sample frequency, minimum of two per set | a) RF within ±15% of daily calibration factor<br>b) Ongoing calibration analytes elute within daily RT windows | 1) Locate and correct source of problem--document actions taken<br>2) Repeat test only for analytes that failed to meet criteria<br>3) After repeated failures, perform system maintenance as per SW846-8000<br>4) Repeat test for all compounds of interest |
| | | Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit (if applicable), or 2) concentration of the ongoing calibration check | 1 MS and 1 MSD per every 20 Air Force project samples | Laboratory established criteria as per SW846-8000 | 1) Flag data<br>2) Analyze a QC check standard containing each analyte that failed criteria<br>3) If recovery for QC check standard is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required<br>4) Reanalyze any samples affected by out-of-control condition |

(Continued)

TABLE 10-3. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8015M (Cont'd) | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[a] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | N/A | N/A |
| | | Surrogate standard spike | Every sample, spike, standard, and reagent blank | Limits determined as per SW846-8000 (limits are to be updated annually on a matrix-by-matrix basis) | 1) Recalculate result, if still out:<br>2) Reanalyze sample, if still out:<br>3) Flag result and document in report that steps 1 and 2 were performed |
| | | Reagent blank | One per day/ instrument | No analytes detected at >3 times the detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented |

(Continued)

TABLE 10-3. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8015M (Cont'd) | | | | | 3) All samples processed with a contaminated blank are to be re-extracted and re-analyzed at no cost to the Air Force<br>4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |
| | | MDL study | Once per year | See Table 8-6 | Detection limits established shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as is specified in the IRP Handbook.
[d] CLP SOW 3/90.

## TABLE 10-4. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8020

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8020 | Volatile Aromatics | Five-point calibration (for all analytes) | Initial calibration prior to sample analysis | a) RSD < %20 for RFs or r ≥0.995 for linear regression<br>b) For quantitation, use average RF if RSD <20%, otherwise use linear regression calibration factor (CF) | Repeat after corrective action |
| | | Second source calibration verification (for all analytes) | Once per five-point calibration | All analytes within ±15% of average RF or CF | 1) Reanalyze second source standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | Refer to:<br>SW846-3500[a]<br>SW846-5030[a]<br>SW846-8000[a]<br>IRP Handbook[b] | Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample | Once per analyst | Analyte-specific limits as per SW846, Table 6 | 1) Recalculate results<br>2) Locate and fix problem with system<br>3) Rerun demonstration for those analytes which did not meet criteria |
| | | Initial daily calibration factor verification | Daily, before sample analysis | Response for any analyte within ±15% of average RF or CF | 1) Perform system maintenance as per SW846-8000 |

(Continued)

TABLE 10-4. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[e] |
|---|---|---|---|---|---|
| SW8020 (Cont'd) | | | | | 2) Prepare new calibration curve for any analyte not meeting criteria<br>3) Reinject any samples analyzed after criteria were exceeded |
| | | Retention time (RT) windows calculated for each analyte | One 72-hour study performed on each GC column and whenever a new column is installed | 1.5% for Megabore, 2.0% for packed columns[d] | 1) Perform maintenance<br>2) Repeat test |
| | | Mid-level Laboratory Control Sample (LCS) (see Section 4.0 tables for list of analytes spiked) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence | Response for any analyte within project limits | 1) Repeat LCS<br>2) Repeat daily calibration |
| | | On-going calibration check | 10% sample frequency, minimum of two per set | a) RF within ±15% of daily calibration factor<br>b) Ongoing calibration analytes elute within daily RT windows | 1) Locate and correct source of problem--document actions taken<br>2) Repeat test only for analytes that failed to meet criteria<br>3) After repeated failures, perform system maintenance as per SW846-8000<br>4) Repeat test for all compounds of interest |

(Continued)

TABLE 10-4. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8020 (Cont'd) | | Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit (if applicable), or 2) concentration of the ongoing calibration check | 1 MS and 1 MSD per every 20 Air Force project samples | a) Laboratory established criteria as per SW846-8000 (see tables in Section 4.0 of QAPP), or<br>b) QC Acceptance Criteria table, SW846-8020 | 1) Flag data<br>2) Analyze a QC check standard containing each analyte that failed criteria<br>3) If recovery for QC check standard is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required<br>4) Reanalyze any samples affected by out-of-control condition |
| | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[a] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | N/A | N/A |

(Continued)

TABLE 10-4. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8020 (Cont'd) | | Surrogate standard spike | Every sample, spike, standard, and reagent blank | Limits determined as per SW846-8000 (limits are to be updated annually on a matrix-by-matrix basis) | 1) Recalculate result, if still out:<br>2) Reanalyze sample, if still out:<br>3) Flag result and document in report that steps 1 and 2 were performed |
| | | Reagent blank | One per day/ instrument | No analytes detected at >3 times the detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br>3) All samples processed with a contaminated blank are to be repurged and reanalyzed at no cost to the Air Force<br>4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |

(Continued)

TABLE 10-4. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8020 (Cont'd) | | Second-column confirmation, to meet IRP requirements[b] | 100% for all positive results except noted laboratory contaminants[e] (also see footnote "f") | Analysis of standards and samples performed on a second GC column of dissimilar phase and retention characteristics within specified holding times | Resampling and reanalysis performed at no cost to government, even if first column analysis was conducted within holding time[b] |
| | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as is specified in the IRP Handbook.
[d] CLP SOW 3/90.
[e] Methylene chloride, acetone, toluene, and 2-butanone are considered to be common laboratory contaminants. Therefore, it is not required that second-column confirmation be performed for the sole purpose of confirming the presence of any one of these four analytes.
[f] For quarterly sampling, second column confirmation not required for sample if all detected compounds were confirmed at least once in previous two quarters.

## TABLE 10-5. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8080

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8080 | Organo-chlorine, Pesticides, and PCBs | Five-point calibration (for all analytes) | Initial calibration prior to sample analysis | a) RSD < %20 for RFs or r ≥0.995 for linear regression<br>b) For quantitation, use average RF if RSD <20%, otherwise use linear regression calibration factor (CF) | Repeat after corrective action |
| | | Second source calibration verification (for all analytes) | Once per five-point calibration | All analytes within ±15% of average RF or CF | 1) Reanalyze second source standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | Refer to:<br>SW846-3500[a]<br>SW846-3600[a]<br>SW846-8000[a]<br>IRP Handbook[b] | Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample. | Once per analyst | Method-specific limits as per SW846, Table 6 | 1) Recalculate results<br>2) Locate and fix problem with system<br>3) Rerun demonstration for those analytes which did not meet criteria |

(Continued)

TABLE 10-5. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8080 (Cont'd) | | Initial daily calibration factor verification using one or more calibration standards (calibration standards must be replaced at least every six months) | Daily, before sample analysis | Response for any analyte within ±15% of average RF or CF | 1) Perform system maintenance as per SW846-8000<br>2) Prepare new calibration curve for any analyte not meeting criteria<br>3) Reinject any samples analyzed after criteria were exceeded<br>4) Replace calibration standards |
| | | Retention time (RT) windows calculated for each analyte | One 72-hour study performed on each GC column and whenever a new column is installed | 1.5% for Megabore, 2.0% for packed columns[d] | 1) Perform maintenance<br>2) Repeat test |
| | | Extracted mid-level Laboratory Control Sample (LCS) (see Section 4.0 tables for list of analytes spiked) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence | Response for any analyte within project acceptance limits | 1) Repeat LCS<br>2) Repeat daily calibration |

(Continued)

TABLE 10-5. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8080 (Cont'd) | | On-going calibration | 10% sample frequency, minimum of two per set | a) RF within ±15% of daily calibration factor<br>b) Ongoing calibration analytes elute within daily RT windows | 1) Locate and correct source of problem--documentation actions taken<br>2) Repeat test only for analytes that failed to meet criteria<br>3) After repeated failures, perform system maintenance as per SW846-8000<br>4) Replace ongoing standard stock solutions<br>5) Repeat test for all compounds of interest |
| | | Breakdown check (Endrin and DDT) | Daily prior to analysis of samples and as needed during analysis | Degradation ≤20% | 1) Clean injection port<br>2) Remove front 2 feet of column<br>3) See EPA SW846-8000[b] |

(Continued)

TABLE 10-5. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8080 (Cont'd) | | Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit (if applicable), or 2) or the concentration of the ongoing calibration check | 1 MS and 1 MSD per every 20 Air Force project samples | a) Laboratory-established criteria as per SW846-8000 (see tables in Section 4.0 of QAPP), or<br>b) QC Acceptance Criteria Table SW846-8080 | 1) Flag data<br>2) Analyze a QC check standard containing each analyte that failed criteria<br>3) If recovery for QC check standard is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required<br>4) Reanalyze any samples affected by the out-of-control condition |
| | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[b] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | N/A | N/A |

(Continued)

TABLE 10-5. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8080 (Cont'd) | | Two surrogate standards spiked into each sample. Decachlorobiphenyl is primary surrogate; TCMX is secondary surrogate | Every sample spike, standard, and reagent blank | Limits determined as per SW846-8000 (limits are to be updated annually on a matrix-by-matrix basis). If low primary surrogate recovery or interferences indicated, calculate secondary surrogate recovery. Proceed with corrective action when both surrogates are out of limits | 1) Recalculate results, if still out:<br>2) Reanalyze sample, if still out:<br>3) Flag result and document in report that steps 1 and 2 were performed |
| | | Reagent blank | One per extraction batch per instrument | No analytes detected at >3 times the detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br>3) All samples processed with a contaminated blank are to be reextracted and reanalyzed at no cost to the Air Force<br>4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |

(Continued)

TABLE 10-5. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8080 (Cont'd) | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits established shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.
[d] CLP SOW 3/90.

## TABLE 10-6. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8140

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8140 | Organophosphorus Pesticides | Five-point calibration (for all analytes) | Initial calibration prior to sample analysis | a) RSD < %20 for RFs or r ≥0.995 for linear regression<br>b) For quantitation, use average RF if RSD <20%, otherwise use linear regression calibration factor (CF) | Repeat after corrective action |
| | | Second source calibration verification | Once per five-point calibration | All analytes within ±15% of average RF or CF | 1) Reanalyze second source standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | Refer to:<br>SW846-3500[a]<br>SW846-3600[a]<br>SW846-8000[a]<br>IRP Handbook[b] | Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample | Once per analyst | Method-specific limits as per SW846, Table 6 | 1) Recalculate results<br>2) Locate and fix problem with system<br>3) Rerun demonstration for those analytes which did not meet criteria |

(Continued)

TABLE 10-6. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8140 (Cont'd) | | Initial daily calibration factor verification using one or more calibration standards (Calibration standards must be replaced at least every six months) | Daily, before sample analysis | Response for any analyte within ±15% of average *RF* or *CF* | 1) Perform system maintenance as per SW846-8000<br>2) Prepare new calibration curve for any analyte not meeting criteria<br>3) Reinject any samples analyzed after criteria were exceeded<br>4) Replace calibration standards |
| | | Retention time (RT) windows calculated for each analyte | One 72-hour study performed on each GC column and whenever a new column is installed | 1.5% for Megabore, 2.0% for packed columns[d] | 1) Perform maintenance<br>2) Repeat test |
| | | Extracted mid-level Laboratory Control Sample (LCS) (see Section 4.0 tables for list of analytes spiked) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence | Response for any analyte within project limits | 1) Repeat LCS<br>2) Repeat daily calibration |

(Continued)

TABLE 10-6. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[f] |
|---|---|---|---|---|---|
| SW8140 (Cont'd) | | On-going calibration check at a representative concentration | 10% sample frequency, minimum of two per set | a) RF within ±15% of daily calibration factor<br>b) Ongoing calibration analytes elute within daily RT windows | 1) Locate and correct source of problem--documentation actions taken<br>2) Repeat test only for analytes that failed to meet criteria<br>3) After repeated failures, perform system maintenance as per SW846-800<br>4) Replace ongoing standard stock solution<br>5) Repeat test for all compounds of interest |
| | | Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit (if applicable), or 2) the concentration of the ongoing calibration check | 1 MS and 1 MSD per every 20 Air Force project samples | a) Laboratory established criteria as per SW846-8000 (see tables in Section 4.0 of QAPP), or<br>b) QC Acceptance Criteria Table SW846-8140 | 1) Flag data<br>2) Analyze a QC check standard containing each analyte that failed criteria<br>3) If recovery for QC check standard is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required<br>4) Reanalyze any sample affected by the out-of-control condition |

(Continued)

TABLE 10-6. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8140 (Cont'd) | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[a] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | N/A | N/A |
| | | Surrogate standard spike | Every sample, spike, standard, and reagent blank | Limits determined as per SW846-8000 (limits are to be updated annually on a matrix-by-matrix basis) | 1) Recalculate result, if still out:<br>2) Reanalyze sample, if still out:<br>3) Flag result and document in report that steps 1 and 2 were performed |
| | | Reagent blank | One per extraction batch per instrument | No analytes detected at >3 times the detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br>3) All samples processed with a contaminated blank are to be re-extracted and reanalyzed at no cost to the Air Force |

(Continued)

TABLE 10-6. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8140 (Cont'd) | | | | | 4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |
| | | Second-column confirmation, to meet IRP requirements[b] | 100% for all positive results above MDL | Confirmation by GC/MS using Method 8270 if detection limits are met or with a second GC column of dissimilar phase and retention characteristics within specified holding times is required | Resampling and reanalysis performed at no cost to government, even if first column analysis was conducted within holding time[b] |
| | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory[b].
[d] CLP SOW 3/90.

TABLE 10-7. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8150

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8150 | Chlorinated Herbicides | Five-point calibration (for all analytes) | Initial calibration prior to sample analysis | a) RSD <20% for RFs or r ≥0.995 for linear regression<br>b) For quantitation, use average RF if RSD <20%, otherwise use linear regression calibration factor (CF) | Repeat after corrective action |
| | | Second source calibration verification | Once per five-point calibration | All analytes within ±15% of average RF or CF | 1) Reanalyze second source standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | Refer to:<br>SW846-8000[a]<br>IRP Handbook[b] | Demonstrate the ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample | Once per analyst | Method-specific limits as per SW846, Table 6 | 1) Recalculate results<br>2) Locate and fix problem with system<br>3) Rerun demonstration for those analytes which did not meet criteria |

(Continued)

TABLE 10-7. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8150 (Cont'd) | | | | | 4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |
| | | Second-column confirmation, to meet IRP[b] requirements | 100% for all positive results above MDL | Confirmation by GC/MS using Method 8270 if detection limits are met or with a second GC column of dissimilar phase and retention characteristics within specified holding times is required | Resampling and reanalysis performed at no cost to government, even if first column analysis was conducted within holding time is required. |
| | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.
[d] CLP SOW 3/90.

TABLE 10-7. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8150 (Cont'd) | | Initial daily calibration factor verification using one or more calibration standards (Calibration standards must be replaced at least every six months) | Daily, before sample analysis | Response for any analyte within ±15% of average RF or CF | 1) Perform system maintenance as per SW846-8000<br>2) Prepare new calibration curve for any analyte not meeting criteria<br>3) Reinject any samples analyzed after criteria were exceeded<br>4) Replace calibration standards |
| | | Retention time (RT) windows calculated for each analyte | One 72-hour study performed on each GC column and whenever a new column is installed | 1.5% for Megabore, 2.0% for packed columns[d] | 1) Perform maintenance<br>2) Repeat test |
| | | Extracted mid-level Laboratory Control Sample (LCS) (see Section 4.0 tables for list of analytes spiked) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence | Response for any analyte within project limits | 1) Repeat LCS<br>2) Repeat daily calibration |
| | | On-going calibration check at a representative concentration | 10% sample frequency, minimum of two per set | a) RF within ±15% of daily calibration factor<br>b) Ongoing calibration analytes elute within daily RT windows | 1) Locate and correct source of problem--document actions taken<br>2) Repeat test only for analytes that failed to meet criteria |

(Continued)

TABLE 10-7. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8150 (Cont'd) | | | | | 3) After repeated failures, perform system maintenance as per SW846-8000<br>4) Replace ongoing standard stock solutions<br>5) Repeat test for all compounds of interest |
| | | Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit (if applicable), or 2) the concentration of the ongoing calibration check | 1 MS and 1 MSD per every 20 Air Force project samples | a) Laboratory-established criteria as per SW846-8000 (see tables in Section 4.0 of QAPP), or<br>b) QC Acceptance Criteria Table SW846-8150 | 1) Flag data<br>2) Analyze a QC check standard containing each analyte that failed criteria<br>3) If recovery for QC check standard is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required<br>4) Reanalyze any samples affected by out-of-control condition. |

(Continued)

TABLE 10-7. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8150 (Cont'd) | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[a] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | N/A | N/A |
| | | Surrogate standard spike | Every sample, spike, standard, and reagent blank. | Limits determined as per SW846-8000 (limits are to be updated annually on a matrix-by-matrix basis) | 1) Recalculate result, if still out:<br>2) Reanalyze sample, if still out:<br>3) Flag result and document in report that steps 1 and 2 have been performed |
| | | Reagent blank | One per extraction batch per instrument | No analytes detected at >3 times the detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br>3) All samples processed with a contaminated blank are to be re-extracted and reanalyzed at no cost to the Air Force |

(Continued)

TABLE 10-7. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8150 (Cont'd) | | | | | 4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |
| | | Second-column confirmation, to meet IRP[b] requirements | 100% for all positive results above MDL | Confirmation by GC/MS using Method 8270 if detection limits are met or with a second GC column of dissimilar phase and retention characteristics within specified holding times is required | Resampling and reanalysis performed at no cost to government, even if first column analysis was conducted within holding time is required. |
| | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.
[d] CLP SOW 3/90.

## TABLE 10-8. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8240

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8240 | Volatile Organics<br><br>Refer to:<br>SW846-8240[a]<br>IRP Handbook[b] | Check of mass spectral ion intensities using BFB | Initially, prior to calibration, again prior to sample analyses and once per every 12-hour shift | Established criteria in Table 3 of SW846-8240[a] | 1) Retune instrument<br>2) Repeat BFB analysis |
| | | Five-point calibration (for all analytes) | Initial calibration prior to sample analysis | 1) SPCCs[d] average RF $\geq 0.30$[a]<br>2) RSD $< 30\%$ for CCC[f] RFs | 1) Repeat after corrective action if either criterion is not met |
| | | Independently prepared calibration verification | Once per five-point calibration | Analytes within 25% of average RF | 1) Reanalyze verification standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | | Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample | Once per analyst | Analyte-specific limits as per SW846, Table 6 | 1) Recalculate results<br>2) Locate and fix the source of the problem<br>3) Rerun demonstration for those analytes which did not meet criteria |

(Continued)

TABLE 10-8. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8240 (Cont'd) | | Initial calibration factor verification | Once per each 12 hours, prior to sample analysis (criteria for these checks must be met prior to sample analysis) (prepare calibration standards weekly) | 1) SPCCs[d] average RF ≥ 0.30[e]<br>2) CCC[f] percent difference <25% from average response factors calculated following initial calibration | 1) Evaluate system and take corrective action<br>2) If source of problem cannot be determined, a new five-point calibration must be generated |
| | | Internal Standards' (IS) Retention Time (RT) and responses check from calibration check standard | Immediately after or during data acquisition of calibration check standard | RT ±30 seconds and EICP[g] within -50% to +100% of last calibration verification (12 hrs) for each IS compound | 1) Inspect MS or GC for malfunctions<br>2) Take appropriate corrective actions<br>3) Mandatory reanalysis of samples analyzed while system was malfunctioning |
| | | Mid-level Laboratory Control Sample (LCS) (see Section 4.0 QAPP tables for list of analytes spiked). If medium-level soil samples analyzed, LCS should be extracted as well. | Daily (as per instrument calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence | Response for any analyte within project limits | 1) Repeat LCS<br>2) Repeat daily calibration |

(Continued)

TABLE 10-8. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8240 (Cont'd) | | On-going calibration check | 10% sample frequency (replace stock standards according to frequency specified in SW846-8240[a]) | QC Acceptance Criteria Table (SW846-8240) | 1) Repeat test only for analytes that failed to meet criteria<br>2) After repeated failures, locate and correct source of problem -- document actions taken<br>3) Repeat test for all compounds of interest |
| | | Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit (if applicable, otherwise 20 µg/L), or 2) the concentration of the ongoing calibration check | 1 MS and 1 MSD per every 20 Air Force project samples | a) Laboratory-established criteria as per SW846-8000 (see tables in Section 4.0 of QAPP), or<br>b) QC Acceptance Criteria Table SW846-8240 | 1) Flag data<br>2) Analyze a QC check standard containing each analyte that failed criteria<br>3) If recovery for QC check standard is outside of designated range (Table 6 of SW846), the system is considered to be out of control. Immediate identification and correction of problem is required<br>4) Reanalyze any samples affected by out-of-control condition |

(Continued)

TABLE 10-8. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8240 (Cont'd) | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[a] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | N/A | N/A |
| | | Surrogate standards spike | Every sample, spike, standard, and reagent blank | Limits determined as per SW846-8000 (limits are to be updated annually on a matrix-by-matrix basis) | 1) Recalculate result, if still out:<br>2) Check instrument performance, take corrective action, if necessary<br>3) Reanalyze sample, if still out:<br>4) Flag result if it does not meet criteria and document in report that steps 1 through 3 were performed |
| | | Reagent blank | Daily prior to sample analysis for each matrix and once per extraction batch for medium-level soil sample analysis up to 20 samples/batch | No analytes detected >3 times the detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br><br>(Corrective Action continued on next page) |

(Continued)

TABLE 10-8. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8240 (Cont'd) | | | | | 3) Repeat initial daily blank analysis and re-extract all medium-level soil samples *prior to* analysis<br><br>4) Sample results associated with reagent blank contamination at $\leq$3 times the detection limit (5 times for common laboratory contaminants) are to be flagged |
| | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as is specified in the IRP Handbook.
[d] SPCC = System Performance Check Compounds.
[e] SPCC for bromoform is $\geq$ 0.25.
[f] CCC = Calibration Check Compounds.
[g] EICP = Extracted Ion Current Profile.

## TABLE 10-9. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8270

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[e] |
|---|---|---|---|---|---|
| SW8270 | Semivolatile Organics<br><br>Refer to:<br>SW846-3500[a]<br>SW846-3600[a]<br>SW846-8270[a]<br>IRP Handbook[b] | Check of mass spectral ion intensities using DFTPP, (4,4'-DDT, pentachlorophenol and benzidine also to be included in tuning standard to verify injection port inertness and GC column performance) | Initially, prior to calibration, again prior to sample analyses and once per every 12-hour shift<br>- Degradation of DDT ≤20%<br>- Benzidine and pentachlorophenol should be present at their normal responses<br>- Peak tailing factor ≤3 | Established criteria in Table 3 of SW846-8270[a] | 1) Retune instrument<br>2) Clean injection port, if necessary<br>3) Remove first 6 to 12 inches of column, if necessary<br>4) Repeat analysis tuning standard |
| | | Five-point calibration for all commercially available analytes | Initial calibration prior to sample analysis (prepare fresh stock standard solution once a year) | 1) %RSD <30% for each individual CCC[c]<br>2) SPCCs[d] average RF ≥0.050 | Repeat after corrective action if any one of the acceptance criteria are not met |
| | | Independently prepared calibration verification | Once per five-point calibration | Analytes within 30% of average RF | 1) Reanalyze verification standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |

(Continued)

TABLE 10-9. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8270 (Cont'd) | | Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample | Once per analyst | Analyte-specific limits as per SW846, Table 6 | 1) Recalculate results<br>2) Locate and fix the source of the problem<br>3) Rerun demonstration for those analytes which did not meet criteria |
| | | Initial calibration factor verification | Once per each 12-hour period, prior to sample analysis (Criteria for these checks must be met prior to sample analysis). Prepare calibration standards weekly | 1) SPCCs[d] average RF $\geq 0.050$<br>2) CCC[e] percent difference $< 30\%$ | 1) Evaluate system and take corrective action<br>2) If source of problem cannot be determined, a new five-point calibration must be generated |
| | | Internal Standards' (IS) Retention Time (RT) and Responses check from calibration check standard | Immediately after or during data acquisition of calibration check standard | RT $\pm 30$ seconds and EICP[f] within -50% to +100% of last calibration verification (12 hours) for each IS compound | 1) Inspect MS or GC for malfunctions<br>2) Take appropriate corrective actions |
| | | Extracted mid-level Laboratory Control Sample (LCS) (see Section 4.0 tables for list of analytes spiked) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration check | Response for any analyte within laboratory established limits | 1) Repeat LCS<br>2) Repeat calibration check |

(Continued)

TABLE 10-9. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8270 (Cont'd) | | On-going calibration check | 10% sample frequency (matrix spike may substitute for one check every 20 samples), minimum of two per set | Laboratory established limits | 1) Locate and correct source of problem--document actions taken<br>2) Repeat test only for analytes that failed to meet criteria<br>3) After repeated failures, perform system maintenance as per SW846-8000<br>4) Repeat test for all compounds of interest |
| | | Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit, if applicable, otherwise near the top of the calibration range, or 2) concentration of ongoing calibration check | 1 MS and 1 MSD per every 20 Air Force project samples | a) Laboratory-established criteria as per SW846-8000 (see tables in Section 4.0 of QAPP), or<br>b) QC Acceptance Criteria Table SW846-8270 | 1) Flag data<br>2) Analyze a QC check standard containing each analyte that failed criteria<br>3) If recovery for QC check standard is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required<br>4) Reanalyze any samples affected by out-of-control condition. |

(Continued)

TABLE 10-9. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8270 (Cont'd) | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[a] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | N/A | N/A |
| | | Surrogate standards spike | Every sample, spike, standard, and reagent blank | Laboratory established limits determined as per SW846-8000 (limits are to be updated annually on a matrix-by-matrix basis). Corrective action required only if more than 1 acid and 1 base surrogate exceeds criteria | 1) Recalculate result, if still out:<br>2) Check instrument performance and take corrective action, if necessary<br>3) Reanalyze sample, if still out:<br>4) Flag result and document in report that steps 1 and 3 were performed |
| | | Reagent blank | Once per extraction batch (up to 20 sample/batch) and each time there is a change in reagents | No analytes detected at >3 times the detection limits except phthalates that are <5 times MDL | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br><br>(Correction Action continued on next page) |

(Continued)

TABLE 10-9. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8270 (Cont'd) | | | | | 3) All samples processed with a contaminated blank are to be re-extracted and reanalyzed at no cost to the Air Force<br>4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |
| | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.
[d] SPCC = System Performance Check Compounds
[e] Calibration Check Compounds
[f] EICP = Extracted Ion Current Profile

## TABLE 10-10. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8280

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8280 | Refer to:<br>SW8280[a]<br>IRP Handbook[b] | Initial calibration multipoint, five levels in triplicate | Prior to sample analysis, annually, and as required by routine calibration checks (replace calibration standard solutions after six months) | % RSD ≤30% for RRFs of each calibration standard (analyzed in triplicate) | 1) Repeat initial calibration<br>2) If still unacceptable, make necessary adjustment<br>3) Repeat initial calibration until criteria are met |
| | | Independently prepared calibration verification | Once per five-point calibration | Analytes within 30% of average RF | 1) Reanalyze verification standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | | Column performance check standard | Prior to sample analysis at the beginning of every 12-hour period | 1) Established criteria in Table 3 of SW846-8280<br>2) ≤25% valley between 1,2,3,4-TCDD and 2,3,7,8-TCDD | Repeat until criteria are met |
| | | Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC reference sample | Once per analyst | Analyte-specific limits as per SW846 | 1) Recalculate results<br>2) Locate and fix the source of the problem with system<br>3) Rerun demonstration for those analytes which did not meet criteria |

(Continued)

TABLE 10-10. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8280 (Cont'd) | | Routine calibration, using single-point calibration standard solution as specified in method | Daily, at beginning and end of each 12-hour period | 1) RRF agreement within 30% of value predicted from multipoint calibration curve<br>2) ±15% or less isotope ratio agreement with theoretical values.<br>3) Established criteria in Table 3 of SW846-8280 | 1) Repeat single-point check<br>2) If still unacceptable, perform new multipoint calibration |
| | | Sensitivity check | Daily, prior to sample analysis | 50:1 S/N from 200 ng/mL 2,3,7,8-TCDD standard | Evaluate instrument; take corrective action |
| | | Extracted mid-level Laboratory Control Sample (LCS) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence | Response for any analyte within ±50% of predicted response until laboratory acceptance limits can be established | 1) Repeat LCS<br>2) Repeat routine calibration |
| | | Extracted duplicate analysis | 5% sample frequency | ≤30% | 1) Obtain a third value<br>2) Make report comment |

(Continued)

TABLE 10-10 (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8280 (Cont'd) | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[a] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | N/A | N/A |
| | | Reagent (extracted) blank | Once per extraction batch (up to 20 samples/batch) and each time there is a change in reagents | No analytes[e] detected at >3 times the detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br>3) All samples processed with a contaminated blank are to be re-extracted and reanalyzed at no cost to the Air Force<br>4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |

(Continued)

TABLE 10-10. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8280 (Cont'd) | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits established that exceed criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

TABLE 10-11. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW8310

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8310 | Polynuclear Aromatic Hydrocarbons | Five-point calibration (for all analytes) | Initial calibration prior to sample analysis | a) RSD <20% for RFs or r ≥ 0.995 for linear regression<br>b) For quantitation, use average RF if RSD <20%, otherwise use calibration factor (CF) from linear regression. | Repeat after corrective action |
| | | *Second source* calibration verification (for all analytes) | Once per five-point calibration | All analytes within ±15% of average RF or CF | 1) Reanalyze second source standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | Refer to:<br>SW846-3500[a]<br>SW846-3600[a]<br>SW846-8000[a]<br>IRP Handbook[b] | Demonstrate ability to generate acceptable accuracy and precision using 4 replicate analyses of a QC check sample | Once per analyst | Method-specific limits as per SW846, Table 6 | 1) Recalculate results<br>2) Locate and fix problem with system<br>3) Rerun demonstration for those analytes which did not meet criteria |

(Continued)

TABLE 10-11. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8310 (Cont'd) | | Initial calibration factor verification using one or more calibration standards (calibration standards must be replaced at least every six months) | Daily, before sample analysis | Response for any analyte within ±15% of predicted response | 1) Perform system maintenance as per SW846-8000<br>2) Prepare new calibration curve for any analyte not meeting criteria<br>3) Reinject any samples analyzed after criteria were exceeded<br>4) Replace calibration standards |
| | | Retention time (RT) windows calculated for each standard | One 72-hour study performed on each GC column and whenever a new column is installed | RT windows ≤3% | 1) Perform maintenance<br>2) Repeat test |
| | | Mid-level Laboratory Control Sample (LCS) (see Section 4.0 tables for list of analytes spiked) | Daily (as per instrument calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence | Response for any analyte within project limits | 1) Repeat LCS<br>2) Repeat daily calibration |

(Continued)

TABLE 10-11. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8310 (Cont'd) | | On-going mid-level calibration check at concentrations specified in SW846-8310, Section 8.2.1 | 10% sample frequency, minimum of two per set | a) RF within ±15% of daily calibration factor<br>b) Ongoing calibration analytes elute within daily RT windows | 1) Locate and correct source of problem--document actions taken<br>2) Repeat test only for analytes that failed to meet criteria<br>3) After repeated failures, perform system maintenance as per SW846-8000<br>4) Repeat test for all compounds of interest<br>5) Replace ongoing standard stock solutions |
| | | Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration (if applicable), or 2) concentration of the ongoing calibration check | 1 MS and 1 MSD per every 20 Air Force project samples | a) Laboratory-established criteria as per SW846-8000 (see tables in Section 4.0 of QAPP), or<br>b) QC Acceptance Criteria Table, SW846-8310 | 1) Flag data<br>2) Analyze a QC check standard containing each analyte that failed criteria<br>3) If recovery for QC check standard is outside of designated range, the system is considered to be out of control. Immediate<br><br>(Corrective Action continued on next page) |

(Continued)

TABLE 10-11. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW8310 (Cont'd) | | | | | identification and correction of problem is required<br>4) Reanalyze any samples affected by out-of-control condition |
| | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per SW846-8000[a] and the IRP Handbook[b] | After analysis of initial five spiked samples; updated on regular basis | NA | NA |
| | | Surrogate standard spike | Every sample spike, standard, and reagent blank | Limits determined as per SW846-8000 (limits are to be updated annually on a matrix-by-matrix basis) | 1) Recalculate result, if still out:<br>2) Reanalyze sample, if still out:<br>3) Flag result and document in report that steps 1 and 2 were performed |

(Continued)

TABLE 10-11. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW8310 (Cont'd) | | Reagent blank | One per instrument per extraction batch | No analytes detected at >3 times the detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br>3) All samples processed with a contaminated blank are to be reanalyzed at no cost to the Air Force<br>4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |
| | | MDL study | Once per year | See MDL table | Detection limits established shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory as is specified in the IRP Handbook.

## TABLE 10-12. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW6010

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW6010 | Trace Metals-ICPES | Mixed calibration standard as per SW846-6010 | Daily, prior to sample analysis | Within ± 5% of expected values | 1) Correct problem according to instrument manufacturer's recommendations<br>2) Reanalyze standard |
| | Refer to:<br>SW846-3005[a]<br>SW846-3050[a]<br>SW846-6010[a]<br>IRP Handbook[b] | Check of calibration standard stability using independent quality control sample | Weekly | Within ±10% of expected value | Prepare fresh primary calibration standard stock |
| | | Single-point instrument check standard (concentration = mid-point of calibration range) | Every 10 samples and at end of analytical run | Within ±5% of expected value | 1) Terminate analysis<br>2) Locate and correct problem<br>3) Recalibrate<br>4) Reanalyze associated samples |
| | | Calibration blank | Every 10 samples and at end of analytical run | Within ±3s of mean blank value | 1) Repeat twice - average results<br>2) If average not within ±3s of background mean, terminate analysis<br><br>(Corrective Action continued on next page) |

(Continued)

TABLE 10-12. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW6010 (Cont'd) | | | | | 3) Locate and correct problem<br>4) Recalibrate<br>5) Reanalyze previous 10 samples |
| | | Mid-level, digested Laboratory Control Sample (LCS) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]) | Project limits; see Tables 4-4 and 4-5 | 1) Reanalyze LCS, if still out<br>2) Correct problem<br>3) Reanalyze associated samples |
| | | Interference check sample | Run at the more frequent of the following:<br>1) Beginning and end of analytical run; or<br>2) Twice during every 8-hour work shift | Within ±20% of expected value for instrument check standard elements | 1) Recalibrate<br>2) See Lab Manager |
| | | Matrix spike (MS)/matrix spike duplicate (MSD) (portion of sample is spiked prior to digestion) | 1 MS and 1 MSD per every 20 air Force project samples | Project precision and accuracy criteria | Flag data |

(Continued)

TABLE 10-12. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW6010 (Cont'd) | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per the IRP Handbook[b] | NA | NA | NA |
| | | Reagent blank | Once per day/ extraction batch | No analytes detected at >3 times detection limits | 1) Investigate source of contamination<br>2) Appropriate corrective action taken and documented.<br>3) All samples processed with a contaminated blank are to be re-digested and reanalyzed at no cost to the Air Force.<br>4) Sample results associated with reagent blank contamination at ≤3 times the detection limits are to be flagged. |

(Continued)

TABLE 10-12. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW6010 (Cont'd) | | MDL Study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits which exceed established criteria shall be submitted to the Air Force for approval prior to analysis of any project samples[b] |
| | | Linear Range Study | Once per year | NA | NA |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

**TABLE 10-13. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR ATOMIC ABSORPTION METHODS**

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW7060<br>SW7421<br>SW7470/<br>7471<br>SW7740 | Metals - AA<br>As<br>Pb<br>Hg<br>Se | Multipoint calibration (minimum of three standards and a reagent blank) | Daily (prepare fresh standards each time a batch of samples is analyzed) | $r \geq 0.995$ | 1) Repeat test<br>2) Take corrective action<br>3) Repeat calibration and test |
| | | Independently prepared calibration verification | Once per multipoint calibration | Within ± 10% of expected value | 1) Reanalyze verification standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | Refer to:<br>SW846-7000[a]<br>IRP Handbook[b] | Calibration blank | After every 10 samples | ≤5 x method detection limit | 1) Rerun<br>2) Clean system<br>3) Rerun samples back to last clean blank |
| | | Calibration check (one standard near mid-range) | After every 10 samples | Within ±10% of expected value | 1) Replace graphite tube if necessary (flameless procedure)<br>2) Recalibrate<br>3) Rerun samples back to last standard that met criteria |

(Continued)

TABLE 10-13. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| AA Methods (Cont'd) | | Digested Laboratory Control Sample (LCS) (for all analytes) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]) | Project limits; see Tables 4-4 and 4-5 | 1) Reanalyze LCS, if still out<br>2) Correct problem<br>3) Reanalyze associated samples |
| | | Presence of interference to be treated as specified in SW846-7000[a] | NA | NA | NA |
| | | Matrix spike (MS)/matrix spike duplicate (MSD) (portion of sample is spiked prior to digestion) | 1 MS and 1 MSD per every 20 Air Force project samples | Project accuracy and precision criteria | Flag data |
| | | Record assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per the IRP Handbook[b] | NA | NA | NA |

(Continued)

TABLE 10-13. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| AA Methods (Cont'd) | | Reagent blank | Once per day/ extraction batch | No analytes detected at >3 times detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken and documented<br>3) All samples processed with a contaminated blank are to be redigested and reanalyzed at no cost to the Air Force<br>4) Sample results associated with reagent blank contamination at ≤3 times the detection limits are to be flagged |
| | | MDL Study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as is specified in the IRP Handbook.

## TABLE 10-14. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW7196

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW7196 | Hexavalent Chromium | Multipoint calibration curve (one blank and five standards) | Daily, prior to sample analysis (prepare fresh standards daily) | $r \geq 0.995$ | Repeat calibration |
| | Refer to: SW846-7196[a] IRP Handbook[b] | Verification check to ensure lack of reducing condition and/or interference | For every sample matrix analyzed | Spike recovery between 85 - 115% | 1) If verification indicates suppressive interference, dilute and reanalyze sample<br>2) Persistent interference indicates need to use alternative method |
| | | Independently prepared calibration verification | Once per multipoint calibration | Within ±15% of expected value | 1) Reanalyze verification standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | | Mid-level, digested Laboratory Control Sample (LCS) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]) or once every 15 samples, whichever is more frequent | Project limits; see Tables 4-4 and 4-5 | 1) Reanalyze LCS, if still out<br>2) Correct problem<br>3) Reanalyze all associated samples |
| | | Matrix spike (MS)/matrix spike duplicate (MSD) | 1 MS and 1 MSD per every 20 Air Force project samples | Project accuracy and precision criteria | Flag data.<br>Precision: Flag data |

(Continued)

TABLE 10-14. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW7196 (Cont'd) | | Laboratory spike duplicate | Once every 10 samples (MS/MSD may substitute for one lab duplicate every 20 samples), or one per batch, whichever is more frequent | Laboratory established precision criteria | 1) Reanalyze<br>2) Flag data |
| | | Reagent blank | Once per day/ extraction batch | No analytes detected at >3 times detection limits | 1) Clean instrument/ equipment<br>2) All samples processed with a contaminated blank are to be re-digested and reanalyzed at no cost to the Air Force<br>3) Sample results associated with reagent blank contamination at ≤3 times the detection limits are to be flagged. |
| | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per the IRP Handbook[b] | NA | NA | NA |

(Continued)

TABLE 10-14. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW7196 (Cont'd) | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP[b] Handbook | Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as is specified in the IRP Handbook.

## TABLE 10-15. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR SW9010/SW9012

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW9010/ SW9012 | Total and Amenable Cyanide | Multipoint calibration curve (one blank and six standards) | Daily, prior to sample analysis (prepare fresh standards daily) | $r \geq 0.995$ | Repeat calibration |
| | | Independently prepared calibration verification | Once per multipoint calibration | Within ±15% of expected value | 1) Reanalyze verification standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | Refer to: SW846-9012[a] IRP Handbook[b] | Distillation check (QC check added to a sample PRIOR to distillation) | Once per analytical batch | Recovery within ±15% | Flag data |
| | | Mid-level Laboratory Control Sample (LCS) (for all analytes) | Daily (as per Instrument Calibration requirements in IRP Handbook[b]) or once every 15 samples, whichever is more frequent | Recovery within ±15% | Prepare fresh primary calibration standard stock |
| | | Matrix spike (MS) matrix spike duplicate (MSD) | 1 MS and 1 MSD per every 20 Air Force project samples | Project accuracy and precision criteria | Flag data |

(Continued)

TABLE 10-15. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| SW9012 (Cont'd) | | Laboratory spike duplicate | Once every 10 samples (MS/MSD may substitute for one lab duplicate every 20 samples), or one per batch, whichever is more frequent | RPD <20% for water, and <30% for soil | Obtain third value |
| | | Reagent blank | Once per day/ extraction batch | No analytes detected at >3 times detection limits | 1) Clean instrument/ equipment<br>2) All samples processed with a contaminated blank are to be redigested and reanalyzed at no cost to the Air Force<br>3) Sample results associated with reagent blank contamination at ≤3 times the detection limits are to be flagged |
| | | Record of assessed accuracy for each matrix studied should be maintained and updated on a regular basis as per the IRP Handbook[b] | NA | NA | NA |

(Continued)

TABLE 10-15. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| SW9012 (Cont'd) | | MDL study | Once per year | Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b] | Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to analysis of any project samples[b] |

[a] Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as is specified in the IRP Handbook.

**TABLE 10-16. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR E504**

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| E504[a] | 1,2-Dibromoethane (EDB)<br>1,2-Dibromo-3-chloropropane (DBCP) | Minimum of 3-point calibration (5-point recommended) | Daily, before sample analysis--at least one multipoint must be performed prior to analysis of Air Force project samples | a) RSD <20% for RF or r ≥0.995 for linear regression<br>b) For quantitation use average RF if RSD <20%, otherwise use linear regression calibration factor | Repeat after corrective action |
| | | Second source calibration verification (for all analytes) | Once per five-point calibration | All analytes within ±15% of average RF or CF | 1) Reanalyze second source standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | | Single point calibration | Daily, before sample analysis (can be used instead of multipoint calibration) | <20% deviation between response of standard and response of sample | Prepare new single-point calibration standard and reanalyze, upon repeated failure, repeat multipoint calibration |
| | | Demonstrate the ability to generate acceptable precision and accuracy | Updated on a regular basis | 80-120% of true value, and standard deviation must meet established criteria | 1) Correct source of problem<br>2) Repeat test |

(Continued)

TABLE 10-16. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| E504 (Cont'd) | | Quality control samples (outside source, PE sample) | Quarterly | Within established acceptance criteria | Take appropriate corrective action |
| | | MDL study | Once per year | See Table 8-16 | Detection limits established shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |
| | | MDL check sample | Weekly | 1) Instrument response must indicate that the laboratory's MDL is distinguishable from instrument background signal<br>2) Recovery between 60-140% of expected value | 1) Reanalyze, if still out<br>2) Take appropriate corrective action |
| | | Laboratory fortified blank | 10% sample frequency | Recovery within 60-140% of expected value | 1) Reanalyze, if still out<br>2) Take appropriate corrective action |
| | | Laboratory fortified sample matrix | 1 MS and 1 MSD per every 20 Air Force project samples | Laboratory established criteria | Flag data |

(Continued)

TABLE 10-16. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| E504 (Cont'd) | | Laboratory reagent blank and field reagent blank (if applicable) | Daily | Demonstrate that interferences from the analytical system are under control | 1) Source of contamination investigated<br>2) Take appropriate corrective action<br>3) All samples processed with a contaminated blank are to be re-extracted and reanalyzed at no cost to the government<br>4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |
| | | Second-column confirmation | 100% for all positive results | Analysis of standards and samples performed on a second GC column of dissimilar phase and retention characteristics or by GC/MS within specified holding time | Resampling and reanalysis performed at no cost to the government, even if first column analysis was conducted within holding time[b] |

[a] Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88/039.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

TABLE 10-17. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR E607

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[a] |
|---|---|---|---|---|---|
| E607[a] | Nitrosamines | Three-point calibration (minimum) for all analytes | Initial calibration prior to sample analysis | a) RSD < 10% for RF or CF<br>b) For quantitation use average RF or CF if RSD < 10% otherwise use calibration curve | Repeat after corrective action |
| | | Second source calibration verification (for all analytes) | Once per three-point calibration | All analytes within ±15% of average RF or CF | 1) Reanalyze second source standard<br>2) Prepare fresh primary calibration stock<br>3) Recalibrate for analytes not meeting criteria |
| | | Initial daily verification of calibration curve | Daily before sample analysis | Response for any analyte ±15% of predicted response | 1) Repeat test with fresh calibration standard, if still out<br>2) Prepare new calibration curve for any analyte not meeting criteria |
| | | Initial demonstration of ability to generate acceptable accuracy and precision | Once per analyst | Precision and accuracy must meet method established criteria | 1) Locate and correct source of problem and repeat the test |

(Continued)

TABLE 10-17. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[e] |
|---|---|---|---|---|---|
| E607 (Cont'd) | | Matrix spike (MS)/ matrix spike duplicate (MSD) | MS and 1 MSD per every 20 Air Force project samples | Must meet method established/laboratory established precision criteria | 1) Analyze quality control check standard, if out<br>2) Identify problem and correct |
| | | Reagent blank | One per extraction batch per instrument or whenever reagents are changed | Demonstrate that interferences from analytical system and glassware are under control | 1) Source of contamination investigated<br>2) Appropriate corrective action taken<br>3) All samples processed with a contaminated blank are to be re-extracted and reanalyzed at no cost to the government<br>4) Sample results associated with reagent blank contamination at $\leq 3$ times the detection limit are to be flagged |

(Continued)

TABLE 10-17. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| E607 (Cont'd) | | Quality Control Check Standard (QCCS) | 10% of sample frequency or less if spike recoveries from samples meet specified quality control criteria | Must meet method established criteria | 1) Identify problem and correct<br>2) Reanalyze sample associated with QCCS |
| | | Second-column confirmation | 100% for all positive results above the MDL | Analysis of standards and samples performed on a second GC column of dissimilar phase or retention characteristics or by GC/MS, within specified holding times | Resampling and reanalysis performed at no cost to the government, even if first column analysis was conducted within holding time[b] |
| | | Accuracy interval | Updated regularly (after 5 to 10 new measurements) | Laboratory derived, from average percent recovery and standard deviation | N/A |
| | | MDL study | Once per year | N/A | Detection limits established shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Federal Register, Volume 49, No. 29, October 26, 1984.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

**TABLE 10-18. SUMMARY OF CALIBRATION AND INTERNAL QUALITY CONTROL PROCEDURES FOR HML 338**

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| HML 338[a] | Organic lead | Multipoint calibration (minimum of three standards and a reagent blank) | Daily, prior to sample analysis | $r \geq 0.995$ | 1) Take appropriate corrective action<br>2) Repeat calibration |
| | | Calibration blank | After every 10 samples | ≤ 5X method detection limit | 1) Rerun<br>2) Clean system<br>3) Rerun samples back to last acceptable blank |
| | | Calibration check (mid-range) | After every 10 samples | Within ±20 of expected value | 1) Recalibrate<br>2) Rerun samples back to last standard that met criteria |
| | | Matrix spike (MS)/ matrix spike duplicate (MSD) | 1 MS and 1 MSD per every 20 Air Force project samples | 1) Spike recoveries within laboratory established limits<br>2) RPD within laboratory established limits | Flag data |

(Continued)

TABLE 10-18. (Continued)

| Analytical Method | Applicable Parameter | Quality Control Check | Minimum Frequency | Acceptance Criteria | Corrective Action[c] |
|---|---|---|---|---|---|
| HML 338 (Cont'd) | | Reagent blank | Once per day/ preparation batch | No analytes detected at >3 times detection limits | 1) Source of contamination investigated<br>2) Appropriate corrective action taken<br>3) All samples processed with a contaminated blank are to be re-extracted and reanalyzed at no cost to the Air Force<br>4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged |
| | | MDL study | Once per year | N/A | Detection limits established shall be submitted to the Air Force for approval prior to the analysis of any project samples[b] |

[a] Tentative method — not approved, Leaking Underground Fuel Tank Field Manual, December 1987.
[b] IRP Handbook, May 1991.
[c] All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory as specified in the IRP Handbook.

# 11.0 PERFORMANCE AND SYSTEMS AUDITS

Systems audits, performance audits, and data quality audits are independent assessments of sample collection and analysis procedures. Audit results are used to evaluate the ability of the system to produce data that fulfill the objectives established for the program, satisfy the quality control (QC) criteria, and identify any areas requiring corrective action. A systems audit is a qualitative review of the overall sampling or measurement system, while performance and data quality audits are quantitative assessments of a measurement system.

The frequency of each type of audit will be established during planning for sampling and analysis tasks. General guidelines for conducting audits are to perform technical systems audits of field procedures for large (more than 50 samples) and/or ongoing sampling programs (i.e., the quarterly groundwater monitoring program). These audits will be conducted during initial stages of field work to identify and correct problems as quickly as possible. Laboratory technical systems audits will be performed prior to establishing analytical services contracts. These audits will be performed by project QA representatives and by Air Force QA representatives.

Custom performance evaluation (PE) audit samples will also be submitted and analyzed for sampling and analysis programs. General guidelines for submitting PE samples are a large (more than 50 samples) and/or ongoing sampling program. The custom PE samples are prepared by Radian's Austin QA Group using certified materials, or are obtained from vendors. The custom PE sample concentrations are certified or confirmed by analysis at one or more laboratories. The analytes and concentrations of interest will be determined by the project director, quality assurance (QA) officer, and QC task leader, based on historical or anticipated conditions.

The custom PE samples will be developed from NBS standard reference materials, NBS traceable materials, U.S. EPA QC materials, or neat compounds of the highest purity available. The PE sample results provide a point-in-time evaluation of data quality related to the program QA objectives. If questions about the PE samples arise, they can be analyzed by independent laboratories (at additional cost) to provide confirmation of the compounds and concentrations in the prepared samples. The samples are prepared in a clean matrix or medium which allows evaluation of the analytical success of the method assuming no matrix interferences. The samples are submitted "single" or "double blind," so

the results provide a realistic assessment of the accuracy of the field samples they were submitted with. In some cases, it may be desirable to prepare a PE sample from an actual sample matrix (e.g., soil). In those cases, the QC staff will coordinate with the Project Director and vendor to ensure that representative materials are collected and provided to the vendor.

The data quality audits to be performed (specific samples or groups of samples to be audited) will be determined during planning stages for each project. The audits will be performed on data for samples collected or analyzed during a specific period of time, or analytical batches or work orders will be selected at random for each task.

Audit programs are currently in place for the quarterly Groundwater Sampling and Analysis Program and the Operable Unit B Phase I Remedial Investigation. The following audits are planned for the quarterly groundwater sampling and analysis program:

- Biannual technical systems audits of groundwater sampling procedures.

- Performance evaluation samples submitted quarterly, consisting of low and high level and blank samples for SW8010, SW8020, and SW6010 analyses. The PE samples for analysis by Method SW8010 will be submitted each quarter, with PE samples for Methods SW8020 and SW6010 submitted on alternating quarters.

- Data quality audits consisting of complete evaluation and verification of field and laboratory data for two work orders each quarter. There will be a minimum of 10 samples on each work order audited.

The following audits are planned for the Operable Unit B Phase I Remedial Investigation:

- Documentation checks will be performed for all activities once per month.

- Technical systems (field) audits will be performed at approximately one per quarter and/or at least once for each of the four field activities including soil, soil gas, and surface water sampling, and soil boring.

- At least one set of PE samples will be submitted to subcontracting laboratories. The specific methods planned for evaluation using PE samples will be documented in the SOW for the project and/or SAP and QC task instructions.

- An on-site laboratory (systems) audit will be performed for the soil gas field procedures and analysis, and for the VOC and PCB field laboratories.

- At least four data quality audits will be performed.

- A minimum of one data quality audit and one field audit will be performed for field analyses, depending on the Scope of Work.

- Technical systems audits of subcontracting laboratories will be conducted during the laboratory evaluation and selection process and during performance of project sample analyses. These audits will be scheduled near the beginning of the field effort to identify and correct any problems early in the project.

In addition to conducting audits for McClellan AFB projects, laboratories undergo audits for various state agencies, and private clients are also conducted. Audit results are generally available for review upon request.

Audit records for contracting laboratories will be reviewed by the contractor's QA representatives to determine whether data produced by the laboratory fulfill the objectives of the program. A systems or data quality audit will be conducted or additional information will be requested if data quality problems are indicated by the review.

Audits are conducted by a person(s) who is familiar with the objectives, principles, and procedures being reviewed, but is independent of the program. A detailed audit checklist is prepared and used for each audit, and contains items that delineate the critical aspects of the procedure under review. All observations are documented, and the checklist is submitted along with the auditor's written assessment and recommendations to the QA representative, project manager, representatives of the audited sampling or analytical task, and other participants as appropriate.

More detailed descriptions of the established audit procedures are provided in the following subsections.

## 11.1 Technical Systems Audit

A technical systems audit is an on-site, qualitative review of the sampling or analytical system. Sampling systems will be audited, preferably at the beginning of the sampling task by the QA Officer or a qualified technical staff member. The QA Officer or staff member conducting the audit is not responsible to project management and can, therefore, act independently. The internal laboratory systems audit results will be used to review laboratory operation and ensure that the technical procedures and documentation are in place and operating to provide data that fulfill the project objectives. These audits are conducted by members of the Research and Engineering QA Group for the Radian laboratory, and by the contractor's QA Officer or designated QA staff members for the other subcontracting laboratories.

Critical items for a laboratory systems audit include:

- Calibration procedures and documentation;
- Completeness of data forms, notebooks, and other reporting requirements;
- Data review and validation procedures;
- Data storage, filing, and recordkeeping procedures;
- Sample custody procedures;
- Quality control procedures, control limits, and documentation;
- Operating conditions of facilities and equipment;
- Documentation of training and maintenance activities; and
- Systems and operations overview.

Critical items for a sampling systems audit include:

- Calibration procedures and documentation for field meters;
- Complete documentation of field logbooks and sampling data sheets;
- Organization and minimization of potential contamination sources while in the field;
- Proper sample collection, storage, and transportation procedures; and
- Compliance with the established chain-of-custody procedures for sample documentation and transfer to the laboratory.

The checklist for each audit will contain detailed questions regarding the critical items, requesting yes/no answers and comments. Examples of standard sampling audit checklists are shown in Figures 11-1, 11-2, and 11-3. A debriefing session is held for all participants to discuss the preliminary audit results. The auditor then completes the audit evaluation and submits an audit report including observations of strengths, deficiencies, and recommendations for improvements.

## 11.2 Performance Audits and Data Quality Audits

Performance audits and data quality audits quantitatively assess the data produced by a measurement system. A performance audit involves submitting certified samples for analysis for each analytical method and/or analytical instrument. The standards for each matrix are selected to reflect the range of concentrations expected for the sampling program. The performance audit answers questions about whether the measurement system is operating within control limits and whether the data produced meet the analytical quality assurance specifications. The data quality audit evaluates data quality indicators, and identifies limitations that may be encountered in data applications. More specifically, critical items for performance evaluation audits are:

- Accuracy and precision of the measurement system;
- The quality control data as compared to the actual data collected;

**GENERAL WATER, SOIL, WASTE, AND WASTEWATER SAMPLING SYSTEMS AUDIT CHECKLIST**

Contract: _____ Date: _____

Site: _____ Auditor: _____

| Yes | No | Comments | Operation |
|---|---|---|---|
| | | | PRESAMPLING OPERATIONS |
| _____ | _____ | _____ | 1. Sample type? (specify) |
| _____ | _____ | _____ | 2. Qualified personnel? |
| _____ | _____ | _____ | 3. Adequate facilities, equipment, and supplies? |
| _____ | _____ | _____ | 4. Sampling locations properly specified? |
| _____ | _____ | _____ | 5. Copy of task instructions or QAPP? Revision #____ |
| _____ | _____ | _____ | 6. Copy of daily sampling schedule? |
| | | | SAMPLING OPERATIONS |
| _____ | _____ | _____ | 1. Samples collected at proper sampling locations? |
| _____ | _____ | _____ | 2. Rinse probe with DI $H_20$ prior to placement? |

Draw diagram describing equipment as used for sampling in the space below.
(Example: type of sample --> Equipment used --> Final packaging.)

_____

(Continued)

**Figure 11-1. Sampling Systems Audit for General Water, Soil, Waste, and Wastewater Sampling**

**GENERAL WATER, SOIL, WASTE, AND WASTEWATER SAMPLING SYSTEMS AUDIT CHECKLIST**
**(Continued)**

| Yes | No | Comments | Operation |
|---|---|---|---|
| | | | SAMPLING OPERATIONS (Continued) |
| ____ | ____ | ____ | 3. Purge appropriate volume prior to sampling (3 well volumes) For this well #____ = ____ gallons. |
| ____ | ____ | ____ | 4. Appropriate sampling technique used to obtain representative sample? |
| ____ | ____ | ____ | 5. Appropriate techniques used to ensure sample integrity and avoid contamination? |
| ____ | ____ | ____ | 6. At least 10% duplicate samples collected? |
| ____ | ____ | ____ | 7. Sufficient volume of sample collected? |
| ____ | ____ | ____ | 8. Suitable sample container used for storage? |
| ____ | ____ | ____ | 9. Sample bottles properly labeled? |
| ____ | ____ | ____ | 10. Sampling data sheet completed in a timely manner? (Within five minutes of activity.) |
| ____ | ____ | ____ | 11. OVA measurements taken and recorded prior to sampling and every 30 minutes during sampling? |

(Continued)

**Figure 11-1. Continued**

**GENERAL WATER, SOIL, WASTE, AND WASTEWATER SAMPLING SYSTEMS AUDIT CHECKLIST**
**(Continued)**

| Yes | No | Comments | Operation |
|---|---|---|---|
| | | | POSTSAMPLING OPERATIONS |
| _____ | _____ | _____ | 1. Decontamination performed according to current procedure? (Soap/water, potable water, Type I or II, reagent grade water, methanol, hexane.) |
| _____ | _____ | _____ | 2. Well capped immediately following removal of pump and prior to decontamination? |
| _____ | _____ | _____ | 3. Sampling date, time, and location properly recorded in logbook? |
| _____ | _____ | _____ | 4. Suitable sample shipping container label used? |
| _____ | _____ | _____ | 5. Chain-of-custody form filled out? |
| _____ | _____ | _____ | 6. Chain-of-custody seal affixed to sample container? |
| _____ | _____ | _____ | 7. Refrigerated sample storage? |
| _____ | _____ | _____ | 8. Overall recordkeeping procedure adequate? |

Additional comments: _____

_____

_____

_____

_____

**Figure 11-1. Continued**

**VOC SAMPLING SYSTEMS AUDIT CHECKLIST**

Contract: _____ Date: _____

Site: _____ Auditor: _____

| Yes | No | Comments | Operation |
|---|---|---|---|
| | | | PRESAMPLING OPERATIONS |
| ____ | ____ | _____ | 1. Qualified Personnel? |
| ____ | ____ | _____ | 2. Spare parts and support equipment available? |
| ____ | ____ | _____ | 3. Sampling apparatus properly assembled and components correctly positioned? |
| ____ | ____ | _____ | 4. Documentation of sample canister preparation? |
| ____ | ____ | _____ | 5. Equipment calibration documentation available? |
| ____ | ____ | _____ | 6. Canister properly labeled? |
| ____ | ____ | _____ | 7. Canisters evacuated and pressure measured? |
| ____ | ____ | _____ | 8. Flow of vacuum regulators set prior to sampling (within QC specifications on control chart)? |
| ____ | ____ | _____ | 9. Sampling system properly purged? |
| | | | SAMPLING OPERATIONS |
| ____ | ____ | _____ | 1. Start/finish times noted and recorded? |
| ____ | ____ | _____ | 2. Coordination of sampling activities? |

(Continued)

**Figure 11-2. VOC Sampling Systems Audit Checklist for Canister Sampling**

**VOC SAMPLING SYSTEMS AUDIT CHECKLIST**
**(Continued)**

| Yes | No | Comments | Operation |
|---|---|---|---|
| | | | SAMPLING OPERATIONS (Continued) |
| ____ | ____ | ____ | 3. Duplicate samples collected? |
| ____ | ____ | ____ | 4. Blank samples collected? |
| ____ | ____ | ____ | 5. Appropriate sampling information recorded in field sample logbook or on field data sheet? |
| ____ | ____ | ____ | 6. Data acquisition system activating samples? |
| | | | POSTSAMPLING OPERATIONS |
| ____ | ____ | ____ | 1. Postsampling canister pressure determined? |
| ____ | ____ | ____ | 2. Canister pressure QC chart completed? |
| ____ | ____ | ____ | 3. Samples properly packaged for shipment? |
| ____ | ____ | ____ | 4. Chain-of-custody documentation completed? |
| ____ | ____ | ____ | 5. Master sample logbook? |
| ____ | ____ | ____ | 6. Bill of lading filed? Laboratory informed of incoming samples and time requirements for next samples? |

Additional comments: _____

_____

_____

_____

**Figure 11-2. Continued**

**SOIL GAS SAMPLING AND ANALYSIS**
**SYSTEMS AUDIT CHECKLIST**
**McCLELLAN AFB**

Contract: _____ Date: _____
Site: _____ Auditor: _____

| Yes | No | Comments | | Operation |
|---|---|---|---|---|
| | | | | **PRESAMPLING OPERATIONS** |
| ___ | ___ | _____ | 1. | What types of samples are being collected? |
| ___ | ___ | _____ | 2. | Are sampling personnel qualified? |
| ___ | ___ | _____ | 3. | Are there adequate facilities, equipment, and supplies? |
| ___ | ___ | _____ | 4. | Were digging permits obtained prior to sampling? |
| ___ | ___ | _____ | 5. | Was a copy of task instructions or work plan on site? |
| ___ | ___ | _____ | 6. | Was a copy of daily sampling schedule available? |
| ___ | ___ | _____ | 7. | Were probes and syringes decontaminated prior to use? |
| ___ | ___ | _____ | 8. | Is presampling decontamination documented? |
| ___ | ___ | _____ | 9. | Was probe blank analyzed prior to sampling? |
| ___ | ___ | _____ | 10. | Were presampling blank criteria met for today's sampling? |
| ___ | ___ | _____ | 11. | Is blank information documented? |

(Continued)

**Figure 11-3. Soil Gas Sampling and Analysis Systems Audit Checklist**

**SOIL GAS SAMPLING AND ANALYSIS SYSTEMS AUDIT CHECKLIST**
(Continued)

| Yes | No | Comments | Operation |
|---|---|---|---|
| | | | SAMPLING OPERATIONS |
| ___ | ___ | _____ | 1. Were samples collected at proper sampling locations? |
| ___ | ___ | _____ | 2. Was probe appropriately placed to correct sampling depth? If not, was this properly documented? |
| ___ | ___ | _____ | 3. Was the generator downwind of sampling location? |
| ___ | ___ | _____ | 4. Was sample pump vacuum set to 5 to 20 inches Hg? |
| ___ | ___ | _____ | 5. Was probe retracted in 2 to 3 inches? How is this measured? |
| ___ | ___ | _____ | 6. Were at least three probe volumes purged prior to sampling? For this probe # ____ = _____ seconds? |
| ___ | ___ | _____ | 7. Was appropriate sampling technique used to obtain representative sample? |
| ___ | ___ | _____ | 8. Were appropriate techniques used to ensure sample integrity and avoid contamination? |
| ___ | ___ | _____ | 9. Was a sufficient volume of sample collected? |
| ___ | ___ | _____ | 10. Were at least 5% duplicate samples collected? |
| ___ | ___ | _____ | 11. Were syringes labelled appropriately? |
| ___ | ___ | _____ | 12. Were suitable sample containers used for short-term storage and transfer to on-site lab? (Should minimize exposure to light.) |
| ___ | ___ | _____ | 13. Was the sampling data sheet and chain-of-custody completed by the samplers in a timely manner (within 5 minutes of activity)? |
| ___ | ___ | _____ | 14. Were PID measurements taken and recorded (as per the Health and Safety Plan)? |

**Figure 11-3. Soil Gas Sampling and Analysis Systems Audit Checklist (continued)**

SOIL GAS SAMPLING AND ANALYSIS SYSTEMS AUDIT CHECKLIST
(Continued)

| Yes | No | Comments | Operation |
|---|---|---|---|
| | | | POSTSAMPLING OPERATIONS |
| ___ | ___ | _____ | 1. Was decontamination performed according to current procedure? Specify. |
| ___ | ___ | _____ | 2. Was the hole sealed immediately following removal of probe? |
| ___ | ___ | _____ | 3. Was chain-of-custody form filled out appropriately? |
| ___ | ___ | _____ | 4. Was the overall recordkeeping procedure adequate? |
| | | | ANALYTICAL OPERATIONS |
| ___ | ___ | _____ | 1. Are analytical personnel qualified? |
| ___ | ___ | _____ | 2. Was sample analyzed within 2 hours of sampling? |
| ___ | ___ | _____ | 3. Was the daily multipoint calibration valid and documented? |
| ___ | ___ | _____ | 4. Is the traceability information adequate for the standards in use? |
| ___ | ___ | _____ | 5. Were 20% duplicate analyses performed? |
| ___ | ___ | _____ | 6. Do duplicate analyses meet the 30% relative percent difference (RPD) acceptance criteria? |
| ___ | ___ | _____ | 7. Have the QC check sample results been calculated and documented in a timely manner? |
| ___ | ___ | _____ | 8. If any compound in the QC check sample exceeded acceptance criteria, were subsequent soil gas samples flagged appropriately? |
| ___ | ___ | _____ | 9. Was the chain-of-custody record completed in a timely manner? |
| ___ | ___ | _____ | 10. Was the analytical results summary form completed? |

**Figure 11-3. Soil Gas Sampling and Analysis Systems Audit Checklist (continued)**

SOIL GAS SAMPLING AND ANALYSIS SYSTEMS AUDIT CHECKLIST
(Continued)

| Yes | No | Comments | Operation |
|---|---|---|---|
| | | | ANALYTICAL OPERATIONS (Continued) |
| ___ | ___ | _____ | 10. Were blanks analyzed following high level samples? |
| ___ | ___ | _____ | 11. Was an analytical blank performed? Was the blank acceptable? |
| ___ | ___ | _____ | 12. Are written standard operating procedures available? |
| ___ | ___ | _____ | 13. Are raw data archived in an acceptable manner? |
| | | | DATA REPORTING |
| ___ | ___ | _____ | 1. Is the data entry procedure adequate to minimize errors in transcription? |
| ___ | ___ | _____ | 2. Are the entries checked (as per project instructions)? |

Additional comments: _____

_____

_____

_____

_____

_____

**Figure 11-C. Soil Gas Sampling and Analysis Systems Audit Checklist (continued)**

- The measurement system operation within established control limits; and
- Significant deviations of quality over time.

These items can identify when a system is outside of acceptable control limits; however, the appropriate corrective action may not always be evident. Appropriate corrective action must be identified in conjunction with the results from the systems or data quality audit.

Specific items for data quality audits include:

- Adequacy of data recording and transfer;
- Precision and bias of resultant data;
- Adequacy of data calculation, generation, and processing;
- Documentation of procedures; and
- Identification of data quality indicators to inform users of limitations and applicability.

Audits of data quality answer questions of whether the data collection efforts need modifications, and whether the use and documentation of quality control procedures are adequate.

The results of all audits performed for McClellan AFB programs will be summarized by the auditor, and reviewed by the project QAO. Copies of the audit results and summary will be transmitted to the project QAO, project manager, quality control task leader, and audit program staff. This will also include discussion of recommended corrective actions or procedural changes, as indicated by the audit results. The audit results and discussion will be incorporated into the QA report for the task or sampling effort (see Section 15.0) and the original audit report will be retained in the project file. Implementation of recommendations will be assessed during subsequent audits or as follow-up activities within the QC task.

# 12.0 PREVENTIVE MAINTENANCE

The primary objective of a preventive maintenance program is to promote the timely and effective completion of a measurement effort. The preventive maintenance program is designed to minimize the downtime of crucial sampling and/or analytical equipment due to expected or unexpected component failure. In implementing this program, efforts are focused in three primary areas:

- Establishment of maintenance responsibilities;
- Establishment of maintenance schedules for major and/or critical instrumentation and apparatus; and
- Establishment of an adequate inventory of critical spare parts and equipment.

Each of these is discussed in the following subsections.

## 12.1 Maintenance Responsibilities

Equipment and apparatus used in environmental measurement programs fall into two general categories:

- Equipment permanently assigned to a specific laboratory (e.g., gas chromatography [GC] laboratory, gas chromatography/mass spectrometry [GC/MS] laboratory, etc.); and
- Field sampling equipment available for use on an as-needed basis (e.g., field meters, pumps, vehicles, etc.).

Maintenance responsibilities for laboratory instruments are assigned to the respective laboratory managers. The laboratory managers then establish maintenance procedures and schedules for each major equipment item. This responsibility may be delegated to laboratory personnel, although the managers retain responsibility for ensuring adherence to prescribed protocol. All laboratories are bound by analytical contractual agreements to maintain the ability to produce data that meet the project objectives and to

follow method specifications. This ensures that adequate spare parts, maintenance, schedules, and emergency repair services are available. Maintenance procedures, logbooks, and documentation of maintenance contracts for major instruments are reviewed during laboratory audits, and are described in the laboratory QA plan and method-specific SOPs.

Maintenance responsibilities for field equipment are assigned to the project director and task leaders for specific sampling tasks. However, the field team using the equipment is responsible for checking the status of the equipment prior to use, and reporting any problems encountered. The field team is also responsible for ensuring that critical spare parts are included as part of the field equipment. Critical spare parts are included in the field equipment checklist.

## 12.2 Maintenance Schedules

The effectiveness of any maintenance program depends to a large extent on adherence to specific maintenance schedules for each major equipment item. Other maintenance activities are conducted on an as-needed basis. Manufacturers' recommendations provide the primary basis for the established maintenance schedules, and manufacturers' service contracts provide primary maintenance for many major instruments (e.g., GC/MS instruments, atomic absorption spectrometers, analytical balances, etc.). Maintenance activities are documented in a maintenance log, which indicates the required frequency for each procedure and also provides for dated entries.

## 12.3 Spare Parts

Along with a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment down time. The inventory includes those parts (and supplies) that:

- Are subject to frequent failure;
- Have limited useful lifetimes; or
- Cannot be obtained in a timely manner should failure occur.

Field sampling task leaders and the respective laboratory managers are responsible for maintaining an adequate inventory of spare parts. In addition to spare parts

and supply inventories, the contractor should maintain an in-house source of backup equipment and instrumentation.

Table 12-1 lists the maintenance tasks, schedule, spare parts, and standard operating procedures for field equipment.

**TABLE 12-1. PREVENTIVE MAINTENANCE SCHEDULE AND CRITICAL SPARE PARTS FOR McCLELLAN AFB RI/FS FIELD EQUIPMENT**

| Equipment | Maintenance Task | Frequency | Critical Spare Parts | SOP |
|---|---|---|---|---|
| Well Wizard® Engine | Check oil level, quarterly tuneups (oil change, air filter replacement). | Daily quarterly -- after each sampling round. | Oil, spark plugs, air filters, backup available on site. | Refill if needed. Tuneup done by local shop. Backup engines. |
| Well Wizard® Compressor | Replace air filter and drive belt. | As needed. | Air filter belt. | Send to manufacturer for other repairs. |
| Well Wizard® Control Box | Release moisture vent. | Hourly. | Backup available over night. | Send to manufacturer for repairs. |
| pH Meters | Keep electronic parts dry.<br>Keep pH sensor wet. | Continuously.<br>Continuously. | Backup available on site. | Send to manufacturer for repairs. |
| Conductivity Meter | Keep electronic parts dry. | Continuously. | Backup available on site. | Send to manufacturer for repairs. |
| Well Wizard® Bladder Pump | Repair broken bladder. | As needed. | Backup available on site. | Replace and send to manufacturer. |
| Water-Level Meters | Replace battery. | As needed. | Backup available on site. | |
| Water Wagon Pump | Tuneup.<br>Check oil level. | Yearly.<br>Weekly. | Oil. | Send to manufacturer for repairs. |
| OVA | Primary filter cleaned and air sampling system checked for leaks.<br>Multipoint calibration.<br>Hydrogen gas cylinder purged. | Daily.<br>Weekly.<br>Bimonthly. | "O" rings. | Send to manufacturer for repairs, backup available. |

(Continued)

TABLE 12-1. (Continued)

| Equipment | Maintenance Task | Frequency | Critical Spare Parts | SOP |
|---|---|---|---|---|
| OVM/MicroTIP | Check filter. | Weekly | Spare filters. | Same as above. |
| Well Wizard® Engine | Check oil level, quarterly tuneups (oil change, air filter replacement). | Daily quarterly -- after each sampling round. | Oil, spark plugs, air filters, backup available on site. | Refill if needed. Tuneup done by local shop. Backup engines. |
| hnu® | Glass plate and grill in probe cleaned.<br>Multipoint calibration. | Daily.<br>Weekly. | Photoionization lamp, batteries. | Send to manufacturer for repair, backup meters available. |
| Sampling vehicles | Tuneup.<br>Check oil level. | Yearly or as needed.<br>Daily (or at least weekly). | Oil, spare tire. | Small tool kit in each vehicle. |
| Sampling Chassis (S.C.) | Tuneup.<br>Check oil level. | Yearly or as needed.<br>Weekly. | Oil, spark plugs. | Small tool kit in each vehicle. |
| Hydraulics System (S.C.) | Check hydraulics. | Daily. | Hydraulic fluid. | Send to authorized repair facility. |
| Generator (for S.C.) | Check oil. | Weekly. | Oil. | Refill as needed. |
| Steam Cleaner (for S.C.) | Replace ignitors. | As needed. | | Send to authorized repair facility. |

# 13.0 DATA ASSESSMENT PROCEDURES

The assessment of measurement data is required to ensure that the quality assurance (QA) objectives for the program are met, and that quantitative measures of data quality are provided. The data assessment calculations and applications used for the McClellan Air Force Base (AFB) Remedial Investigation/Feasibility Study (RI/FS) are based on the United States Environmental Protection Agency (U.S. EPA) Guidelines for Assessing and Reporting Data Quality for Environmental Measurements, January 1983. McClellan project data assessment and validation procedures are provided in the Data Validation SOP (Appendix A).

A distinction must be made between routine quality control that is conducted as a part of laboratory operations, and the project-related data assessment process conducted after the data have been reported. It must be assumed that the planning, standard procedures, and monitoring activities conducted during the sampling and analysis process have served to control the process as much as possible to produce data of sufficient quality for project needs. After the data have been reported, it is necessary to identify any part of the process that could not be controlled, and to what extent that may affect the quality of the reported data.

The routine quality control procedures conducted in the laboratory are established in the published methods, this document, and the analytical standard operating procedures (SOPs). The laboratory is responsible for following those procedures and operating the analytical systems within statistical control limits. These procedures include proper instrument maintenance, calibration and calibration checks, and internal quality control sample analyses at the required frequencies (i.e., reagent blanks, surrogate spikes, matrix spike/matrix spike duplicate [MS/MSD], laboratory control samples, laboratory duplicates). When reporting the sample data, the laboratory is required to provide the results of associated QC sample analyses so the project staff can evaluate the performance of the analytical process.

Problems occur in spite of all precautions taken in planning and execution of the sampling and analysis task. In these cases, the data assessment conducted by the project QA staff after the data have been reported must identify the problem, determine which data are affected, state how these data may be limited for use in the intended applications, and make recommendations for corrective actions as necessary.

The discussion of data assessment presented in this section pertains to the project-related assessment of data that have been reported after laboratory analyses have been completed.

Data assessment procedures established for the McClellan AFB RI/FS include:

- Initial review of analytical and field data for complete and accurate documentation, holding time compliance, and required frequency of QC samples;
- Evaluation of blank results to identify systematic contamination;
- Statistical calculations for accuracy and precision using the appropriate quality control sample results;
- Estimates of completeness, in terms of the percent of valid unqualified data; and
- Assigning data qualifier flags to the data as necessary to reflect limitations identified by the process.

Qualified data will be discussed in the task reports, and data flags will be transmitted to users via data tables from the database and in analytical data reports.

Some basic statistical calculations used in the data assessment process are presented in Table 13-1, and a discussion of specific applications to environmental sample results is presented. Additional statistical measures may be applied to the data to assess quantitation limits or other quality-related parameters. The calculations and procedures used will be documented in the technical report where the results are presented.

## 13.1 Blank Data Assessment

Reagent blank results indicate whether any of the contaminants reported in sample results may be attributed to laboratory sources (reagents, glassware, instrumentation) and were not likely present in the sampled medium. The most common laboratory contaminants are methylene chloride, phthalates, acetone, toluene, and 2-butanone; these are

## TABLE 13-1. STATISTICAL CALCULATIONS

| Statistic | Symbol | Formula | Definition | Uses |
|---|---|---|---|---|
| Mean | $\bar{X}$ | $\left(\sum_{i=1}^{n} X_i\right) / n$ | Measure of central tendency | |
| Standard Deviation | S | $\left[\sum_{i=1}^{n} \left(X_i - \bar{X}\right)^2 / n-1\right]^{1/2}$ | Measure of relative scatter of the data | |
| Relative Standard Deviation | RSD | $\left(S / \bar{X}\right) \times 100$ | Relative standard deviation, adjusts for magnitude of observations | Used to assess precision for replicate results |
| Pooled RSD | $RSD_P$ | $\left[\sum_{i=1}^{n} (RSD_i)^2 df_i / \sum_{i=1}^{n} df_i\right]^{1/2}$ | Measure of overall variability of a series | Used to assess overall performance for compounds with multiple measurements |
| Relative Percent Difference | RPD | $\left[(X_1 - X_2) / (X_1 + X_2)/2\right] \times 100$ | Measure of variability that adjusts for the magnitude of observations | Used when there are only two observations; mathematically related to RSD |

(Continued)

TABLE 13-1. (Continued)

| Statistic | Symbol | Formula | Definition | Uses |
|---|---|---|---|---|
| Relative Percent Difference | RPD | $RSD\ (2)^{1/2}$ | SAME AS ABOVE | SAME AS ABOVE |
| Average Relative Percent Difference | $\overline{RPD}$ | $\frac{RPD}{n}$ | Average relative percent difference--analogous pooled RSD for duplicate measurements | Used to assess overall performance for compounds with multiple measurements |
| Confidence Interval | CI | $X \pm t_{(\alpha,n-1)} S / n^{1/2}$ | Interval about X that contains the true value, with probability $\alpha$ | Assign intervals or error bars to measurement data |
| Percent Recovery | R | $(X_{meas} / X_{true}) \times 100$ | Recovery of spiked compound in pure matrix | Recovery of QCCS, method spikes |
| Percent Recovery | R | $\lvert$ value of spiked sample $-$ value of unspiked sample $\rvert$ / Value of added spike $\times 100$ | Recovery of spiked compound in sample matrix | MS and MS/MSD recovery |

X = Observation (concentration)
n = Number of observations
df = Degrees of freedom, usually (n-1)
t = Statistic from Students' "t" distribution

recognized as being ubiquitous in the laboratory environment and controlling them to within acceptable low levels is part of standard laboratory procedures.

If contamination from these compounds is reported in reagent blanks, the samples associated with the blank, either the same analytical or extraction batch, may be qualified to indicate that some or all of these compounds may be from laboratory sources. If the concentrations reported in the samples are similar to the blank concentrations, it is likely that all of the contamination was introduced, and this assessment is made in the QA/QC report for the sampling task. Samples with blank contamination problems will be assigned a data qualifier flag.

Results for other types of blanks such as equipment, ambient, or trip blanks are assessed individually. The probable source of contamination is identified and the associated sample results are qualified as necessary. For example, if equipment blank results show contamination, and the sample collected from the bailer shows the same compound, the sample results will be qualified to indicate the probable level of introduced contamination. Samples collected before and after the blank are also evaluated to determine the potential sources and impacts of carryover from the sampling equipment.

## 13.2 Accuracy

As previously defined, accuracy is associated with correctness, and is a comparison between a measured value and a known, or 'true' value. Accuracy is calculated from matrix spike, Laboratory Control Sample (LCS) results, and surrogate spike results.

Spike results are reported by the laboratory as percent recovery and are compared to the accuracy objectives stated in Section 4.0. Results that do not satisfy the objectives are assigned a data qualifier flag to indicate uncertainty associated with inaccuracy.

Laboratory Control Samples are spikes of a subset of method analytes into a water matrix. The LCSs are taken through sample preparation and analysis to assess statistical control of the method. If LCS recovery is outside the established limits, samples from the same preparation and/or analytical batch may be influenced. The system must be assessed to determine the reason for the out-of-control occurrence, and corrective action may be indicated, up to and including reanalysis of affected samples. Matrix spike results are

generally more sample-specific. If a systematic interference is exhibited, results for samples collected from similar conditions and/or handled in the same batch will be examined. If any results appear atypical and could be related, those results may also be qualified. The flagged data will be discussed in the QA/QC report for the sampling task, and specific limitations such as poor or enhanced recovery for specific compounds will be stated. Further investigation or corrective action may be taken to find methods to reduce the interferences.

Surrogate spike results are also reported and used to assess recovery of target analytes on a sample by sample basis and provide a measure of system performance as well as matrix interference. Surrogate spike recoveries are compared to recovery limits. Any results outside the limits are flagged on laboratory reports and in the database. Any corrective action taken in the laboratory is documented in laboratory performance records and/or discussed in the comment section of the data report.

Confidence intervals can be calculated for an analytical method if performance evaluation samples are submitted or a series of LCSs are analyzed. The results are used to define confidence intervals (see Table 13-1) for the recovery of each compound analyzed.

## 13.3 Precision

Precision is a measure of variability between duplicate or replicate analyses, and is calculated for field and laboratory replicates. By definition, field or total, precision incorporates laboratory precision. Precision is calculated as the relative percent difference (RPD) between duplicate samples or analyses, or matrix spike/matrix spike duplicates as appropriate. The calculated RPDs are compared to the objectives stated in Section 4.0. Results that do not satisfy the objectives are assigned a data qualifier flag indicating uncertainty associated with imprecision.

An average RPD may be calculated and reported as a measure of overall analytical precision for compounds with multiple measurements. The specific samples collected or analyzed in duplicate are flagged if they do not satisfy the QA objectives. In addition, associated samples may be flagged to indicate variability due to poor precision. For poor field duplicate precision, samples collected by the same sampling team, from the same equipment, from similar matrices (soil samples), or on the same day may be affected; close evaluation of those results should indicate the most likely source of variability, and the corresponding samples will be qualified as warranted. For poor laboratory precision,

samples processed and analyzed in the same batch will be more closely evaluated, and any anomalous results will be qualified.

The QA coordinator is responsible for ensuring that data qualifier flags are assigned to the data as required by the established QC criteria, and that they are reported and understood by project staff using the data for specific applications. The QA coordinator is also responsible for initiating corrective actions for analytical problems identified during the QC data assessment process. These corrective actions range from verifying that the method was in statistical control during the analytical runs, to re-analysis of the sample, or resampling.

## 13.4 Completeness

Completeness is calculated after the QC data have been evaluated, and the results applied to the measurement data. In addition to results identified as being outside of the QC limits established for the method, the occurrence of matrix effects, broken or spilled samples, or samples that could not be analyzed for any other reason are included in the assessment of completeness. The percentage of valid, unqualified results is reported as completeness.

The completeness percentage is calculated by dividing the number of valid, unqualified results minus numbers of possible results not analyzed because of broken or spilled samples, etc., by the possible total number of individual analyte results.

For example, if from a total of 1,000 individual analyte-measurement results (both detected and nondetected), 40 detected results were qualified and 10 analyte results were not reported because of a spilled sample, the completeness percentage is calculated as follows:

$$\frac{950}{1{,}000} \times 100\% = 95\%$$

# 14.0 CORRECTIVE ACTION

During the course of the McClellan Air Force Base (AFB) Remedial Investigation/Feasibility Study (RI/FS) Program, it is the responsibility of the Project Manager, Task Leaders, Quality Assurance (QA) Coordinator, and sampling team members to see that all measurement procedures are followed as specified and that measurement data meet the prescribed acceptance criteria. In the event a problem arises, it is imperative that prompt action be taken to correct the problem. The corrective action process described here is specific to Radian Corporation. Each contractor should have an equivalent, formal process in place to ensure that needed corrective actions are taken and documented.

## 14.1 Corrective Action Report (CAR)

Problems that require corrective action are documented by the use of a Corrective Action Report or a similar type of report, as presented in Figure 14-1. The on-site QA Coordinator, task leaders, or other project member initiates the corrective action request in the event that quality control (QC) results exceed acceptability limits, or upon identification of some other problem or potential problem and is followed up by the Technical Director or QA Officer. Corrective action is also initiated by the QA Coordinator based upon QC data or audit results. Corrective actions range from use of data qualifier flags, to reanalysis of the sample or samples affected, to resampling and re-analysis, to recommending a change in procedures, depending upon the severity of the problem.

## 14.2 Recommendation for Corrective Action (RCA)

A system for issuing formal Recommendations for Corrective Action (RCAs) should exist for addressing problems signaling significant and systematic deficiencies identified through independent quality assurance review. Recommendations for corrective actions are issued only by a member of the Quality Assurance (QA) Group, or a designee in a specific QA role. Each RCA addresses a specific problem or deficiency, usually identified during QA audits of laboratory or project operation. An example RCA form is presented as Figure 14-2. Each of these formal written recommendations requires a written response from the responsible party (i.e., to whom the RCA was issued). A summary of the "unresolved" RCAs is prepared by the QA group on a monthly basis and issued to Radian management. These reports list all RCAs that have been issued, the manager responsible for the work area, and the current status of each RCA. Each RCA requires the response and

# Corrective Action Report (CAR)

## Part I Initial Information (Furnished by Originator)

Originator: _____

SAM #: _____ Method #: _____

Client: _____ Matrix: _____

Date:

Urgency Level:

Requires resolution for immediate job ☐

Requires resolution for future jobs ☐

To Person Responsible for Action: _____

**Present Situation Requiring Action:**

Site / Lab: Type: QC Limit ☐ Documentation ☐ System ☐ Other ☐

Date / Time Identified:

Description of Situation: (attach supporting data if available)

**Recommended Corrective Action or Improvement:**

Description: Implemented by:

Figure 14-1. Corrective Action Report

**Part II Reply / Resolution (Furnished by Technical Director)**

Proposed by: Date: CAR #:

Description: Scheduled Implementation:

**Part III Implemented Correction Action (by: )**

Description: Date Implemented:

**Part IV Follow-Up Required: Yes ☐ No ☐ (by: )**

Verified by: Date: Comments:

**Figure 14-1. Corrective Action Report (continued)**

RESEARCH & ENGINEERING

# RECOMMENDATION FOR CORRECTIVE ACTION

**A. Initial Information**

| RCA NO.: | DATE: | URGENCY LEVEL ☐ |
|---|---|---|
| ORIGINATOR: | APPROVED BY: | 1. Potential for major data loss or invalidation.<br>2. Potential for failure to achieve data quality objectives.<br>3. Suggested improvement. |
| ORGANIZATION/INDIVIDUAL RESPONSIBLE FOR ACTION: | | |

**B. Problem Identification**

| SITE/LAB: | SYSTEM: | DATE PROBLEM IDENTIFIED: |
|---|---|---|
| DESCRIPTION OF PROBLEM: | | |

**C. Recommended Corrective Action**

| DESCRIPTION: | IMPLEMENT BY: |
|---|---|

**D. Problem Resolution**

| PLANNED CORRECTIVE ACTION: | PROPOSED BY: | DATE PROPOSED: | SCHEDULED IMPLEMENTATION: |
|---|---|---|---|
| IMPLEMENTED CORRECTIVE ACTION: | | | DATE IMPLEMENTED: |

**E. QA Verification**

| VERIFIED BY: | DATE: | COMMENTS: |
|---|---|---|

White: Return for Implemented Corrective Action  Yellow: Return for Planned Corrective Action  Pink: File copy  Gold: Originator's Copy

Information Copies Distribution: _____ _____ _____ _____ _____ _____ _____

Figure 14-2. Recommendation for Corrective Action

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verification by the QA group that the corrective action has been implemented before the status is changed on the monthly report. In the event that there is no response to an RCA within 30 days, or the proposed corrective action is disputed, the recommendation and/or conflict is pursued to successively higher management levels until the issue is resolved. The corrective action scheme is shown in the form of a flow chart in Figure 14-3.

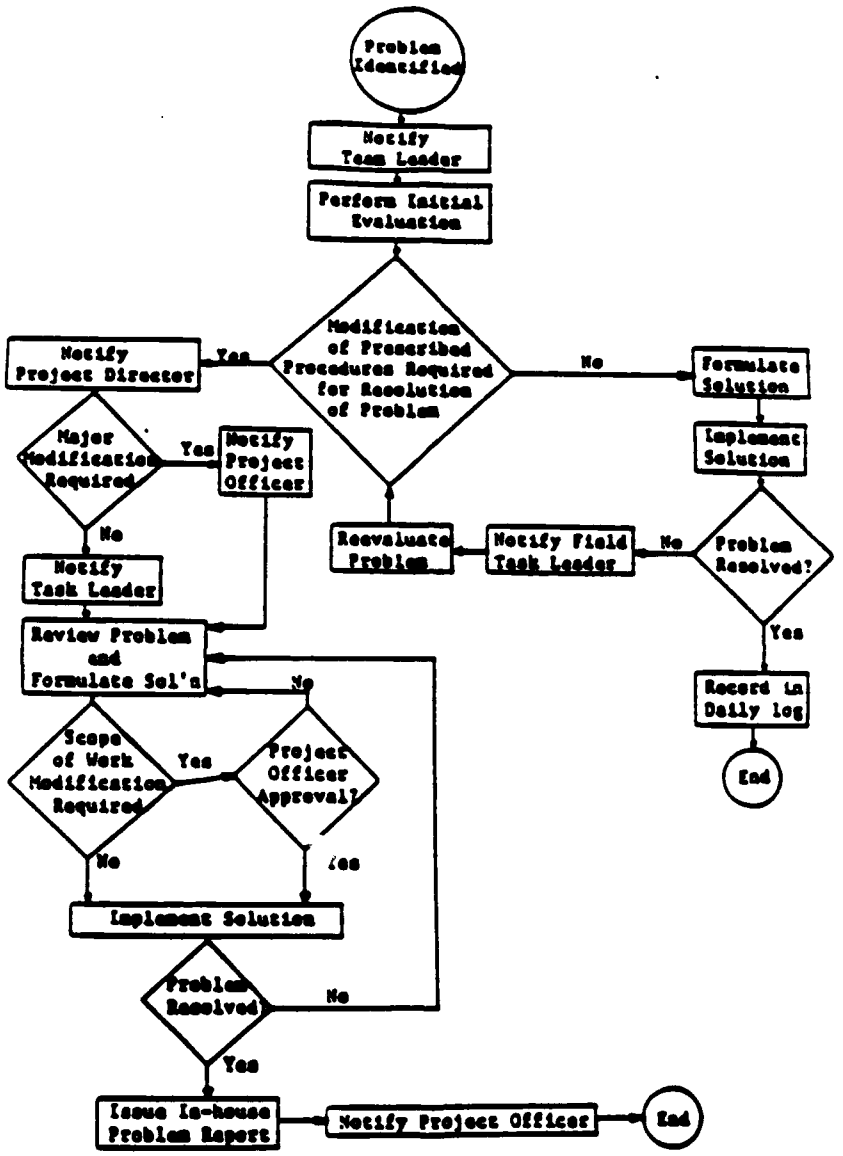


**Figure 14-3. Radian Corrective Action Flowchart**

0491-078-59

## 15.0 QUALITY ASSURANCE REPORTS

The Quality Assurance (QA) Coordinator and Quality Control (QC) task members will issue quality assurance reports to the project management, task leaders, and laboratory supervisors describing the results of QC measurements, performance audits, and systems audits performed for each sampling and analysis task. Quality assurance reports for tasks that involve collecting samples over a long-term period (one quarter or longer) will be prepared on a monthly or quarterly basis as appropriate. Audit results will be summarized in the reports; detailed audit results and checklists will be submitted according to the procedures described in Section 11.0. The information presented in the quality assurance reports will be incorporated as an appendix into the reports prepared for the task or in separate task QA/QC reports, and follow United States Environmental Protection Agency (U.S. EPA) format or a format specified in the statement of work (SOW) for the task.

In addition to reporting QA activities and QC data assessment results in technical reports and memoranda as described, a summary of QA/QC activities for all current RI/FS activities will be provided in the monthly progress reports. This will include discussion of audits conducted or planned, any specific problems identified or resolved during the reporting period, and provide an overview of general QA/QC activities conducted in support of field work and reporting for all RI/FS tasks.

An example of the content and format for Quality Assurance Reports is presented in the following outline:

McCLELLAN AIR FORCE BASE
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
QUALITY ASSURANCE REPORTS

1.0 Summary of sampling and analytical activity and highlights of quality assurance results

2.0 Measurement data accuracy, precision, and completeness (per sample matrix and method)

3.0 Results of systems audits

4.0 Results of performance audits

5.0 Significant quality assurance problems and recommended action

## 15.1 Quality Assurance and Quality Control Reporting Formats

Two types of quality control results will be reported as appropriate for each sampling and analytical task:

- Sampling Quality Control
  - -- Equipment blank analyses
  - -- Trip blank analyses
  - -- Ambient blank analyses
  - -- Field duplicate sample analyses
- Analytical Quality Control
  - -- Laboratory duplicate analyses
  - -- Matrix spike analyses
  - -- Reagent blank analyses
  - -- Matrix spike duplicate analyses
  - -- Laboratory Control sample analyses
  - -- Surrogate spike analyses

These data will be reported in tables by method and matrix, as described in Section 9.0.

Tables summarizing all QC data for the task will be prepared. The range of the results for each type of data (blanks, spikes), the total number of samples, and number of acceptable results will be indicated.

## 16.0 SITE MANAGEMENT

This section briefly discusses the general aspects of Site Management.

### 16.1 Base Point of Contact (BPOC)

SM - ALC/EMR, QA Officer
Building 250 HH
McClellan AFB, CA 95652-5990
(916) 643-1250

The Quality Assurance (QA) Officer will observe field activities to ensure tasks are conducted according to Project Work Plans and Quality Assurance Project Plan (QAPP).

### 16.2 Coordination of Field Activities

Field tasks such as the implementation of a remedial investigation will require the organization, scheduling and other field logistics for contractor personnel and their subcontractors. An experienced contractor geologist or engineer will provide program logistics and communication between the Air Force, the field teams, subcontractors, county inspection personnel, and contractor task management regarding the routine, daily activities. The field coordinator will be equipped with a telephone paging device which will improve field communications, and field activities will be conducted from an office located in the contractors' lot at McClellan Air Force Base (AFB).

### 16.3 Location of Field Office Facilities

Each contractor will direct field operations from an office located at the contractor's lot at McClellan AFB. These facilities will provide contractor and subcontractor personnel with office space and telephone access.

### 16.4 Site and Equipment Security

Certain field activities will require the use of a drilling or similar subcontractor. The subcontractor will erect a temporary chain link fence in the vicinity of the contrac-

tor's lot in which to store equipment and supplies. When activities require equipment to be left overnight at a site on base, all equipment and vehicles will be secured to prevent any unauthorized removal. In the event that equipment is required to be left overnight at a site off base, the subcontractor will be responsible for providing security.

## 16.5 Sources of Potable Water

There are two sources of base-supplied potable water in the vicinity of the contractor's lot located west of Building 685. There is a spigot and an overhead tank filling discharge line located within a few hundred feet west of Building 685.

## 16.6 Location of Decontamination Area

Decontamination of field equipment will occur at a designated area. Groundwater sampling equipment will be decontaminated at the Groundwater Treatment Plant (GWTP). Equipment used for other field activities (i.e., drilling, soil sampling) will be decontaminated at a designated area near the contractor's staging area.

## 16.7 Site Access

Sites requiring access permission will occur both on and off base. Access for sites on base will be coordinated through McClellan AFB Environmental Management (EM) as per Air Force policy. This includes obtaining digging permits, and coordination with facilities and units where site activities will take place. Off-base site access and acquisition of required property easements will be conducted by the U.S. Army Corp of Engineers in coordination with the Air Force. The contractor will assist these organizations whenever possible and will obtain the Sacramento County Health Department application and water well permits.

## 16.8 Cuttings and Groundwater Disposal

Soil cuttings produced during drilling operations or other field activities will be screened for the presence of volatile organic compounds (VOCs) and stored in designated waste bins or drums on base, as described in Section 5.2. These materials will be disposed according to the protocol prescribed in the following document (Figure 16-1) or as directed under the McClellan AFB Soils and Debris Management Plan (Radian, 1991).

EMC/G. Burton/lf/23Jan93/REMOVAL.DOC

EM 23 JAN 1986

Removal of Material/Debris from Base

SP

1. Effective immediately, request you implement the following procedures to ensure proper disposition of all excavated material/debris leaving the base.

a. All vehicles carrying loads of excavated material or debris will be stopped by the sentries prior to leaving the gate.

b. The sentries will check to see if the vehicle operator has a completed Refuse Material Transportation Permit (Attachment 1) or a copy of a Uniform Hazardous Waste Manifest (Attachment 2).

c. The sentry will note the date and time the vehicle leaves the base and return the completed Refuse Material Transportation Permit to EM. Copies of the uniform hazardous waste manifest do not need to be returned to EM.

2. Vehicles not having the above documentation cannot leave the base and must be sent back to the job site until proper approval is obtained.

3. These procedures do not apply to routine garbage/refuse U.S. Eagle trucks that service the base.

4. Questions or comments regarding these requirements may be directed to Mr Gary Burton, EMC, 3-1250.

Signed

J. THOMAS LAMELL, Col, USAF
Director, Environmental Mgmt

2 Atch
1. Refuse Material Transportation Permit
2. Uniform Hazardous Waste Manifest

Figure 16-1. Protocol for Removal of Material

Groundwater collected during drilling or sampling operations will be transported to the McClellan AFB Groundwater Treatment Plant for disposal according to base protocol.

## 16.9 Site Health and Safety

A detailed discussion of site and project health and safety is presented in the contractor's Remedial Investigation/Feasibility Study (RI/FS) Health and Safety Plan.

# APPENDIX A

## Standard Operating Procedures for Nonstandard Analytical Methods; Data Validation Standard Operating Procedure

STANDARD OPERATING PROCEDURES

- for -

# The Determination of Volatile Halogenated and Aromatic Organic Compounds

- by -

Gas Chromatography

NET

NATIONAL
ENVIRONMENTAL
TESTING, INC.

# STANDARD OPERATING PROCEDURES

- for -

## The Determination of Volatile Halogenated and Aromatic Organic Compounds

- by -

Gas Chromatography

Applicable to U.S. EPA Methods:
8010 and 8020

NET Pacific, Inc.
435 Tesconi Circle
Santa Rosa, CA 95401

NET Pacific Divisional Manager_____

NET Pacific Regional QA Manager_____

This S.O.P. is suitable for use in all laboratories in the NET Pacific Region. Santa Rosa and Burbank Division magnetic repository: Altos systems/netsr!usr/sop/srmobile.8010.

## Preface

NET Pacific Inc.
Standard Operating Procedures No:

# The Determination of Volatile Halogenated and Aromatic Organic Compounds

- by -

Gas Chromatography

This Standard Operating Procedure consists of copies of the cited reference methods, together with specific procedures which are unique to the NET Pacific, Inc. regional laboratories. This SOP is not complete without the attached reference methods which are presented in the following appendices:

Appendix A:

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The mention of trade names or products does not constitute endorsement by National Environmental Testing, Inc.

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## 1.0 Scope and Application

This SOP is applicable to the analysis of groundwater, liquids, and solids by USEPA methods 8010-M/8020-M which may contain volatile halogenated and aromatic organic compounds. The group of analytes reported is listed in Table 1 along with the reference method and required detector for each analyte.

Because certain modifications to the USEPA methods 8010 and 8020 have been made, the methods described herein are referred to as 8010-M and 8020-M. These modifications do not detract from the effectiveness, or the precision and accuracy of the original methods. The specifics of these changes have been identified in the appropriate sections of this SOP.

The and 8000 series methods are intended for use in the analysis of substances to determine compliance with the USEPA RCRA and State of California LUFT procedures and regulations.

The 601-M and 602-M methods are intended for the analysis of wastewater discharge samples and other various activities related to the Clean Water Act or NPDES program.

## 2.0 Summary of Method

Soil, solid and liquid waste samples, methanolic extracts and groundwater samples (8010-M/8020-M,5030) are subjected to a purge and trap process. This process efficiently transfers the volatile analytes to a gas chromatograph for analysis. The gas chromatograph is temperature programmed to separate the volatile analytes and is equipped with a HECD and PID in series to achieve detection. Positive compound ID is established through second column confirmation.

### 2.1 Sample Preservation and handling

2.1.1 Two 40 ml glass screw cap VOA vials with teflon-faced silicone septum are recommended for all types of samples (minimum volume is 25 ml).

2.1.2 All samples must be iced or refrigerated (4 deg C) from time of collection until analysis. Liquid samples require pH adjustment with HCl (pH< 2) and chlorine elimination (if present) with sodium thiosulfate (10 mg/40 ml per 5 ppm chlorine). Soil samples require minimum air space containment.

2.1.3 All samples must be analyzed within 14 days of collection.

## 3.0 Safety

As a rule, all reagents, pure standard materials, solutions and samples should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) on each known material in the laboratory must be read and understood prior to handling. A NIOSH/MESA approved toxic gas respirator and gloves should be worn when handling high concentrations of carbon tetrachloride, chloroform, 1,4-dichlorobenzene and vinyl-chloride.

Each analyst should be aware of others working nearby, and take necessary precautions for their safety as well.

TABLE 1

| Parameter | EPA Reference | Detector | Std. Mix | ICV |
|---|---|---|---|---|
| Benzene | 8020 | PID | B | Y |
| Bromodichloromethane | 8010 | HECD | B | Y |
| Bromoform | 8010 | HECD | B | Y |
| Bromomethane | 8010 | HECD | C | Y |
| Carbon Tetrachloride | 8010 | HECD | A | Y |
| Chlorobenzene | 8010,8020 | HECD/PID | A | Y |
| Chloroethane | 8010 | HECD | C | Y |
| 2-Chloroethylvinyl ether | 8010 | HECD | A | N |
| Chloroform | 8010 | HECD | A | Y |
| Chloromethane | 8010 | HECD | C | Y |
| Dibromochloromethane | 8010 | HECD | A | Y |
| 1,2-Dichlorobenzene | 8010,8020 | HECD/PID | D | Y |
| 1,3-Dichlorobenzene | 8010,8020 | HECD/PID | D | Y |
| 1,4-Dichlorobenzene | 8010,8020 | HECD/PID | D | Y |
| 1,1-Dichloroethane | 8010 | HECD | A | Y |
| 1,2-Dichloroethane | 8010 | HECD | B | Y |
| 1,1-Dichloroethene | 8010 | HECD | A | Y |
| trans-1,2-Dichloroethene | 8010 | HECD | B | Y |
| 1,2-Dichloropropane | 8010 | HECD | A | Y |
| cis-1,3-Dichloropropene | 8010 | HECD | B* | Y |
| trans-1,3-Dichloropropene | 8010 | HECD | B* | Y |
| Ethylbenzene | 8020 | PID | B | Y |
| Methylene Chloride | 8010 | HECD | A | Y |
| 1,1,2,2-Tetrachloroethane | 8010 | HECD | B | Y |
| Tetrachloroethene | 8010 | HECD | A | Y |
| Toluene | 8020 | PID | B | Y |
| 1,1,1-Trichloroethane | 8010 | HECD | B | Y |
| 1,1,2-Trichloroethane | 8010 | HECD | A | Y |
| Trichloroethene | 8010 | HECD | A | Y |
| Trichlorofluoromethane | 8010 | HECD | A | Y |
| Vinyl Chloride | 8010 | HECD | C | Y |
| Xylenes | 8020 | PID | D | N |

PID - Photoionization Detector
HECD - Hall Electroconductivity Detector

Standard mixes:
A= Purgeable A Supelco cat# 4-8851M 200 ug/ml
B= Purgeable B Supelco cat# 4-8852M 200 ug/ml
C= Purgeable C Supelco cat# 4-8853M 200 ug/ml
D= Custom Prep Supelco cat# 200 ug/ml

* Mixed cis and trans isomers. Porportions stated on data sheet.

ICV
Y= Yes , N= No.

## 4.0 Reagents and Materials

4.1 Apparatus, materials, and reagents are described in the reference methods cited. However, combining of the methods, 601/602 or 8010/8020, requires the use of certain reagents and materials which meet the criteria of each method. These and other additions are as follows:

4.1.1 Trap - 25 cm X 0.105 cm ID stainless steel containing 1/3 2,6-diphenylene oxide polymer (Tenax), 1/3 silica gel, and 1/3 coconut charcoal.

4.1.2 Column 1 - 2 X 2 mm ID stainless steel or glass packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent.

4.1.3 Column 2 - 75m X 0.53 mm ID, 3 um film thickness DB-624 megabore column.

4.1.4 Column 3 - 60 m X 0.75 mm ID, 1.5 um film thickness Vocol wide bore capillary or equivalent.

4.1.5 Detector Coupler - 1/16 in. stainless steel tubing, or 0.53 mm ID inert fused silica tubing at a minimum length so as to join the two detectors (HECD and PID) together. Needed only if detectors have not been joined by the manufacturer.

4.1.6 Methyl Alcohol - Purge and Trap grade (Burdick & Jackson) or equivalent.

4.1.7 Sample Purger (water) - 25 ml fritted sparge glassware.

4.1.8 Sample Purger (solids) - 25 ml needle sparge glassware with 1/2 in. or 3/4 in. mouth for easy loading of soil/solid samples (8010/8020 low level method).

4.1.9 Extraction Vessel (solids) - 16 oz. wide mouth glass jar equipped with a teflon faced screw cap. Used for methanolic extractions of soil/solid samples (8010/8020 high level method).

4.1.10 Gases - Helium, ultra high purity 99.999%. Hydrogen, ultra high purity 99.999%.

4.1.11 Gas Purifiers - oxygen, water, and organic removing gas filters.

4.1.12 Gas Particulate Filters - 15 um or less fritted filters. Placed in-line after gas purifier to remove particulates which may damage instrument parts and/or impede instrument performance.

## 5.0 Interferences

### 5.1 Wastewater:

Refer to Method 601, section 3, page 2, and Method 602, section 3, page 2.

### 5.2 Groundwater, Soil, Solid and Liquid Waste:

Refer to SW-846, Third Edition, Method 5030, Section 3, page 2.

## 6.0 Procedure

### 6.1 Instrument Operation

#### 6.1.1 Daily Start-up Procedure (Check list):

##### 6.1.1.1 Hall Electroconductivity Detector:

- Power switch on
- Cell switch on
- Reactor gas (hydrogen) on (100 ml/min)
- Solvent pump switch on
- Solvent flow set at 0.05 ml/min.
- Reactor heater switch on
- Reactor temperature set at 850 deg F
- Electrometer set at range "X".
- Vent time set at 0.75 min.

Solvent flow in the return line and an orange glow in the reactor tower should be visible.

##### 6.1.1.2 Photoionization Detector:

- Power switch on
- Amplifier switch on
- Lamp switch on
- Temp. reading about 220 deg C
- Electrometer set at attn 2

The blue glow of the UV lamp should be visible at the rear of the lamp housing.

##### 6.1.1.3 Purge and Trap:

- Power switch on
- Valve and desorb line temp. set at 100 deg C
- Trap "purge ready" temp. set at 30 deg C
- Trap desorb temp. set at 180 deg C
- Trap bake-out temp. set at 225 deg C
- Purge time set at 11 min.
- Desorb time set at 4 min.
- Bake-out time set at to at least 6 minutes.
- Purge flow set at 40 ml/min.

6.1.1.4 Gas Chromatograph:

| | | |
|---|---|---|
| Column 1 | : | 2 m X 2 mm ID, silated glass or S.S. |
| Packing | : | 1% SP-1000 on Carbopack B (60/80 mesh) |
| Carrier Gas | : | Helium (UHP) |
| Flow Rate | : | 40.0 ml/min. |
| Injector Temp: | | 200 deg C |
| Detector Base Temp: | | 250 deg C |
| Temp. Program: | | Hold 3 min. at 45 deg C, ramp 8 deg C/min to 220 deg C, hold 15 min. |
| Column 2 | : | 75m x 0.53mm, 3.0 um film |
| Packing | : | DB-624 |
| Carrier Gas | : | Helium (UHP) |
| Flow Rate | : | 8 ml/min. |
| Injector Temp: | | 180 deg C |
| Detector Base Temp: | | 250 deg C |
| Temp. Program: | | Hold 7 min. at 35 deg C, ramp 5 deg C/min to 70 deg C, ramp 12 deg C/min to 110 deg C, hold 2 min, ramp 10 degC/min to 160 deg C, hold 6 min. |
| Column 3 | : | 60 M X 0.75 mm ID, 1.5 um film, VOCOL borosilicate glass column (Supelco) |
| Carrier Gas | : | Helium (UHP) |
| Flow Rate | : | 10 ml/min. |
| Injector Temp: | | 190 deg C |
| Detector Base Temp: | | 250 deg C |
| Temp. Program: | | Hold 4 min. at 35 deg C, ramp 4 deg C/min to 190 deg C, hold 5 min. |
| Make-up Gas | : | Helium (UHP) @ 20 ml/min. |

6.1.2 Shut-Down Procedure:

1. Cool down each instrument to room temperature
2. Turn off gases
3. Turn off power switches.

## 6.2 Instrument Calibration Procedure

### 6.2.1 External Standard Calibration:

Analyze the calibration working standards at each of the five levels described in Section 6.3.3.1 of this SOP. At each level and for each parameter, calculate the response factor,

$$RF = As/Ms$$

where,

As = Area or height counts of the parameter std.
Ms = Mass of the parameter std. purged (ng)

For each parameter of the five point calibration, calculate the following:

1. Average RF (response factor)
2. Standard deviation (SD) of the RF
3. Relative standard deviation (RSD) of the average RF
4. Average retention time
5. SD of the average retention time
6. RSD of the average retention time

### 6.2.2 Internal Standard Calibration:

Follow the procedure above (Section 6.2.1) except calculate the response factor as follows:

$$RF = (As)(Mis)/(Ais)(Ms)$$

where,
As = Area or height counts of the parameter std.
Ms = Mass of the parameter std. purged (ng)
Ais = Area or height counts (same as As) of the internal std.
Mis = Mass of the internal std. purged (ng)

## 6.3 Preparation and Sources of Standards Solutions:

### 6.3.1 Stock Standard Solutions:

Stock standard solutions must be logged into the standards log book upon receipt. Assign the standard the next Working Reference number WR### from the standard log book and complete the log book entry with the following information:

- Supplier name
- Lot Number
- Expiration Date
- List of compounds with concentrations.
- Date Received
- your Initials

Store standards in freezer at -10 to -20 deg C separate from samples.

6.3.1.1 Source of Calibration Standards:

Supelco stock standard solutions "Purgeable A","Purgeable B", "Purgeable C", and a custom stock standard solution containing all three dichlorobenzene isomers and all three xylene isomers, together contain all the analytes of intrest at 200 ug/ml each.

6.3.1.2 Source of Initial Calibration Verification Standard:

A stock standard solution available from Ultra scientific (purge mix-100) is used to prepare the second source standard for initial calibration verification. The concentration of analytes present is 20 ug/ml.

6.3.1.3 Source of Surrogate Spike Solution:

Supelco's 601/8010 internal standard stock solution containing bromochloromethane, 2-bromo-1-chloropropane and 1,4-dichlorobutane at 20,000 ug/ml each is used to prepare the 8010 surrogate spike mixture. Ultra scientific's fluorobenzene stock solution at 2,000 ug/ml is used to prepare the 8020 surrogate spike solution.

6.3.2 Preparation of Working Standard Solutions :

The concentrations of these aqueous standards are in the low ppb range (ng/ml). They are prepared in a 5 ml glass gas-tight syringes, equipped with a teflon syringe valve, from the 5-10 ppm concentrates. Aqueous standards and samples are unstable and fragile. Therefore preparation and handling should be done only at the time of analysis. Avoid shaking, heat, or any action creating bubbles.

Stock standard solutions are combined and diluted in a 10.0 ml class A volumetric flask as follows:

Working Standard solutions must be logged into the standards log book upon preparation. Assign the standard the next Working Standard number WS### from the standard log book and complete the log book entry with the following information:

- Standard Prep (Calibration, Surrogate, etc.)
- Standards Working Reference Number (*WR###*)
- List of compounds with concentrations.
- Standard preparation:
  Initial volume of *WR###* standard used
  Finial Volume of prepared standard
  Solvent used for dilution
  Finial concentration of compounds.

- Date of preparation
- your Initials

Store standards in freezer at -10 to -20 deg C separate from samples. All of the liquid stock standards and subsequent methanolic dilutions must be replaced after 6 months, or earlier if daily check standard comparison indicates a problem.

6.3.2.1 Calibration Standard Concentrate

| Stock Solution | Initial Volume | Finial Volume | Resultant Concentration |
|---|---|---|---|
| Purgeable A | 250 ul | 10.0 ml | 5.0 ug/ml |
| Purgeable B | 250 ul | 10.0 ml | 5.0 ug/ml |
| Purgeable C | 500 ul | 10.0 ml | 10.0 ug/ml |
| Custom DBC's + xylenes | 250 ul | 10.0 ml | 5.0 ug/ml |

Transfer to 1 ml vials. Use one vial per week. Label vials with working standard reference number from standards log book.

6.3.2.2 Initial Calibration Verification Standard Concentrate

| Stock Solution | Initial Volume | Finial Volume | prepared Concentration |
|---|---|---|---|
| Ultra PMX-100 | -- | -- | 20.0 ug/ml |

6.3.2.2 Surrogate Standard Spiking Concentrate.

| Stock Solution | Initial Volume | Finial Volume | Resultant Concentration |
|---|---|---|---|
| Supelco 601/8010 | 50 ul | 10.0 | 100 ug/ml |
| Ultra Fluorobenzene | 500 ul | 10.0 | 100 ug/ml |

6.3.3 Standard Solutions:

6.3.3.1 Calibration Working Standards:

Used for instrument calibration as described in section 12.0 of this SOP. These are prepared at five different concentration levels for each of the 8010/8020 compounds. The aliquot volume of concentrate and corresponding aqueous concentration is listed in table 3. Prepare each level as follows:

1. Fill the syringe, valve closed, with organic free reagent water.
2. Insert the plunger so as not to create any bubbles.
3. Open the valve and compress the plunger to the 5 ml mark (5 ml syringe).
4. Point the syringe tip up and pull back plunger slightly.
5. Through the tip, add the appropriate volume of calibration standard concentrate.
6. Through the tip add 5.0 ul of the surrogate standard spiking concentrate.
7. Close the valve, and mix gently by inverting the syringe several times.
8. Introduce the syringe contents into the purge and trap sparger, and begin the analysis.

TABLE 3

| Aliquot of calib std. Mix (ul) | The 5ppm Std. Analyte conc. in 5 ml water (ppb,ng/ml) | The 10ppm std. analyte conc. in 5 ml water (ppb,ng/ml) |
|---|---|---|
| 2.0 | 2.0 | 4.0 |
| 5.0 | 5.0 | 10.0 |
| 8.0 | 8.0 | 16.0 |
| 20.0 | 20.0 | 40.0 |
| 30.0 | 30.0 | 60.0 |

6.3.3.2 Initial Calibration Verification Working Standard:

1. Fill the syringe, valve closed, with organic free reagent water.
2. Insert the plunger so as not to create any bubbles.
3. Open the valve and compress the plunger to the 5 ml mark (5 ml syringe).
4. Point the syringe tip up and pull back plunger slightly.
5. Through the tip, add 5.0 ul of Initial Calibration Verification standard concentrate.
6. Through the tip, add 5.0 ul of the surrogate spiking concentrate.
7. Close the valve, and mix gently by inverting the syringe several times.
8. Introduce the syringe contents into the purge and trap sparger, and begin the analysis.

TABLE 4

| Aliquot of 20 ppm ICV working Std. (ul) | Analyte conc. in 5 ml water (ppb,ng/ml) |
|---|---|
| 5.0 | 20.0 ng/ml |

6.3.3.3 Preparation of Daily Working Standard:

Used to check the instrument calibration on each working day as described in section 13.0 of this SOP.

1. Fill the syringe, valve closed, with organic free reagent water.
2. Insert the plunger so as not to create any bubbles.
3. Open the valve and compress the plunger to the 5 ml mark (5 ml syringe).
4. Point the syringe tip up and pull back plunger slightly.
5. Through the tip, add 20.0 ul volume of calibration standard concentrate.
6. Through the tip, add 5.0 ul of the surrogate standard spiking concentrate.
7. Close the valve, and mix gently by inverting the syringe several times.
8. Introduce the syringe contents into the purge and trap sparger, and begin the analysis.

TABLE 5

| Aliquot of Calib Std Mix (ul) | Std mix A, B, D Analyte conc. in 5 ml water (ppb,ng/ml) | Std mix C Analyte conc. in 5 ml water (ppb,ng/ml) |
|---|---|---|
| 20.0 | 20.0 ng/ml | 40.0 ng/ml |

6.4 Matrix Spike:

Used to measure the precision and accuracy of sample analysis as described in section 17.0 of this SOP. The sample spike concentration level is determined as follows:

1. Near the same level as that of the analyte found in the sample. Or,
2. At a level in the mid-range of the calibration curve. Or,
3. At the Maximum Contaminant Level (MCL) of the analyte.

6.4.1 Aqueous Sample Spikes:

Prepare by following steps 1 through 8 above (section 6.3.3.1) except substituting the aqueous sample for the organic free water in step 1.

6.4.2 Solid Sample Spikes (low level):

After sample introduction to the sparger, follow steps 1 through 8 above (section 6.3.3.1). No substitutions.

6.4.3 Solid Sample Spikes (high level):

Following the addition of the methanol to the extraction vessel, add the appropriate volume of either a stock standard solution or secondary dilution standard solution of a parameter set (duplicate matrix spike) or surrogate set (surrogate sample spike).

## 6.5 Sample Preparation Techniques

### 6.5.1 Groundwater, Soil, Solid and Liquid Waste:

Soil Prep:

-a Remove sample from refrigeration immediately prior to subsampling for analysis.
-b Remove the protective cap from the soil sample container.
-c Remove the first three quarters to one inch of the sample and discard.
-d With a narrow spatula collect and insert a representative one gram portion of the sample to a pre-tared purge tube.
-e Record the actual weight of sample to 0.01 grams.
-f Add two mls of organic free water.
-g Quickly cap purge tube.
-h Use vortexer to suspend the sample in solution (15 seconds).
-i Connect purge tube to purge & trap three way valve assembly.
-j Fill 5 ml glass gas tight syringe with 3 mls of organic free reagent water.
-k Through the tip, add 10.0 ul of surrogate standard spiking solution.
-l If analysis is Matrix Spike or Matrix Spike Duplicate inject into syringe 10 ul of standard spiking solution.
-m Quickly connect the syringe to the Lur-Lock hub on the three way valve.
-n Set the syringe valve to receive the contents of the syringe. Inject syringe contents into the purge tube.
-o Set syringe valve to purge cycle position.
-p Disconnect syringe from valve assembly.
-q Begin Purge.

-r Rinse syringe three times with organic free water by drawing up to full capacity three times and expelling to waste.
-s Disconnect syringe plunger from housing.

Ground Water Prep:

-a. Fill the syringe, valve closed, with organic free reagent water.
-b. Insert the plunger so as not to create any bubbles.

-c. Open the valve and compress the plunger to the 5 ml mark (5 ml syringe).
-d. Point the syringe tip up and pull back plunger slightly.

-e. Also through the tip, add 10.0 ul of the surrogate standard spiking concentrate.
-f. If sampling is a Matrix Spike or Matrix Spike Duplicate add through the tip, add the 10 ul calibration standard concentrate.
-g. Close the valve, and mix gently by inverting the syringe several times.
-h. Introduce the syringe contents into the purge and trap sparger, and begin the analysis.
-i Rinse syringe three times with organic free water by drawing up to full capacity three times and expelling to waste.
-j Disconnect syringe plunger from housing.

Liquid Waste Prep:

Place about 49 ml of methanol into a short neck 50 ml glass stoppered volumetric flask. Let sit unstoppered for a few minutes to allow wetted surfaces to dry. Weigh the flask to the nearest 0.1 mg. Quickly add five or more drops of the compound to the flask directly above the surface of the methanol, avoiding contact with the neck wall. Reweigh immediately.

Record weight of sample. Bring volumetric flask to volume with methanol. Mix well and perform additional dilutions as required to prepare a 10 ppm by sample weight concentration. Inject 10 ul of diluted sample to 5 mls organic free water and analyze.

6.6 Documentation

6.6.1 Analysis Log Book

Record in the sample preparation and analysis log book the following information:

- Sample weight/volume
- Methanol volume
- Surrogate/matrix spike compounds
- Surrogate/matrix spike concentrations (of the concentrate)
- Surrogate/matrix spike concentrate volume
- Dilutions
- Observations
- Date of extraction and/or analysis

6.6.2 Instrument Run Log Book:

This log book is used to record every instrument run. For each run, the following information is entered:

- Date
- Analyst initials
- Sample or standard I.D.
- Method reference
- Instrument and detector I.D.
- Comments

Note: The analyst signs the log book only after the above criteria (Section 13.1) have been met. And thus, begins the analysis of samples.

6.6.3 Daily Standard Log:

This log (computer file) is used to record every daily standard. for each parameter, the following information is entered:

- Date
- Concentration
- Daily response factor
- Calibration factor
- Calibration date
- Percent deviation
- Retention time
- Organic std. log book reference

The print-out of the daily standard is filed appropriately and the computer file is saved on a diskette to be used later for graphing control charts (% deviation vs. date). Alternatively, the above information can be manually logged and graphed.

6.6.4 Corrective Action Log Book:

This log book is used to document problems, out-of-control situations, or performance criteria failure.

Section 7.14 of this SOP specifies what is to be recorded in the Corrective Action Record.

6.7 Computational Method

6.7.1 External Standard Technique:

Using the analyte calibration factor (determined in Section 12.1 of this SOP), calculate the concentration of each parameter found in the sample as follows:

6.7.1.1 Aqueous Samples:

Concentration (ppb, ng/ml or ug/l) = (Ap)(DF)/(CF)(Vs)

where,

Ap = Area or height counts of the parameter found in the sample
DF = Dilution factor from any dilutions performed on the sample
CF = Calibration factor of the parameter (1/ng)
Vs = Volume of sample purged (ml)

6.7.1.2 Non-Aqueous Samples:

A. Direct Purge (low level method)

Concentration (ppb, ng/g or ug/kg) = (Ap)/(CF)(Ws)

B. Methanolic Extraction (high level method)

Concentration (ppb, ng/g or ug/kg ) = (Ap)(DF)(Ve)/(CF)(Ws)(Va)

Where,

Ap = Area or height counts of the parameter found in the sample
CF = Calibration factor of the parameter (1/ng)
Ws = Weight of the sample (g)
DF = Dilution factor of any dilutions performed on the extract
Ve = Volume of methanol used to extract the sample (ml)
Va = Volume of methanol aliquot purged (ml)

6.7.2 Internal Standard Technique:

Using the parameter calibration factor (determined in Section 12.2 of this SOP), calculate the concentration of each parameter found in the sample as follows:

6.7.2.1 Aqueous Samples:

Concentration (ppb, ng/ml or ug/l) = (Ap)(Mpi)(DF)/(Api)(CF)(Vs)

Where,

Ap, DF and Vs have the same definitions as above in Section 15.1.1
Mpi = Mass of the internal std. purged (ng)
Api = Area or height counts of the internal std. in the sample run
CF = Calibration factor of the parameter (internal standard method)

6.7.2.2 Non-Aqueous Samples:

A. Direct Purge (low level method)

Concentration (ppb, ng/g or ug/kg) = (Ap)(Mpi)/(CF)(Ws)

B. Methanolic Extraction (high level method)

Concentration (ppb, ng/g or ug/kg) =
(Ap)(Mpi)(DF)(Ve)/(Api)(CF)(Ws)(Va)

Where,

Ap, DF, Ve, Va, and Ws have the same definitions as above in Sections 15.1.2

Mpi = Mass of the internal std. purged (ng)
Api = Area or height counts of the internal std. in the sample run
CF = Calibration factor of the parameter (internal standard method)

6.8 Maintenance

6.8.1 Preventative Maintenance (Procedures):

6.8.1.1 Purge and Trap Device (Tekmar LSC-2 or LSC-2000):

Maintenance procedures for both models are found in the LSC-2000 User Manual.

1. Trap replacement - Section 6.1, 6.2, page 65 through 66
2. Sample lines cleaning - Section 6.3, page 67
3. Glassware cleaning - Section 6.4, page 67 through 68.
4. Leak testing - Section 3.3, page 16 through 17.

6.8.1.2 Gas Chromatograph

1. Injector port cleaning and septa replacement:
   A. Remove chromatographic column.
   B. Remove injector nut and septa.
   C. Clean injector nut and top of port with methanol.
   D. Squirt some methanol into port opening.
   E. Roll up a ham-wipe or kleenex lengthwise and douse with methanol. Insert through bottom of port and with a twisting motion, run the soaked rag all the way up to the top. Twist and pull the rag out.
   F. Put on a new septa and screw on the injector nut.

2. Detector port cleaning:

   A. Clean the detector port the same way as the injector. This will require the removal of the detector tower.

3. Electronics cleaning:

   A. Cool down the GC to room temperature.
   B. Turn off the power and unplug the instrument.
   C. Remove the top covers.
   D. Carefully slide out all the the baby printed circuit boards and lay them down on styrofoam pads.
   E. With a strong stream of nitrogen, thoroughly blow out the circuit board compartment and the mother board.
   F. Individually blow off all of the baby boards.
   G. Reinstall the boards and the covers.

4. Leak testing - Place a drop of leak detector solution on each joint and inspect for bubbles.

6.8.1.3 Hall Electroconductivity Detector (OI Model 4420):

Maintenance procedures are described in the OI instruction manual Part No. 165846

1. Reactor tube replacement:

   A. Section 39, page 39

2. Conductivity cell cleaning, union and transfer line replacement:

   A. Section 39, page 44

3. Solvent and resin replacement:

   A. Section 37, page 37 through 38

6.8.1.4 Photoionization Detector (OI Model 4430):

Maintenance procedures are found in the OI Instruction Manual. Part number 186678.

1. UV lamp cleaning - Section 15, page 16
2. UV lamp replacement - Section 15, page 15

Warning*** Do not disconnect column if conductivity solvent pump is on- this may destroy the PID.

6.8.1.4 Gas Purifiers and Particulate Filters:

1. Replacement - Torque nuts firmly, avoid stripping.

6.8.2 Operational Maintenance (Scheduling):

6.8.2.1 Routine Maintenance Schedule: Minimum requirements.

TABLE 4

| Instrument | Component # | Maximum Interval |
|---|---|---|
| Purge and Trap | 1,2,3,4 | 1 Month |
| Gas Chromatograph | 1,2 | 1 Month |
| | 3,4 | 6 Months |
| HECD | 1 | 1 Month |
| | 2,3 | 6 Months |
| PID | 1,3 | 1 Month |
| | 2 | 6 Months |
| Gas Purifiers | 1 | 1 Year |

6.8.2.2 Non-Routine Maintenance:

The following components are maintained on a "need only" basis. For some components, action is taken quite often, for others very rarely.

TABLE 5

| Instrument | Component # | Frequency |
|---|---|---|
| Purge and Trap | 1,2,3 | Often |
| Gas Chromatograph | N/A | N/A |
| HECD | N/A | N/A |
| PID | 4 | Rarely |
| | 5 | Seldom |
| Gas Purifiers | N/A | N/A |

6.8.3 Maintenance Notebook:

Maintenance schedules, receipts for service calls (parts and labor), and receipts for major replacement parts are kept in this notebook.
The in-house maintenance records are kept in this book. The information kept on each instrument includes:

- Instrument name
- Manufacturer
- Model
- Serial number
- Purchase date
- Date of maintenance
- Description of maintenance
- Initials and comments

## 7.0 Quality Control

### 7.1 Data Quality Objectives and Quality Control Specifications

NET's Quality Control objectives are to conduct analyses in a manner so that the data quality meets the user's requirements. Data quality objectives are measured in terms of being complete, precise, accurate, representative, and comparable.

NET Pacific, Inc. conducts specific activities in order to ensure that the data generated will be fit for use by the client and will meet NET's quality specifications. These activities and specifications define NET's Quality Control program. The goal is to produce analyses of known and acceptable quality.

Quality Control specifications are maintained for the following data quality indicators:

Analyses Quality Objectives:
1. Maintain calibration stability.
2. Ensure freedom from contamination.
3. Ensure accurate measurements.
4. Ensure precise measurements.
5. Control the analytical process within specifications.

Related Quality Control Activities:
1. Analyses of initial and continuing calibration check samples.
2. Analyses of laboratory (reagent) and/or field (travel or trip) blanks.
3. Analyses of matrix spikes (actual samples spiked with the analyte).
4. Analyses of duplicate samples in the form of matrix spike and matrix spike duplicates (MS and MSD's).
5. Analyses of surrogates and Laboratory Control Samples (method standards, SRM's or QC samples).

Because of the nature of the analytical process, not all methods require or can utilize all of the above Quality Control activities. However, some methods require additional QC tasks and have associated QC specifications. The Quality Control activities required by this SOP are derived from the sources listed and presented in the following sequence:

1. The State of California Environmental Laboratory Accreditation Program.
2. The NET National Quality Assurance Plan, January 10, 1990.
3. The NET Pacific Inc., Quality Assurance Plan , Oct. 1990.
4. The reference method(s) from which this SOP was derived.
5. Clients' Quality Assurance Project Plans.

In most cases, the requirements and specifications are presented as direct quotations from the source materials. Whenever differences occur, the most stringent requirements shall apply. The analyst is not permitted to modify the specifications or requirements. Any changes to this SOP that the analyst wishes to make should be accomplished through the process of a Request to Modify SOP submittal.

Clients may have specialized quality control or quality assurance requirements which are different than those specified in the SOP. In those cases, the analysts should refer to the specific Quality Assurance Project Plans which address the needs of that client. QAPP's are retained by the District Quality Assurance Coordinator. References to those documents should also be found in The Laboratory Information Management System - Lab Sys. Analysts should consult the appropriate QAPP's to ensure compliance with customer QA/QC requirements.

The analyst is advised that the ELAP, Corporate, NET Pacific and method requirements will be met and that the client's requirements will be treated as an adjunct or additional quality specification also to be met.

7.2 Calculations of Spike Recoveries:

Individual component recoveries of the MS and MSD will be calculated by the analyst using the following equation:

$$\text{MS or MSD Percent Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where SSR = Spiked Sample Results in concentration units.
SR = Sample Results in concentration units.
SA = Spike Added in concentration units.

7.3 Calculation of Relative Percent Difference (RPD)

Unless the method prohibits it, duplicate analyses will be made on spiked samples. These samples are called the matrix spike (MS) and the Matrix Spike Duplicate (MSD). The relative percent difference, or the RPD, between the two measurements for each analyte of interest is computed.

The RPD is computed as follows:

$$RPD = \frac{|D1 - D2|}{(D1 + D2)/2} \times 100$$

Where RPD = Relative Percent Difference between the MS and MSD values.
D1 = First sample value (MS) expressed in concentration units.
D2 = Duplicate sample value (MSD) expressed in concentration units.

The RPD is computed as an unsigned value. That is, the difference between D1 and D2 is an absolute value.

Percent recoveries of the MS and MSD should not be used for the computation. Actual values expressed in concentration units will be used.

7.4 Instrument Performance Criteria

External Calibration:

1. The r squared linear regression coefficient must be 0.995 or greater or the Relative Standard Deviation of the Response factors must be less than 15 percent.
2. The relative standard deviation of the retention time must be less than 2 percent.

Internal Calibration:

1. The minimum average response factor for the following five compounds must be meet.

| | |
|---|---|
| Chloromethane | 0.300 |
| 1,1 - Dichloroethane | 0.300 |
| Bromoform | 0.250 |
| 1,1,2,2 - Tetrachloroethane | 0.300 |
| Chlorobenzene | 0.300 |

7.5 Initial Calibration Checks for Method ____:

A second source standard containing all analytes of interest (ICVS) is analyzed after each five point calibration. The ICVS response for each compound must be within 70 to 130 percent of the true value for the calibration curve to be accepted.

## 7.6 Continuing Calibration Check Requirements for Method

A mid level standard prepared from the calibration standard concentrate is analyzed at the beginning of each day, after every 10 runs and at the end of each day. The percent recovery for each compound must be within 85 to 115 percent. If the CCVS recovery is not meet the analyst must:

1. Inspect equipment for probable cause and reanalyze the CCVS.
2. If the CCVS reanalysis is not acceptable then perform instrument maintenance and re-calibrate with a new five point curve.

Samples preceding a CCVS that does not meet the 85 to 115 percent recovery criteria do not require reanalysis as long as surrogate recovery criteria are meet. The data for samples not bracketed by two acceptable CCVS may be suspect. Flag these samples with a #CCVS for reporting purposes.

## 7.7 Daily Standard Retention Times:

The retention time for each parameter in the daily standard must be within three standard deviations of the average retention time determined in calibration (the retention time window). If the time lies outside the window, the instrumentation should be inspected for problems and corrected. If the time is still out, a new window must be established from a least three standard runs.

## 7.8 Daily Method Blank:

A method blank is analyzed each day after the first CCVS, or ICVS. A method blank must be run after the twentieth sample in a a day. To verify that carryover contamination is not a problem a method blank is analayzed following any high level sample or standard. This is accomplished by placing the method blank in the same autosampled position as the high sample to be analyzed the next time the autosampler cycles.
If contamination of the method blank is detected at a level above the reporting limit, the source of the contamination must be located and eliminated and a contamination free state demonstrated by the analysis of another blank before analysis of samples may proceed.

## 7.9 Method Requirements for Matrix Spikes

An Matrix Spike and Matrix Spike Duplicate set is performed at a matrix specific frequency of one set per 10 samples or one set per instrument per day whichever is more frequent. Percent Recovery is monitored for the following indicator compounds:

| | |
|---|---|
| 1,1-Dichloroethene | Benzene |
| Trichloroethene | Toluene |
| Chlorobenzene | |

Compound and matrix specific control limits for accuracy (percent recovery) are generated using the mean +/- 2 sigma (Warning Limits) and mean +/- 3 sigma (Control Limits) for thirty data points. If 30 points are not available, control limits are set to 50 to 150 percent recovery.

7.10 Method Requirements for Precision of the Analysis

Precession is demonstrated by calculating the relative percent difference concentrations measured in MS/MSD pairs. Matrix specific control limits are established for RPD from thirty data points. If thirty points are not available the control limit is set to less than 30 percent RPD.

7.11 Method Requirements for the Analysis of Surrogates

Surrogates are added to each standard, sample and QC sample analyzed. Control limits for percent recovery are generated from the method blank analysis. If thirty points are not available, control limits are set at 70 to 120 percent recovery.

7.12 QC Specifications, Acceptance Limits and QC Charts: (effective to 9-91)

Method Blanks < MDL
ICVS 70 to 130 percent recovery
CCVS 85 to 115 percent recovery
Water sample MS/MSD

| Compound | Percent Recovery | RPD |
|---|---|---|
| 1,1-Dichloroethene | 61-125 | <8 |
| Trichloroethene | 80-142 | <9 |
| Chlorobenzene | 73-119 | <8 |
| Benzene | 71-121 | <9 |
| Toluene | 79-127 | <8 |

Soil Sample MS/MSD

| Compound | Percent Recovery | RPD |
|---|---|---|
| 1,1-Dichloroethene | 70-120 | <30 |
| Trichloroethene | 70-120 | <30 |
| Chlorobenzene | 70-120 | <30 |
| Benzene | 70-120 | <30 |
| Toluene | 70-120 | <30 |

7.12.1 Use Of Quality Control Charts:

Quality Control charts will be utilized to distinguish indeterminate (random) variation from determinate and gross errors (assignable cause).

Quality Control charting has a much wider use, such as process capability. Those activities shall not be discussed here.

Data from Quality Control analyses will be utilized by the analyst in a real time mode to determine the acceptability of the analyses performed. Quality control sample analyses - spiked sample matrix, surrogate spike and spike sample duplicates will be the primary means by which data quality is determined. The analyst will evaluate ongoing or current QC data in a "real-time" mode to determine trends or short term changes in the recoveries of the performance parameters.

7.13 Out-of-Control Conditions:

If the analyses of the blanks, continuing calibration, check sample analyses, matrix spike, matrix spike duplicate, surrogates or laboratory control samples are beyond acceptance limits, corrective action must be taken immediately and documented.

7.13.1 Problem Identification, Resolution and Corrective Action in the Laboratory:

This section pertains to the identification of technical or procedural problems in the laboratory, and the resolution of those problems. Areas where problems may occur are sample receipt or log-in, sample preservation, instrument malfunctions, calibration or standards preparation, out-of-control QC sample analyses and sample preparation problems.

The objectives of the NET Pacific corrective action program are to:

1. Identify problems as they arise from whatever source.

2. Take the appropriate action to resolve the problem, both in the short-term and to effect permanent solutions.

3. Ensure that any analyses involved with a problem or Corrective Action is fit for use and met NET Pacific's Quality Assurance requirements prior to reporting.

4. Document the process that an actual problem was discovered, identified and corrected. The documentation should address what part of the process was out-of-control; when did the incident occur and when was it corrected; who discovered and connected the problem; what was the nature of the corrective action; what was done to prevent occurrence; and why did the incident happen.

7.13.2 Corrective Action Activities:

Activities which lead to the implementation of an effective corrective action are:

1. Problem recognition or detection of out-of-control situations.

2. Decide a priori who will investigate the problem.

3. Investigate the problem and determine its cause.

4. Determine a solution or corrective action, both for the purpose of effecting an immediate solution and a permanent solution.

5. Fully implement the corrective action.

6. Verify that corrective action has resulted in a long-term to the problem.

7. Document the completed process.

7.13.3 Closed-Loop Corrective Action System:

Experience has shown that most problems will not be prevented from reoccurring in the future until positive action has been taken. The significant characteristic of an effective corrective system is the step that closes the loop-the determination to make a change if the system demands it.

Corrective actions should be a continual part of the laboratory system for quality, and they should be formally documented. Corrective action is not complete until it is demonstrated that the action has effectively and permanently corrected the problem. Diligent follow-up is probably the most important requirement of a successful corrective action system.

7.14 Corrective Actions For This SOP, Out-Of-Control Conditions

7.14.1 Calibrations: Initial and Continuing

Out-of-control conditions exist whenever the calibration of the measurement system cannot be verified initially or subsequently. The verification of calibration systems is typically conducted through the analyses of quality control samples or standards prepared from alternate source materials. Reference materials or standards from the NIST or U.S. EPA may also be used. Commercial vendors also provide materials called ICVS or CCVS (initial or continuing calibration verification samples). The point is this: that the calibration is done is not sufficient. The acceptability of the calibration must be demonstrated and documented.

Specifications vary from method to method, but generally the requirement is that the analyses of known standards or reference materials must meet the 95 percent confidence interval published for those substances. Some methods have specific requirements for the type, kind and concentration of analytes to be measured for calibration verifications. Additionally, performance specifications expressed as percent recoveries of true values are often included. Those method requirements should be followed.

Whenever ICVS or CCVS performance specifications cannot be met, the calibration is out-of-control. Work must proceed no further on the analysis of actual samples until control of the process is restored.

A recommended course of action to follow whenever ICVS or CCVS out-of-control conditions exist is:

1. Reanalyse the ICVS or CCVS sample. If acceptable data is generated, proceed with the normal analyses sequence. Document the problem and its resolution on the Exception/Corrective Action report.

2. If reanalysis of the original ICVS or CCVS is not successful, consider preparing and analysing a new ICVS or CCVS. If successful, complete the QC Exception report and proceed normally.

3. If the ICCV or CCVS cannot be analyzed within specifications, the problem may lie either with the standards, the calibration curve (or response factor) or the instrument performance.

Consider preparing one or more fresh calibration standard solutions if one or more data points appear questionable. Prepare an entire set if warranted. Evaluate whether new standard source material should be acquired and utilized. Evaluate balances and volumetric apparatus for accuracy/precision problems. Evaluate storage systems: refrigeration, containers, photosensitive reactions, biodegradation, etc. Evaluate age and use history of source materials.

Try refitting calibration curves or response factors by dropping questionable points. Replot curves and recompute ICVS or CCVS data. If improvements occur, marginal or unacceptable data points may be the root of calibration problems.

Reanalyze the ICVS or CCVS and determine if specifications are met. If so, complete the QC Exception report and proceed with sample analyses.

4. If the preparation of new calibration standards, new ICVS or CCVS solutions and/or new curve fitting techniques do not help, the problem is likely instrumental in nature. Consult the maintenance log for indication of similar past problems.

Solutions to instrumental problems go beyond the scope of this section. The instrument manufacturers trouble shooting and maintenance section should be consulted. Consult with laboratory supervisor personnel and instrument service representatives. Record any trouble shooting and results of those activities in the instrument log book.

Recalibrate after instrument trouble shooting. Analyze the ICVS and CCVS as required.

7.14.2 Blanks: Process, Field, Travel and Trip:

An Out-of-Control condition exists whenever the level of the analyte(s) of interest are found in the blank(s) at a level which exceeds the analyte MDL.

For conventional analysis, analytes contaminating the blank become a serious problem whenever they reach the reporting interval. They start to become a problem whenever they are found in excess of the MDL. So corrective action should be taken whenever blanks are found in excess of the MDL's's.

For some methods, it is considered acceptable practice to subtract the level of analytes found in blanks from the actual sample analyte level. For example, it is considered acceptable to subtract blanks in the gravimetric oil and grease method. However, unless the method specifically provides for blank subtraction, levels of contamination should be reported, but not corrected in the sample.

Whenever blanks are found to contain the analyte or interest, the analyst must determine the source and level of the contaminate. Reagents, glassware, bench tops, lab coats and apparatus are all potential sources of contamination.

The role of the analyst is to ensure that reagents, solvents, and acids are sufficiently pure so as not to introduce the analyte of interest through contamination. Labware should be sufficiently cleaned so the possibility of contamination is acceptably low.
Data that has unacceptable associated blanks must not be reported.

7.14.3 Recoveries of Matrix Spikes, Matrix Spike Duplicates, Surrogates and Laboratory Control Samples:

Analytical processes are determined as being in control, in part, by comparing recoveries of the matrix spike (MS) and the matrix spike duplicate (MSD) with acceptance (performance) standards. The method performance specifications may be expressed as a fixed per cent recovery, as in the CLP methodology. Acceptable recoveries may also be determined through statistical process control, the preferred method.

Recoveries of the MS or MSD analytes which fall outside the acceptance levels may be caused by problems with the sample preparation steps: digestion, extraction, gas-sparing and extract clean-up. Or, with the analyses finish: loss of calibration, instrument drift, auto injector problems, calculations, etc. However, in some cases, poor recoveries of the MS/MSD are caused by the sample matrix itself. The matrix may interfere with the process that is used to isolate the analyte from the sample. The remaining sample matrix, if not completely separated from the analyte, may interfere with the instrumental finish of the analysis. An oil sample diluted for PCB analysis may sequester or quench an ECD if the oil level is too high. Collectively, the effects are called matrix interference effects.

If the recoveries of the MS and MSD are both comparatively high or low, one may presume that the difficulties were not caused by some gross error to one, but not the other spiked sample. Conversely, error could be suspected if the recovery was high or low for one spike, but not the other. In those cases, limited information may be gained about whether the analytical process was or is actually in control.

Because the recoveries of the MS and MSD may be effected by the analytical process and the sample matrix, an approach to identifying poor recoveries caused by matrix effects is through the analysis of surrogate analytes and Laboratory Control Samples (LCS's).

With many organic analytical procedures, the use of surrogate compounds is required or recommended. Surrogates act on behalf of the actual analytes and are expected to suffer the same consequences of loss, interference or other deleterious effects. Surrogate spikes are especially helpful and different from analyte spikes in that surrogates are "spiked" into blanks, calibration standards, actual samples and the MS/MSD's. This allows one the mechanism to compare and relate the results of recoveries of the analytes to spikes in samples with the surrogate recoveries in the blank samples and CCVS's.

For example, out-of-control (OOC) recoveries of the MS/MSD, but not the surrogate in blanks, could suggest matrix effects. OOC recoveries for the MS/MSD's and surrogates in the sample, but not the blanks, would strongly support matrix interference problems.

Poor surrogate recoveries in the blank would strongly suggest process problems, either in the sample preparation or analysis stage. Since a blank represents the most desirable sample matrix for analysis, it is considered unusual that the surrogate in the blank would not meet specifications. This example would probably require reanalysis or reprepparation of the sample and reanalysis.

Additional information about process control can be gained through the analysis of Laboratory Control Samples (LCS's). LCS's represent a constant, and in the case of "method" standards, a relatively simple matrix. LCS's such as NIST Standard Reference Materials (SRM's) will have a "real" matrix from which to judge both the matrix and process effects. Repetitive analysis of the LCS is recommended because a historical data base can be accumulated. From this, the process acceptance limits can be determined for that particular matrix - soil, fish tissue, filter papers, petroleum products, etc.

If the MS and MSD are OOC but the LCS is in control, the sample itself from which the MS and MSD were prepared should be suspected. Caution should be exercised about rejecting an entire batch of sample analyses because the matrix will vary from one sample to the next. That is, sample analysis within the batch may be fully acceptable but the MS or MSD within the batch may give poor recoveries. Evaluate the surrogate and LCS recoveries to determine the process matrix problems.

7.15 Data Validation (analyst and Supervisory Review)

7.15.1 Sample Holding Time Requirement:

All samples must be analyzed within 14 days of collection. Date of the analysis shall be recorded by the analyst on the benchsheet, verified by the supervisor, and conformity to the holding time requirements checked by the group leader.

7.15.2 Analyte Confirmation Techniques:

Analyte confirmation is achieved with a second chromatographic column (dissimilar to the first) or by GC/MS. The results are recorded on the analysis benchsheet as a positive or negative confirmation.

7.15.3 Sample Preservation Verification:

7.15.3.1 Aqueous Samples:

At the time of analysis, the analyst shall perform a quick pH, chlorine, and temperature check using pH paper, potassium iodine paper, and the thermometer reading of the sample refrigerator, respectively. The results will be recorded on the analysis benchsheet along with headspace observations. Conformity to preservation requirements will be checked by the group leader.

7.15.3.2 Non-Aqueous Samples:

At the time of extraction (high level method) or analysis (low level method), the analyst shall record the thermometer reading of the sample refrigerator on the preparation or analysis benchsheet. Conformity to preservation requirements will be checked by the group leader.

## 7.16 Data Reporting and Management

### 7.16.1 Data Reporting:

#### 7.16.1.1 Analytical Report (bench level):

Following data reduction, the analyst shall:

1. Record the parameter concentrations found in the sample or spike on the analysis benchsheet. The units of concentration shall be clearly specified. Numeric values will consist of three significant figures with the exception of reporting values past the decimal place of the reporting limit.

2. Transcribe sample results from the analysis benchsheet to the "LAB SYS" computer system.

3. Submit the analysis benchsheet to the supervisor for technical review and corrections.

#### 7.16.1.2 Analytical Report (intermediate level):

The supervisor shall submit the preparation and Analysis package to the group leader for overall review. If all the criteria are met (surrogate and duplicate spike recoveries, RPD's, holding times, preservations, etc.), the group leader approves the release of the results on the "LAB SYS" and formed into a presentable analytical report for submission to the client. The laboratory director reviews this final report, and if acceptable signs and dates it, signifying release to the client.

#### 7.16.1.3 Analytical report (final level):

Approved analytical results are retrieved from "LAB SYS" and formed into a presentable analytical report for submission to the client. The laboratory director reviews this final report, and if acceptable signs and dates it, signifying release to the client.

### 7.16.2 Data Management:

All records, raw data, and reports are, at minimum, stored in had copy form for three years. Nearly all of this information is stored on magnetic media (for quick access) for a minimum of one year.

## 7.17 Method Detection Limits:

See Apendix B.
The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.
MDL's are calculated in accordance with 40 CFR, Part 136, Appendix B, Revision 1.11.

The deviation of a MDL over time is instrument dependent. As long as instrumentation is careful maintained and thus, instrument sensitivities maintained, the MDL should remain fairly stable. To verify this, a standard at the MDL concentration should be analysed three times every three months.

7.18 Waste Handling and Disposal

7.18.1 Waste Handling:

All types of waste shall be properly contained and stored awaiting disposal. Leak proof, acid, base, and organic solvent resistant containers shall be used. The containers shall be properly marked so as to indicate their contents. Flammable, acidic, basic, and corrosive wastes shall be stored separately from each other and in a safe manner.

The types of waste encountered by the use of this SOP are, but not limited to:

- Methanol extracts
- Analyzed and unanalyzed sample remains
- Expired standards, neat and solutions

7.18.2 Waste Disposal

Disposal regulations are highly localized. Physical storage, waste volumes, and storage times shall be determined and adhered to by the laboratory director.

8.0 Reference Citations (categorised by sample matrix)

8.1 Wastewater:

"Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater" USEPA, July 1988.

8.1.1 Preparation and Analysis:

Method 601 "Purgeable Halocarbons" and Method 602 "Purgeable Aromatics."

8.2 Groundwater, Soil, Solid and Liquid Waste:

"Test Methods for Evaluating Solid Waste." USEPA, November 1986. SW-846 Third Edition, Volume 1B.

8.2.1 Groundwater:

Method 5030, Section 1.0 through 7.2, and 7.3.1

8.2.2 Water Miscible Liquid Waste:

Method 5030, Section 1.0 through 7.2, and 7.3.2

8.2.3 Soil, Solid and Non-Water Miscible Liquid Waste:

Method 5030, Section 1.0 through 7.2, and 7.3.3.1 (low level method) or 7.3.3.2 (high level method).

8.4 Other References

1. Tekmar Company, Purge and Trap analysis of Priority Pollutants in Water According to EPA Method 601, TEK/DATA 8032181.

# METHOD 8010

## HALOGENATED VOLATILE ORGANICS

### 1.0 SCOPE AND APPLICATION

1.1 Method 8010 is used to determine the concentration of various volatile halogenated organic compounds. Table 1 indicates compounds that may be analyzed by this method and lists the method detection limit for each compound in reagent water. Table 2 lists the practical quantitation limit for other matrices.

### 2.0 SUMMARY OF METHOD

2.1 Method 8010 provides gas chromatographic conditions for the detection of halogenated volatile organic compounds. Samples can be analyzed using direct injection or purge-and-trap (Method 5030). Ground water samples must be analyzed using Method 5030. A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved by a halogen-specific detector (HSD).

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the analytes from interferences that may occur and for analyte confirmation.

### 3.0 INTERFERENCES

3.1 Refer to Method 5030 and 8000.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field sample blank prepared from reagent water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

### 4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 Gas Chromatograph - Analytical system complete with gas chromatograph suitable for on-column injections or purge-and-trap sample introduction and all required accessories, including detector, analytical columns, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended.

4.1.2 Columns

4.1.2.1 Column 1 - 8 ft x 0.1 in i.d. stainless steel or glass column packed with 1% SP-1000 on Carbopack-B 60/80 mesh or equivalent.

4.1.2.2 Column 2 - 6 ft x 0.1 in i.d. stainless steel or glass column packed with chemically bonded n-octane on Porasil-C 100/120 mesh (Durapak) or equivalent.

4.1.3 Detector - Electrolytic conductivity (HSD).

4.2 Sample introduction apparatus - Refer to Method 5030 for the appropriate equipment for sample introduction purposes.

4.3 Syringes - 5-mL Luerlok glass hypodermic and a 5-mL, gas-tight with shutoff valve.

4.4 Volumetric flasks - 10-, 50-, 100-, 500-, and 1,000-mL with a ground-glass stopper.

4.5 Microsyringes - 10-, 25-uL with a 0.006 in i.d. needle (Hamilton 702N or equivalent) and a 100-uL.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 ASTM Type II Water (ASTM D1193-77 (1983)). All references to water in the method refer to ASTM Type II unless otherwise specified.

5.3 Stock standards - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standards in methanol using assayed liquids or gases, as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood.

5.3.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

5.3.2 Add the assayed reference material, as described below.

5.3.2.1 Liquids - Using a 100-uL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.3.2.2 Gases - To prepare standards for any compounds that boil below 30°C (e.g. bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas rapidly dissolves in the methanol. This may also be accomplished by using a lecture bottle equipped with a Hamilton Lecture Bottle Septum (#86600). Attach Teflon tubing to the side-arm relief valve and direct a gentle stream of gas into the methanol meniscus.

5.3.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter (ug/uL) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.3.4 Transfer the stock standard solution into a bottle with a Teflon lined screw-cap. Store, with minimal headspace, at -10°C to -20°C and protect from light.

5.3.5 Prepare fresh standards every 2 months for gases of for reactive compounds such as 2-chloroethylvinyl ether. All other standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

5.4 Secondary dilution standards - Using stock standard solutions, prepare in methanol secondary dilution standards, as needed, that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Step 5.5 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace for volatiles and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.5 Calibration standards - Calibration standards at a minimum of five concentration levels are prepared in water from the secondary dilution of the stock standards. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Each standard should contain each analyte for detection by this method (e.g. some or all of the compounds listed in Table 1 may be included). In order to prepare accurate aqueous standard solutions, the following precautions must be observed.

5.5.1 Do not inject more than 20 uL of alcoholic standards into 100 mL of water.

5.5.2 Use a 25-uL Hamilton 702N microsyringe or equivalent (variations in needle geometry will adversely affect the ability to deliver reproducible volumes of methanolic standards into water).

5.5.3 Rapidly inject the alcoholic standard into the filled volumetric flask. Remove the needle as fast as possible after injection.

5.5.4 Mix aqueous standards by inverting the flask three times only.

5.5.5 Fill the sample syringe from the standard solution contained in the expanded area of the flask (do not use any solution contained in the neck of the flask).

5.5.6 Never use pipets to dilute or transfer samples or aqueous standards.

5.5.7 Aqueous standards are not stable and should be discarded after 1 hour, unless properly sealed and stored. The aqueous standards can be stored up to 24 hours, if held in sealed vials with zero headspace.

5.6 Internal standards (if internal standard calibration is used) - To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compounds recommended for use as surrogate spikes (Step 5.7) have been used successfully as an internal standards, because of their generally unique retention times.

5.6.1 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest as described in Step 5.5.

5.6.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Steps 5.3 and 5.4. It is recommended that the secondary dilution standard be prepared at a concentration of 15 ug/mL of each internal standard compound. The addition of 10 uL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 ug/L.

5.6.3 Analyze each calibration standard according to Section 7.0, adding 10 uL of internal standard spiking solution directly to the syringe.

5.7 Surrogate standards - The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate halocarbons. A combination of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane is recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Step 5.3, add a volume to give 750 ug of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix, and dilute to volume for a concentration of 15 ng/uL. Add 10 uL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. If the internal standard calibration procedure is used, the surrogate compounds may be added directly to the internal standard spiking solution (Step 5.6.2).

5.8 Methanol, $CH_3OH$. Pesticide quality or equivalent. Store away from other solvents.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1.

## 7.0 PROCEDURE

7.1 Volatile compounds are introduced into the gas chromatograph either by direct injection or purge-and-trap (Method 5030). Method 5030 may be used directly on ground water samples or low-level contaminated soils and sediments. For medium-level soils or sediments, methanolic extraction, as described in Method 5030, may be necessary prior to purge-and-trap analysis.

7.2 Gas chromatography conditions (Recommended)

7.2.1 Column 1 - Set helium gas flow at 40 mL/min flow rate. Set column temperature at 45°C for 3 minutes; then program an 8°C/min temperature rise to 220°C and hold for 15 minutes.

7.2.2 Column 2 - Set helium gas flow at 40 mL/min flow rate. Set column temperature at 50°C for 3 minutes; then program a 6°C/min temperature rise to 170°C and hold for 4 minutes.

7.3 Calibration - Refer to Method 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.

7.3.1 Calibration must take place using the same sample introduction method that will be used to analyze actual samples (see Step 7.4.1).

7.3.2 The procedure for internal or external calibration may be used. Refer to Method 8000 for a description of each of these procedures.

7.4 Gas chromatographic analysis

7.4.1 Introduce volatile compounds into the gas chromatograph using either Method 5030 (purge-and-trap method) or the direct injection method (see Step 7.4.1.1). If the internal standard calibration technique is used, add 10 uL of internal standard to the sample prior to purging.

7.4.1.1 Direct injection - In very limited applications (e.g. aqueous process wastes), direct injection of the sample into the GC system with a 10 uL syringe may be appropriate. The detection limit is very high (approximately 10,000 ug/L); therefore, it is only permitted when concentrations in excess of 10,000 ug/L are expected or for water-soluble compounds that do not purge. The system must be calibrated by direct injection (bypassing the purge-and-trap device).

7.4.2 Follow Step 7.6 of Method 8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence.

7.4.3 Table 1 summarizes the estimated retention times on the two columns for a number of organic compounds analyzable by this method. An example of the separation achieved by Column 1 is shown in Figure 1.

7.4.4 Record the sample volume purged or injected and the resulting peak sizes (in area units or peak heights).

7.4.5 Calculation of concentration is covered in Step 7.8 of Method 8000.

7.4.6 If analytical interferences are suspected, or for the purpose of confirmation, analysis using the second GC column is recommended.

7.4.7 If the response for a peak is off-scale, prepare a dilution of the sample with reagent water. The dilution must be performed on a second aliquot of the sample which has been properly sealed and stored prior to use.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 8000 for gas chromatographic procedures. Quality control to ensure the proper operation of the purge-and-trap device is covered in Method 5030.

8.2 Mandatory quality control to validate the GC system operation is found in Method 8000, Step 8.6.

8.2.1 The quality control check sample concentrate (Method 8000, Step 8.6) should contain each parameter of interest at a concentration of 10 ug/mL in methanol.

8.2.2 Table 3 indicates the calibration and QC acceptance criteria for this method. Table 4 gives method accuracy and precision as functions of concentration for the analytes of interest. The contents of both Tables should be used to evaluate a laboratory's ability to perform and generate acceptable data by this method.

8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if recovery is within limits (limits established by performing QC procedure outlined in Method 8000, Step 8.10).

8.3.1 If recovery is not within limits, the following is required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

## 9.0 METHOD PERFORMANCE

9.1 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 8.0-500 ug/L. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 4.

9.2 The accuracy and precision obtained will be determined by the sample matrix, sample introduction technique, and by the calibration procedure used.

## 10.0 REFERENCES

1. Bellar, T.A., and J.J. Lichtenberg, J. Amer. Water Works Assoc., 66(12), pp. 739-744, 1974.

2. Bellar, T.A., and J.J. Lichtenberg, Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds, in Van Hall (ed.), Measurement of Organic Pollutants in Water and Wastewater, ASTM STP 686, pp. 108-129, 1979.

3. Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters. Category 11 - Purgeables and Category 12 - Acrolein, Acrylonitrile, and Dichlorodifluoromethane. Report for EPA Contract 68-03-2635 (in preparation).

4. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

5. Provost, L.P., and R.S. Elder, "Interpretation of Percent Recovery Data," American Laboratory, 15, pp. 58-63, 1983.

6. "EPA Method Validation Study 23, Method 601 (Purgeable Halocarbons)," Report for EPA Contract 68-03-2856 (in preparation).

TABLE 1.
CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS FOR HALOGENATED VOLATILE ORGANICS

| Compound | Retention time (min) | | Method detection limit[a] (ug/L) |
|---|---|---|---|
| | Col. 1 | Col. 2 | |
| Benzyl chloride[b] | | | |
| Bis(2-chloroethoxy)methane[c] | | | |
| Bromobenzene | | | |
| Bromodichloromethane | 13.7 | 14.6 | 0.10 |
| Bromoform | 19.2 | 19.2 | 0.20 |
| Bromomethane | | | |
| Carbon tetrachloride | 13.0 | 14.4 | 0.12 |
| Chlorobenzene | 24.2 | 18.8 | 0.25 |
| Chloroethane | 3.33 | 8.68 | 0.52 |
| 2-Chloroethyl vinyl ether | 18.0 | | 0.13 |
| Chloroform | 10.7 | 12.1 | 0.05 |
| 1-Chlorohexane | | | |
| Chloromethane | 1.50 | 5.28 | 0.08 |
| Chloromethylmethyl ether[c] | | | |
| Chlorotoluene[b] | | | |
| Dibromochloromethane | 16.5 | 16.6 | 0.09 |
| Dibromomethane | | | |
| 1,2-Dichlorobenzene | 34.9 | 23.5 | 0.15 |
| 1,3-Dichlorobenzene | 34.0 | 22.4 | 0.32 |
| 1,4-Dichlorobenzene | 35.4 | 22.3 | 0.24 |
| Dichlorodifluoromethane[b] | | | |
| 1,1-Dichloroethane | 9.30 | 12.6 | 0.07 |
| 1,2-Dichloroethane | 11.4 | 15.4 | 0.03 |
| 1,1-Dichloroethylene | 8.0 | 7.72 | 0.13 |
| trans-1,2-Dichloroethylene | 10.1 | 9.38 | 0.10 |
| Dichloromethane | | | |
| 1,2-Dichloropropane | 14.9 | 16.6 | 0.04 |
| trans-1,3-Dichloropropylene | 15.2 | 16.6 | 0.34 |
| 1,1,2,2-Tetrachloroethane | 21.6 | | 0.03 |
| 1,1,1,2-Tetrachloroethane | | | |
| Tetrachloroethylene | 21.7 | 15.0 | 0.03 |
| 1,1,1-Trichloroethane | 12.6 | 13.1 | 0.03 |
| 1,1,2-Trichloroethane | 16.5 | 18.1 | 0.02 |
| Trichloroethylene | 15.8 | 13.1 | 0.12 |
| Trichlorofluoromethane | 7.18 | | |
| Trichloropropane | | | |
| Vinyl chloride | 2.67 | 5.28 | 0.18 |

[a] Using purge-and-trap method (Method 5030).
[b] Demonstrated very erratic results when tested by purge-and-trap.
[c] Demonstrated poor purging efficiency.

TABLE 2.
DETERMINATION OF PRACTICAL QUANTITATION LIMITS
(PQL) FOR VARIOUS MATRICES[a]

| Matrix | Factor[b] |
|---|---|
| Ground water | 10 |
| Low-level soil | 10 |
| Water miscible liquid waste | 500 |
| High-level soil and sludge | 1250 |
| Non-water miscible waste | 1250 |

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

[b]PQL = [Method detection limit (Table 1)] X [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet-weight basis.

TABLE 3.
CALIBRATION AND QC ACCEPTANCE CRITERIA[a]

| Parameter | Range for Q (ug/L) | Limit for s (ug/L) | Range for x (ug/L) | Range P, $P_s$ (%) |
|---|---|---|---|---|
| Bromodichloromethane | 15.2-24.8 | 4.3 | 10.7-32.0 | 42-172 |
| Bromoform | 14.7-25.3 | 4.7 | 5.0-29.3 | 13-159 |
| Bromomethane | 11.7-28.3 | 7.6 | 3.4-24.5 | D-144 |
| Carbon tetrachloride | 13.7-26.3 | 5.6 | 11.8-25.3 | 43-143 |
| Chlorobenzene | 14.4-25.6 | 5.0 | 10.2-27.4 | 38-150 |
| Chloroethane | 15.4-24.6 | 4.4 | 11.3-25.2 | 46-137 |
| 2-Chloroethylvinyl ether | 12.0-28.0 | 8.3 | 4.5-35.5 | 14-186 |
| Chloroform | 15.0-25.0 | 4.5 | 12.4-24.0 | 49-133 |
| Chloromethane | 11.9-28.1 | 7.4 | D-34.9 | D-193 |
| Dibromochloromethane | 13.1-26.9 | 6.3 | 7.9-35.1 | 24-191 |
| 1,2-Dichlorobenzene | 14.0-26.0 | 5.5 | 1.7-38.9 | D-208 |
| 1,3-Dichlorobenzene | 9.9-30.1 | 9.1 | 6.2-32.6 | 7-187 |
| 1,4-Dichlorobenzene | 13.9-26.1 | 5.5 | 11.5-25.5 | 42-143 |
| 1,1-Dichloroethane | 16.8-23.2 | 3.2 | 11.2-24.6 | 47-132 |
| 1,2-Dichloroethane | 14.3-25.7 | 5.2 | 13.0-26.5 | 51-147 |
| 1,1-Dichloroethene | 12.6-27.4 | 6.6 | 10.2-27.3 | 28-167 |
| trans-1,2-Dichloroethene | 12.8-27.2 | 6.4 | 11.4-27.1 | 38-155 |
| 1,2-Dichloropropane | 14.8-25.2 | 5.2 | 10.1-29.9 | 44-156 |
| cis-1,3-Dichloropropene | 12.8-27.2 | 7.3 | 6.2-33.8 | 22-178 |
| trans-1,3-Dichloropropene | 12.8-27.2 | 7.3 | 6.2-33.8 | 22-178 |
| Methylene chloride | 15.5-24.5 | 4.0 | 7.0-27.6 | 25-162 |
| 1,1,2,2-Tetrachloroethane | 9.8-30.2 | 9.2 | 6.6-31.8 | 8-184 |
| Tetrachloroethene | 14.0-26.0 | 5.4 | 8.1-29.6 | 26-162 |
| 1,1,1-Trichloroethane | 14.2-25.8 | 4.9 | 10.8-24.8 | 41-138 |
| 1,1,2-Trichloroethane | 15.7-24.3 | 3.9 | 9.6-25.4 | 39-136 |
| Trichloroethene | 15.4-24.6 | 4.2 | 9.2-26.6 | 35-146 |
| Trichlorofluoromethane | 13.3-26.7 | 6.0 | 7.4-28.1 | 21-156 |
| Vinyl chloride | 13.7-26.3 | 5.7 | 8.2-29.9 | 28-163 |

Q = Concentration measured in QC check sample, in ug/L.

s = Standard deviation of four recovery measurements, in ug/L.

x = Average recovery for four recovery measurements, in ug/L.

P, $P_s$ = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria are from 40 CFR Part 136 for Method 601 and were calculated assuming a QC check sample concentration of 20 ug/L.

TABLE 4.
METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

| Parameter | Accuracy, as recovery, x' (ug/L) | Single analyst precision, $s_r'$ (ug/L) | Overall precision, S' (ug/L) |
|---|---|---|---|
| Bromodichloromethane | 1.12C-1.02 | 0.11x+0.04 | 0.20x+1.00 |
| Bromoform | 0.96C-2.05 | 0.12x+0.58 | 0.21x+2.41 |
| Bromomethane | 0.76C-1.27 | 0.28x+0.27 | 0.36x+0.94 |
| Carbon tetrachloride | 0.98C-1.04 | 0.15x+0.38 | 0.20x+0.39 |
| Chlorobenzene | 1.00C-1.23 | 0.15x-0.02 | 0.18x+1.21 |
| Chloroethane | 0.99C-1.53 | 0.14x-0.13 | 0.17x+0.63 |
| 2-Chloroethylvinyl ether[b] | 1.00C | 0.20x | 0.35x |
| Chloroform | 0.93C-0.39 | 0.13x+0.15 | 0.19x-0.02 |
| Chloromethane | 0.77C+0.18 | 0.28x-0.31 | 0.52x+1.31 |
| Dibromochloromethane | 0.94C+2.72 | 0.11x+1.10 | 0.24x+1.68 |
| 1,2-Dichlorobenzene | 0.93C+1.70 | 0.20x+0.97 | 0.13x+6.13 |
| 1,3-Dichlorobenzene | 0.95C+0.43 | 0.14x+2.33 | 0.26x+2.34 |
| 1,4-Dichlorobenzene | 0.93C-0.09 | 0.15x+0.29 | 0.20x+0.41 |
| 1,1-Dichloroethane | 0.95C-1.08 | 0.08x+0.17 | 0.14x+0.94 |
| 1,2-Dichloroethane | 1.04C-1.06 | 0.11x+0.70 | 0.15x+0.94 |
| 1,1-Dichloroethene | 0.98C-0.87 | 0.21x-0.23 | 0.29x-0.04 |
| trans-1,2-Dichloroethene | 0.97C-0.16 | 0.11x+1.46 | 0.17x+1.46 |
| 1,2-Dichloropropane[b] | 1.00C | 0.13x | 0.23x |
| cis-1,3-Dichloropropene[b] | 1.00C | 0.18x | 0.32x |
| trans-1,3-Dichloropropene[b] | 1.00C | 0.18x | 0.32x |
| Methylene chloride | 0.91C-0.93 | 0.11x+0.33 | 0.21x+1.43 |
| 1,1,2,2-Tetrachloroethane | 0.95C+0.19 | 0.14x+2.41 | 0.23x+2.79 |
| Tetrachloroethene | 0.94C+0.06 | 0.14x+0.38 | 0.18x+2.21 |
| 1,1,1-Trichloroethane | 0.90C-0.16 | 0.15x+0.04 | 0.20x+0.37 |
| 1,1,2-Trichloroethane | 0.86C+0.30 | 0.13x-0.14 | 0.19x+0.67 |
| Trichloroethene | 0.87C+0.48 | 0.13x-0.03 | 0.23x+0.30 |
| Trichlorofluoromethane | 0.89C-0.07 | 0.15x+0.67 | 0.26x+0.91 |
| Vinyl chloride | 0.97C-0.36 | 0.13x+0.65 | 0.27x+0.40 |

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in ug/L.

$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration of x, in ug/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of x, in ug/L.

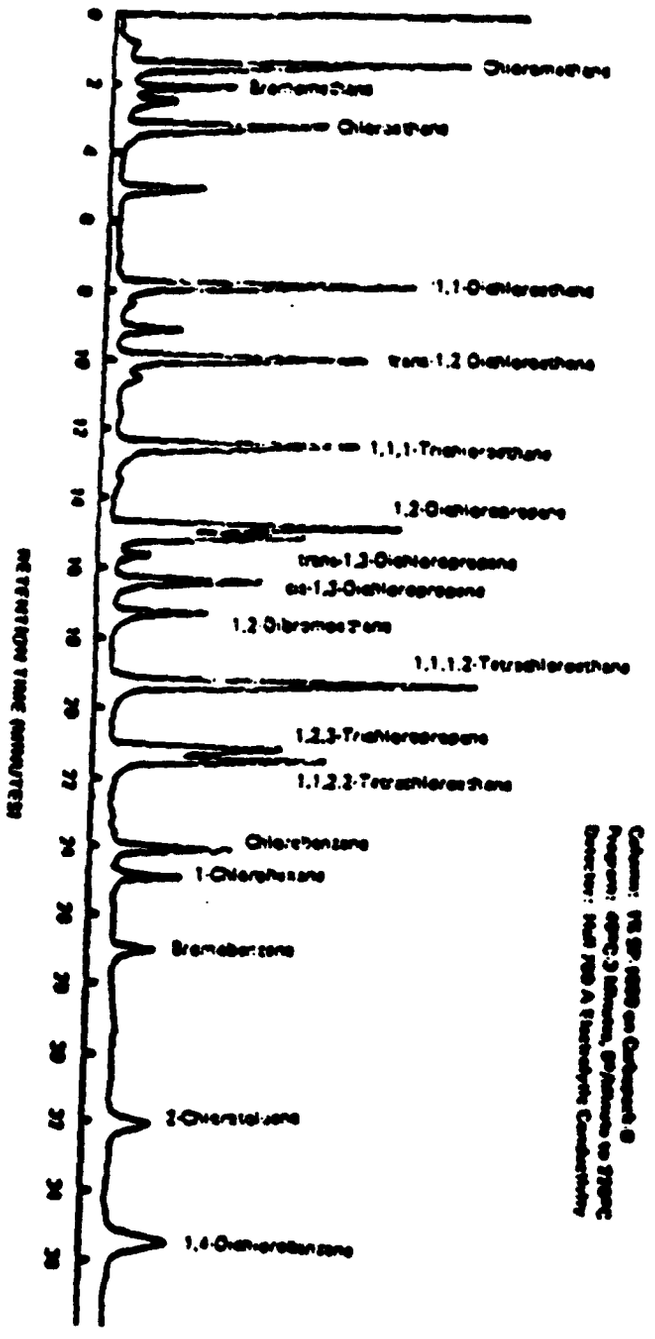
C = True value for the concentration, in ug/L.

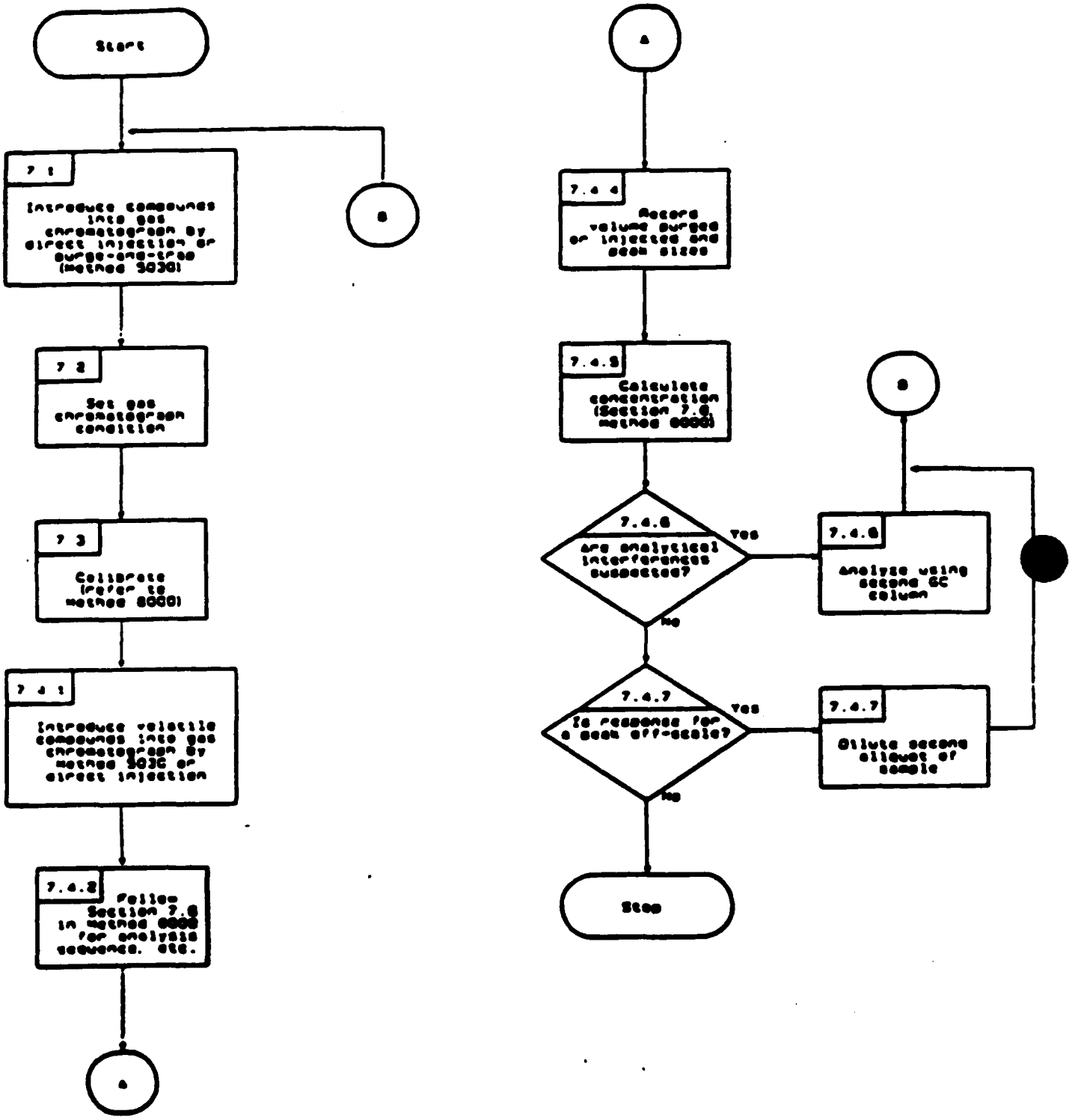
x = Average recovery found for measurements of samples containing a concentration of C, in ug/L.

[a]From 40 CFR Part 136 for Method 601.

[b]Estimates based upon the performance in a single laboratory.

FIGURE 1.
GAS CHROMATOGRAM OF HALOGENATED VOLATILE ORGANICS




Start
7.1
Introduce compounds into gas chromatograph by direct injection or purge-and-trap (method 3030)
7.2
Set gas chromatograph condition
7.3
Calibrate (refer to method 8000)
7.4.1
Introduce volatile compounds into gas chromatograph by method 3030 or direct injection
7.4.2
Follow Section 7.6 in method 8000 for analysis sequence, etc.
A
A
7.4.4
Record volume purged or injected and peak sizes
7.4.5
Calculate concentration (Section 7.6 method 8000)
7.4.6
Are analytical interferences suspected?
Yes
No
B
7.4.8
Analyze using second GC column
7.4.7
Is response for a peak off-scale?
Yes
No
7.4.9
Dilute second aliquot of sample
Stop

# METHOD 8020

## AROMATIC VOLATILE ORGANICS

### 1.0 SCOPE AND APPLICATION

1.1 Method 8020 is used to determine the concentration of various aromatic volatile organic compounds. Table 1 indicates compounds which may be determined by this method and lists the method detection limit for each compound in reagent water. Table 2 lists the practical quantitation limit (PQL) for other matrices.

### 2.0 SUMMARY OF METHOD

2.1 Method 8020 provides chromatographic conditions for the detection of aromatic volatile compounds. Samples can be analyzed using direct injection or purge-and-trap (Method 5030). Ground water samples must be determined using Method 5030. A temperature program is used in the gas chromatograph to separate the organic compounds. Detection is achieved by a photo-ionization detector (PID).

2.2 If interferences are encountered, the method provides an optional gas chromatographic column that may be helpful in resolving the analytes from the interferences and for analyte confirmation.

### 3.0 INTERFERENCES

3.1 Refer to Method 5030 and 8000.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field sample blank prepared from reagent water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

### 4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph:

4.1.1 Gas Chromatograph: Analytical system complete with gas chromatograph suitable for on-column injections or purge-and-trap sample introduction and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended.

TABLE 1. CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS FOR AROMATIC VOLATILE ORGANICS

| Compound | Retention time (min) | | Method detection limit[a] (ug/L) |
|---|---|---|---|
| | Col. 1 | Col. 2 | |
| Benzene | 3.33 | 2.75 | 0.2 |
| Chlorobenzene | 9.17 | 8.02 | 0.2 |
| 1,4-Dichlorobenzene | 16.8 | 16.2 | 0.3 |
| 1,3-Dichlorobenzene | 18.2 | 15.0 | 0.4 |
| 1,2-Dichlorobenzene | 25.9 | 19.4 | 0.4 |
| Ethyl Benzene | 8.25 | 6.25 | 0.2 |
| Toluene | 5.75 | 4.25 | 0.2 |
| Xylenes | | | |

[a] Using purge-and-trap method (Method 5030).

TABLE 2. DETERMINATION OF PRACTICAL QUANTITATION LIMITS (PQL) FOR VARIOUS MATRICES[a]

| Matrix | Factor[b] |
|---|---|
| Ground water | 10 |
| Low-level soil | 10 |
| Water miscible liquid waste | 500 |
| High-level soil and sludge | 1250 |
| Non-water miscible waste | 1250 |

[a]Sample-PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

[b]PQL = [Method detection limit (Table 1)] X [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet-weight basis.

4.1.2 Columns:

4.1.2.1 Column 1: 6-ft x 0.082-in I.D. #304 stainless steel or glass column packed with 5% SP-1200 and 1.75% Bentone-34 on 100/120 mesh Supelcort or equivalent.

4.1.2.2 Column 2: 8-ft x 0.1-in I.D. stainless steel or glass column packed with 5% 1,2,3-Tris(2-cyanoethoxy)propane on 60/80 mesh Chromosorb W-AW or equivalent.

4.1.3 Detector: Photoionization (PID) (h-Nu Systems, Inc. Model PI-51-02 or equivalent).

4.2 Sample introduction apparatus: Refer to Method 5030 for the appropriate equipment for sample introduction purposes.

4.3 Syringes: A 5-mL Luerlok glass hypodermic and a 5-mL, gas-tight with shutoff valve.

4.4 Volumetric flask: 10-, 50-, 100-, 500-, and 1,000-mL with a ground-glass stopper.

4.5 Microsyringe: 10- and 25-uL with a 0.006-in I.D. needle (Hamilton 702N or equivalent) and a 100-uL.

## 5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as a water in which an interferent is not observed at the method detection limit (MDL) of the parameters of interest.

5.2 Stock standards: Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standards in methanol using assayed liquids. Because of the toxicity of benzene and 1,4-dichlorobenzene, primary dilutions of these materials should be prepared in a hood.

5.2.1 Place about 9.8 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

5.2.2 Using a 100-uL syringe, immediately add two or more drops of assayed reference material to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

5.2.3 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter (ug/uL) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction

to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

5.2.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at 4°C and protect from light.

5.2.5 All standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

5.3 Secondary dilution standards: Using stock standard solutions, prepare in methanol secondary dilution standards, as needed, that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Paragraph 5.4 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace for volatiles and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.4 Calibration standards: Calibration standards at a minimum of five concentration levels are prepared in reagent water from the secondary dilution of the stock standards. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Each standard should contain each analyte for detection by this method (e.g., some or all of the compounds listed in Table 1 may be included). In order to prepare accurate aqueous standard solutions, the following precautions must be observed.

5.4.1 Do not inject more than 20 uL of alcoholic standards into 100 mL of reagent water.

5.4.2 Use a 25-uL Hamilton 702N microsyringe or equivalent (variations in needle geometry will adversely affect the ability to deliver reproducible volumes of methanolic standards into water).

5.4.3 Rapidly inject the alcoholic standard into the filled volumetric flask. Remove the needle as fast as possible after injection.

5.4.4 Mix aqueous standards by inverting the flask three times only.

5.4.5 Fill the sample syringe from the standard solution contained in the expanded area of the flask (do not use any solution contained in the neck of the flask).

5.4.6 Never use pipets to dilute or transfer samples or aqueous standards.

5.4.7 Aqueous standards are not stable and should be discarded after 1 hr, unless properly sealed and stored. The aqueous standards can be stored up to 24 hr, if held in sealed vials with zero headspace.

5.5 Internal standards (if internal standard calibration is used): To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compound, alpha,alpha,alpha-trifluorotoluene recommended for use as a surrogate spiking compound (Paragraph 5.6) has been used successfully as an internal standards.

5.5.1 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest as described in Section 5.4.

5.5.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 5.2 and 5.3. It is recommended that the secondary dilution standard be prepared at a concentration of 15 ug/mL of each internal standard compound. The addition of 10 uL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 ug/L.

5.5.3 Analyze each calibration standard according to Section 7.0, adding 10 uL of internal standard spiking solution directly to the syringe.

5.6 Surrogate standards: The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate compounds (e.g, alpha,alpha,alpha-trifluorotoluene) recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Section 5.2, add a volume to give 750 ug of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix, and dilute to volume for a concentration of 15 ng/uL. Add 10 uL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. If the internal standard calibration procedure is used, the surrogate compounds may be added directly to the internal standard spiking solution (Paragraph 5.5.2).

5.7 Methanol: pesticide quality or equivalent. Store away from other solvents.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1.

## 7.0 PROCEDURE

7.1 Volatile compounds are introduced into the gas chromatograph either by direct injection or purge-and-trap (Method 5030). Method 5030 may be used directly on ground water samples or low-level contaminated soils and sediments. For medium-level soils or sediments, methanolic extraction, as described in Method 5030, may be necessary prior to purge-and-trap analysis.

7.2 Gas chromatography conditions (Recommended):

7.2.1 Column 1: Set helium gas flow at 36 mL/min flow rate. The temperature program sequences are as follows: For lower boiling compounds, operate at 50°C isothermal for 2 min; then program at 6°C/min to 90°C and hold until all compounds have eluted. For higher boiling range of compounds, operate at 50°C isothermal for 2 min; then program at 3°C/min to 110°C and hold until all compounds have eluted. Column 1 provides outstanding separations for a wide variety of aromatic hydrocarbons. Column 1 should be used as the primary analytical column because of its unique ability to resolve para-, meta-, and ortho-aromatic isomers.

7.2.2 Column 2: Set helium gas flow at 30 mL/min flow rate. The temperature program sequence is as follows: 40°C isothermal for 2 min; then 2°C/min to 100°C and hold until all compounds have eluted. Column 2, an extremely high-polarity column, has been used for a number of years to resolve aromatic hydrocarbons from alkanes in complex samples. However, because resolution between some of the aromatics is not as efficient as with Column 1, Column 2 should be used as a confirmatory column.

7.3 Calibration: Refer to Method 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.

7.3.1 Calibration must take place using the same sample introduction method that will be used to analyze actual samples (see Section 7.4.1).

7.3.2 The procedure for internal or external calibration may be used. Refer to Method 8000 for a description of each of these procedures.

7.4 Gas chromatographic analysis:

7.4.1 Introduce volatile compounds into the gas chromatograph using either Method 5030 (purge-and-trap method) or the direct injection method. If the internal standard calibration technique is used, add 10 uL of internal standard to the sample prior to purging.

7.4.1.1 Direct injection: In very limited applications (e.g., aqueous process wastes), direct injection of the sample into the GC system with a 10 uL syringe may be appropriate. The detection limit is very high (approximately 10,000 ug/L); therefore, it is only permitted when concentrations in excess of 10,000 ug/L are expected or for water-soluble compounds that do not purge. The system must be calibrated by direct injection (bypassing the purge-and-trap device).

7.4.2 Follow Section 7.6 of Method 8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence.

7.4.3 Table 1 summarizes the estimated retention times and detection limits for a number of organic compounds analyzable using this method. An example of the separation achieved by Column 1 is shown in Figure 1. Figure 2 shows an example of the separation achieved using Column 2.

7.4.4 Record the sample volume purged or injected and the resulting peak sizes (in area units or peak heights).

7.4.5 Calculation of concentration is covered in Section 7.8 of Method 8000.

7.4.6 If analytical interferences are suspected, or for the purpose of confirmation, analysis using the second GC column is recommended.

7.4.7 If the response for a peak is off-scale, prepare a dilution of the sample with reagent water. The dilution must be performed on a second aliquot of the sample which has been properly sealed and stored prior to use.

## 8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures and Method 8000 for gas chromatographic procedures. Quality control to ensure the proper operation of the purge-and-trap device is covered in Method 5030.

8.2 Mandatory quality control to validate the GC system operation is found in Method 8000, Section 8.6.

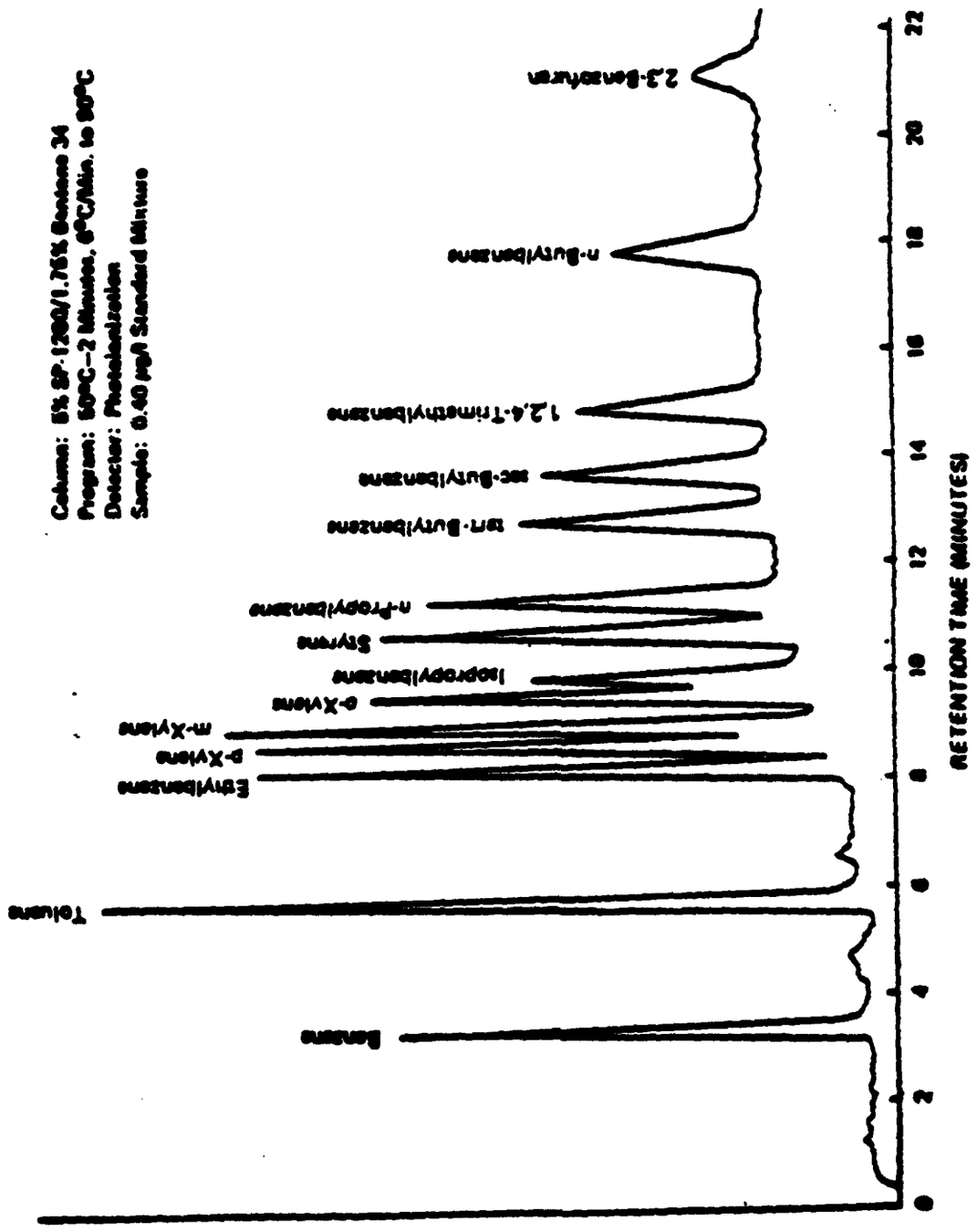



Figure 1. Chromatogram of aromatic volatile organics (column 1 conditions).

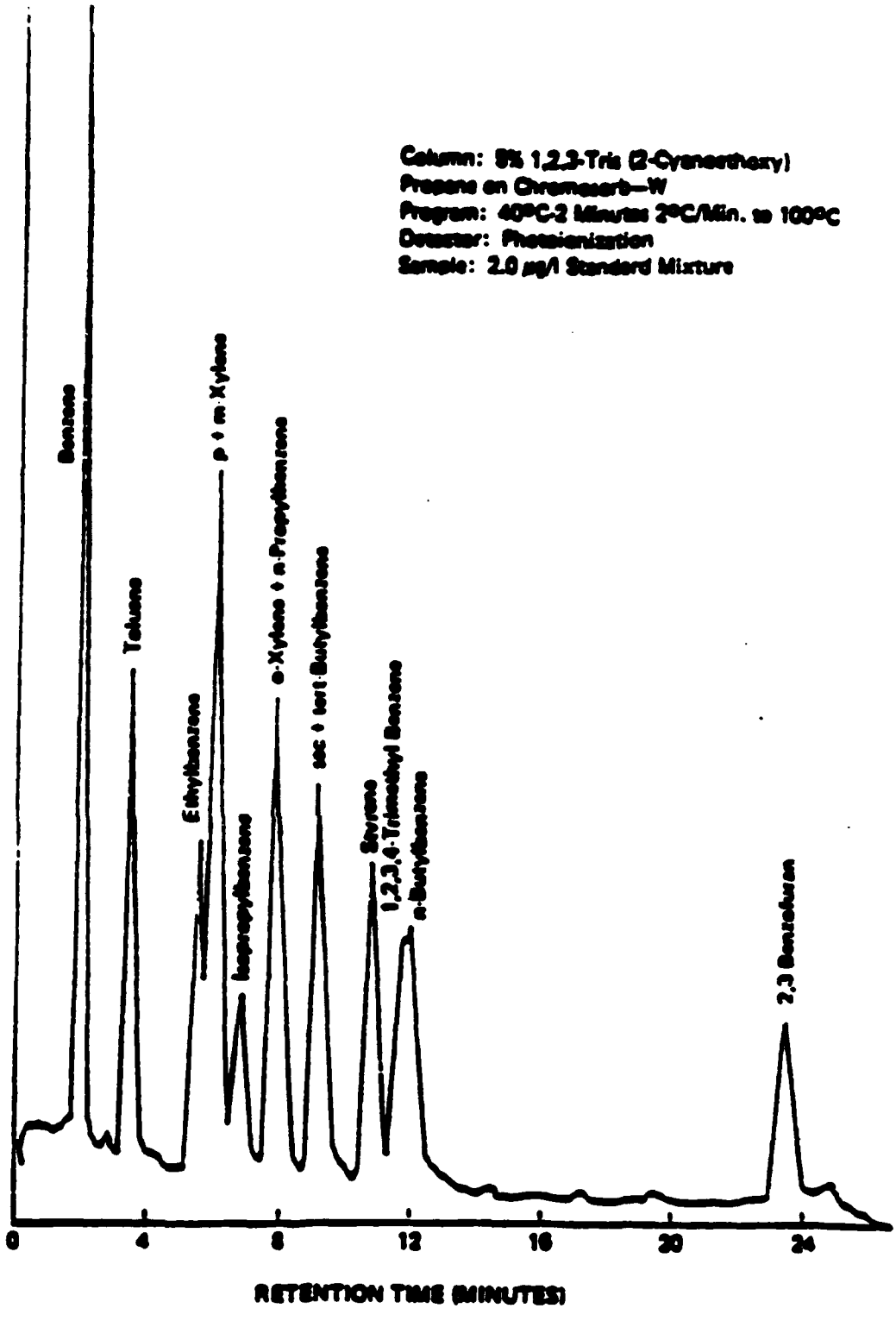



Figure 2. Chromatogram of aromatic volatile organics (column 2 conditions).

8.2.1 The quality control check sample concentrate (Method 8000, Section 8.6) should contain each parameter of interest at a concentration of 10 ug/mL in methanol.

8.2.2 Table 3 indicates the calibration and QC acceptance criteria for this method. Table 4 gives method accuracy and precision as functions of concentration for the analytes of interest. The contents of both Tables should be used to evaluate a laboratory's ability to perform and generate acceptable data by this method.

8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if recovery is within limits (limits established by performing QC procedure outlined in Method 8000, Section 8.10).

8.3.1 If recovery is not within limits, the following is required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

## 9.0 METHOD PERFORMANCE

9.1 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 2.1-500 ug/L. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 4.

9.2 The accuracy and precision obtained will be determined by the sample matrix, sample introduction technique, and by the calibration procedure used.

## 10.0 REFERENCES

1. Bellar, T.A., and J.J. Lichtenberg, J. Amer. Water Works Assoc., 66(12), pp. 739-744, 1974.

2. Bellar, T.A., and J.J. Lichtenberg, Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds, in Van Hall (ed.), Measurement of Organic Pollutants in Water and Wastewater, ASTM STP 686, pp. 108-129, 1979.

3. Dowty, B.J., S.R. Antoine, and J.L. Laseter, "Quantitative and Qualitative Analysis of Purgeable Organics by High Resolution Gas Chromatography and Flame Ionization Detection," in Van Hall, ed., Measurement of Organic Pollutants in Water and Wastewater. ASTM STP 686, pp. 24-35, 1979.

4. Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters. Category 11 - Purgeables and Category 12 - Acrolein, Acrylonitrile, and Dichlorodifluoromethane. Report for EPA Contract 68-03-2635 (in preparation).

5. "EPA Method Validation Study 24, Method 602 (Purgeable Aromatics)," Report for EPA Contract 68-03-2856 (in preparation).

6. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

7. Provost, L.P., and R.S. Elder, "Interpretation of Percent Recovery Data," American Laboratory, 15, pp. 58-63, 1983.

TABLE 3. CALIBRATION AND QC ACCEPTANCE CRITERIA[a]

| Parameter | Range for Q (ug/L) | Limit for s (ug/L) | Range for $\bar{X}$ (ug/L) | Range P, $P_s$ (%) |
|---|---|---|---|---|
| Benzene | 15.4-24.6 | 4.1 | 10.0-27.9 | 39-150 |
| Chlorobenzene | 16.1-23.9 | 3.5 | 12.7-25.4 | 55-135 |
| 1,2-Dichlorobenzene | 13.6-26.4 | 5.8 | 10.6-27.6 | 37-154 |
| 1,3-Dichlorobenzene | 14.5-25.5 | 5.0 | 12.8-25.5 | 50-141 |
| 1,4-Dichlorobenzene | 13.9-26.1 | 5.5 | 11.6-25.5 | 42-143 |
| Ethylbenzene | 12.6-27.4 | 6.7 | 10.0-28.2 | 32-160 |
| Toluene | 15.5-24.5 | 4.0 | 11.2-27.7 | 46-148 |

Q = Concentration measured in QC check sample, in ug/L.

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, $P_s$ = Percent recovery measured.

[a]Criteria are from 40 CFR Part 136 for Method 602 and were calculated assuming a QC check sample concentration of 20 ug/L. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 1.

TABLE 4. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION

| Parameter | Accuracy, as recovery, x' (ug/L) | Single analyst precision, $s_r'$ (ug/L) | Overall precision, S' (ug/L) |
|---|---|---|---|
| Benzene | 0.92C+0.57 | 0.09X+0.59 | 0.21X+0.56 |
| Chlorobenzene | 0.95C+0.02 | 0.09X+0.23 | 0.17X+0.10 |
| 1,2-Dichlorobenzene | 0.93C+0.52 | 0.17X-0.04 | 0.22X+0.53 |
| 1,3-Dichlorobenzene | 0.96C-0.04 | 0.15X-0.10 | 0.19X+0.09 |
| 1,4-Dichlorobenzene | 0.93C-0.09 | 0.15X+0.28 | 0.20X+0.41 |
| Ethylbenzene | 0.94C+0.31 | 0.17X+0.46 | 0.26X+0.23 |
| Toluene | 0.94C+0.65 | 0.09X+0.48 | 0.18X-0.71 |

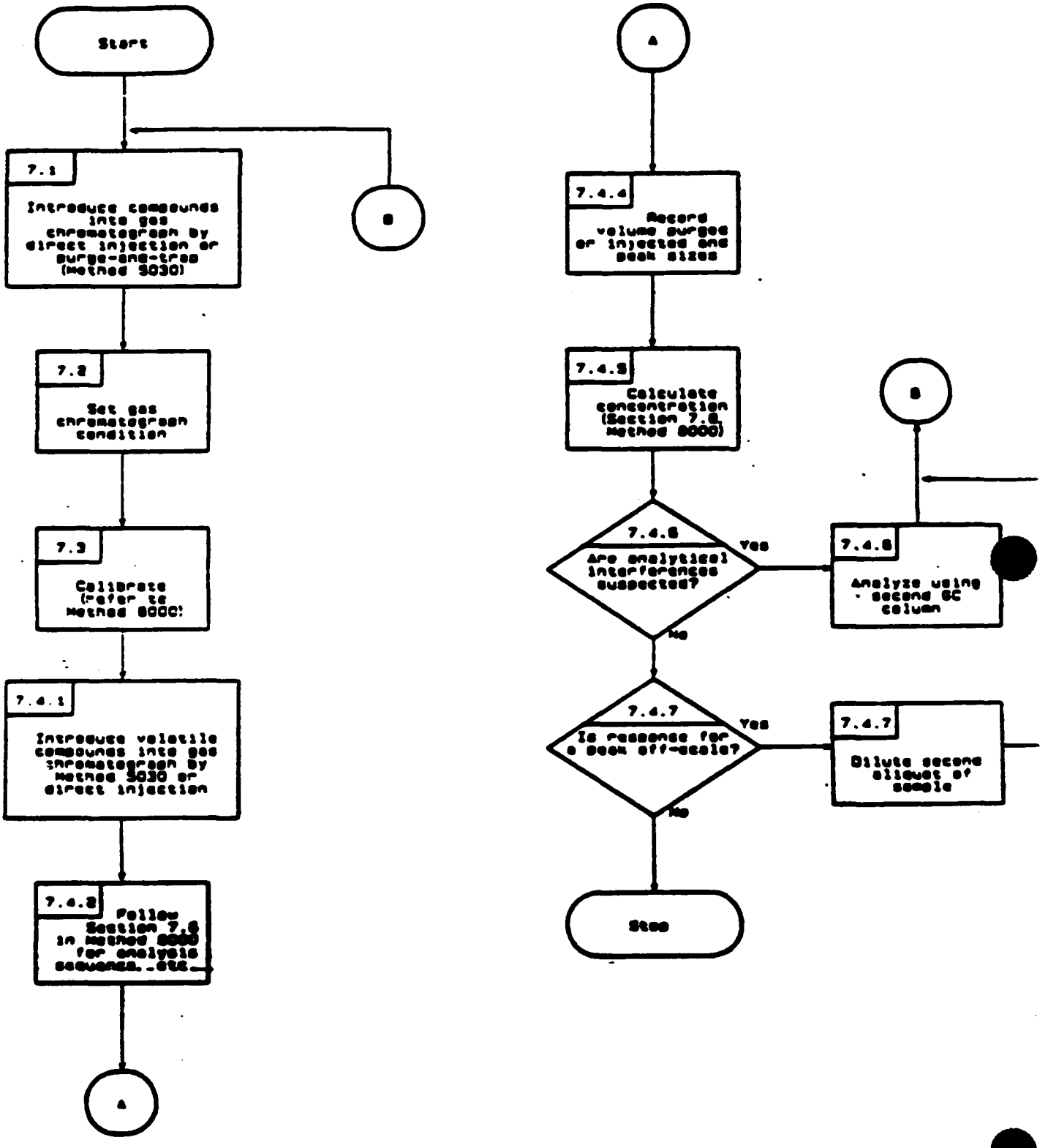
x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in ug/L.

$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration of X, in ug/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of X, in ug/L.

C = True value for the concentration, in ug/L.

X = Average recovery found for measurements of samples containing a concentration of C, in ug/L.


Start
7.1
Introduce compounds into gas chromatograph by direct injection or purge-and-trap (Method 5030)
B
7.2
Set gas chromatograph condition
7.3
Calibrate (refer to Method 8000)
7.4.1
Introduce volatile compounds into gas chromatograph by Method 5030 or direct injection
7.4.2
Follow Section 7.6 in Method 8000 for analysis sequence, etc.
A
A
7.4.4
Record volume purged or injected and peak sizes
7.4.5
Calculate concentration (Section 7.6, Method 8000)
B
7.4.6
Are analytical interferences suspected?
Yes
7.4.8
Analyze using second GC column
No
7.4.7
Is response for a peak off-scale?
Yes
7.4.7
Dilute second aliquot of sample
No
Stop

APPENDIX B

## MDL STUDY

FIELD SERVICES DIVISION: Mobile Laboratory #3

| Analyte: | CLM/VC | ClE/BrM | C13FlM | 1,1-DCE | DCM |
|---|---|---|---|---|---|
| Method Reference: | 8010 | 8010 | 8010 | 8010 | 8010 |
| Instrument: | HP 5890 | | | | |
| Analyst: | DFM | | | | |
| Date: | 6/17/91 | | | | |
| Spk Matrix: | Water | | | | |
| Spk Conc: | 5ng/5ml | | | | |
| Result 1: | 12 | 15 | 6.3 | 3.6 | 8.7 |
| Result 2: | 9.6 | 12 | 5.5 | 4.2 | 11 |
| Result 3: | 9.7 | 11 | 4.4 | 2.6 | 8.8 |
| Result 4: | 6.7 | 8.2 | 3.4 | 3.3 | 6.8 |
| Result 5: | 6.6 | 8.2 | 3.3 | 3.3 | 6.8 |
| Result 6: | 6.0 | 7.6 | 3.0 | 2.9 | 6.4 |
| Result 7: | 5.1 | 6.6 | 2.5 | 1.7 | 2.5 |
| Mean: | 8.0 | 9.8 | 4.1 | 3.1 | 7.3 |
| Std Dev: | 2.3 | 2.8 | 1.3 | 0.72 | 2.4 |
| Signal / Noise Ratio | 3.5 | 3.5 | 3.2 | 4.3 | 3.0 |
| MDL: | 1.4 | 1.8 | 0.82 | 0.45 | 1.5 |
| RL: | 3.0 | 4.0 | 2.0 | 1.0 | 3.0 |

(2)

## MDL STUDY

FIELD SERVICES DIVISION: Mobile Laboratory #3

| Analyte: | t-1,2-DCE | 1,1-DCA | CHCl3 | 1,1,1-TCA | CCl4 |
|---|---|---|---|---|---|
| Method Reference: | 8010 | | | | |
| Instrument: | HP 5890 | | | | |
| Analyst: | DFM | | | | |
| Date: | 6/17/91 | | | | |
| Spk Matrix: | Water | | | | |
| Spk Conc: | 5ng/5ml | | | | |
| Result 1: | 5.7 | 4.4 | 6.3 | 8.4 | 4.8 |
| Result 2: | 5.7 | 4.2 | 2.8 | 7.8 | 4.8 |
| Result 3: | 4.3 | 3.4 | 2.4 | 6.3 | 3.7 |
| Result 4: | 3.0 | 2.1 | 1.6 | 4.9 | 2.6 |
| Result 5: | 2.7 | 2.2 | 1.6 | 4.6 | 2.4 |
| Result 6: | 2.7 | 2.0 | 1.4 | 4.3 | 2.2 |
| Result 7: | 2.0 | 1.6 | 1.3 | 3.7 | 2.2 |
| Mean: | 3.7 | 2.8 | 2.5 | 5.7 | 3.3 |
| Std Dev: | 1.4 | 1.1 | 1.6 | 1.7 | 1.1 |
| Signal / Noise Ratio | 2.6 | 2.5 | 1.7 | 3.3 | 3.0 |
| MDL: | 0.88 | 0.69 | | 1.1 | 0.69 |
| RL: | 2.0 | 1.5 | | 2.5 | 1.5 |

(2)

## MDL STUDY

FIELD SERVICES DIVISION: Mobile Laboratory #3

| Analyte: | 1,2-DCA | TCE | 1,2-DCP | CHBrCl2 | c-1,3-DCP |
|---|---|---|---|---|---|
| Method Reference: | 8010 | | | | |
| Instrument: | HP 5890 | | | | |
| Analyst: | DFM | | | | |
| Date: | 6/17/91 | | | | |
| Spk Matrix: | Water | | | | |
| Spk Conc: | 5ng/5ml | | | | |
| Result 1: | 4.3 | 4.6 | 4.8 | 3.5 | 5.7 |
| Result 2: | 4.5 | 5.0 | 5.1 | 3.6 | 5.1 |
| Result 3: | 3.6 | 4.1 | 3.0 | 2.4 | 4.0 |
| Result 4: | 2.4 | 2.8 | 2.5 | 1.8 | 2.4 |
| Result 5: | 2.4 | 2.7 | 2.5 | 1.6 | 2.3 |
| Result 6: | 2.1 | 2.3 | 2.4 | 1.4 | 2.1 |
| Result 7: | 1.9 | 2.1 | 2.1 | 1.3 | 1.9 |
| Mean: | 3.0 | 3.4 | 3.2 | 2.2 | 3.4 |
| Std Dev: | 1.0 | 1.1 | 1.1 | 0.91 | 1.4 |
| Signal / Noise Ratio | 3.0 | 3.1 | 2.9 | 2.4 | 2.4 |
| MDL: | 0.63 | 0.69 | 0.69 | 0.57 | 0.88 |
| RL: | 1.5 | 1.5 | 1.5 | 1.5 | 2.0 |

(4)

# MDL STUDY

FIELD SERVICES DIVISION: Mobile Laboratory #3

| Analyte: | PCE | ClB | 1,1,2,2TCA | 1,3-DCB | 1,4-DCB |
|---|---|---|---|---|---|
| Method Reference: | 8010 | | | | |
| Instrument: | HP 5890 | | | | |
| Analyst: | DFM | | | | |
| Date: | 6/17/91 | | | | |
| Spk Matrix: | Water | | | | |
| Spk Conc: | 5ng/5ml | | | | |
| Result 1: | 6.2 | 1.6 | 9.9 | 1.0 | 4.5 |
| Result 2: | 5.9 | 2.0 | 8.1 | 0.99 | 4.1 |
| Result 3: | 4.8 | 0.0 | 5.2 | 0.86 | 3.2 |
| Result 4: | 3.2 | 0.0 | 4.1 | 0.0 | 2.5 |
| Result 5: | 3.1 | 0.81 | 0.0 | 0.0 | 1.9 |
| Result 6: | 2.8 | 0.0 | 3.5 | 0.0 | 1.8 |
| Result 7: | 2.4 | 3.1 | 14 | 1.8 | 1.6 |
| Mean: | 4.1 | 1.1 | 6.4 | 0.66 | 2.8 |
| Std Dev: | 1.5 | 1.1 | 4.3 | 0.63 | 1.1 |
| Signal / Noise Ratio | 2.7 | 1.0 | 1.5 | 1.0 | 2.5 |
| MDL: | 0.94 | | | | 0.69 |
| RL: | 2.0 | | | | 1.5 |

MDL STUDY

FIELD SERVICES DIVISION: Mobile Laboratory #3

| | | | | | |
|---|---|---|---|---|---|
| Analyte: | 1,2-DCB | | | | |
| Method Reference: | 8010 | | | | |
| Instrument: | HP 5890 | | | | |
| Analyst: | DFM | | | | |
| Date: | 6/17/91 | | | | |
| Spk Matrix: | Water | | | | |
| Spk Conc: | 5ng/5ml | | | | |
| Result 1: | 2.6 | | | | |
| Result 2: | 2.8 | | | | |
| Result 3: | 1.3 | | | | |
| Result 4: | 0.81 | | | | |
| Result 5: | 0.80 | | | | |
| Result 6: | 0.72 | | | | |
| Result 7: | 0.71 | | | | |
| Mean: | 1.4 | | | | |
| Std. Dev. | 0.86 | | | | |
| Signal / Noise Ratio | 1.2 | | | | |
| MDL: | | | | | |
| RL: | | | | | |

# RADIAN SOP FOR ON-SITE SOIL GAS ANALYSIS FOR THE McCLELLAN OU B RI, OCTOBER 1991 THROUGH MARCH 1992

## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the protocol for analyzing soil gas samples on site using a HP-5890 II gas chromatograph (GC) with capillary column and photoionization detector/electron capture detector (PID/ECD).

## 2.0 EQUIPMENT

- Hewlett Packard 5890 series II GC (2) with PID and ECD detectors.
- Hewlett Packard 3396 integrator (2).
- Glass 50 cc syringe with 1-½ inch S.S. luer lok needle and interchangeable ground glass barrel, Popper & Sons.
- Pressure Lok A-2 gas tight syringe, 250 μL (2), Alltech & Associates, 050031.
- Mininert Teflon® on/off valve with luer lok fitting, Alltech & Associates, 654051.
- DB 624 capillary column, 30 m x 0.53 mm (2), Curtis Matheson Scient., 256-434.
- Syringe cleaning oven capable of maintaining 160°C ±5°.

## 3.0 CERTIFIED GAS STANDARDS

- Aromatic hydrocarbon gas standard containing 100 ppmv each of benzene, toluene, o-xylene, and p-xylene in UHP nitrogen, 140 cubic feet, Scott Specialty Gas.

- Chlorinated hydrocarbon gas standard containing 2 ppmv each of cis-1,2-dichloroethene, trans-1,2-dichloroethene, chloroform, 1,1,1-trichloroethane, trichloroethene, and tetrachloroethene in UHP nitrogen, 140 cubic feet, Scott Specialty Gas.

- A Scott Specialty Gas single component gas standard containing 10 ppmv of vinyl chloride in UHP $N_2$.

- Working standards are prepared from the Scott Specialty Gas standards in low pressure 15 L stainless steel canisters. The working standards are prepared by Radian Austin's QA group by dilution with UHP $N_2$. The halogenated hydrocarbons are 100 ppbv in the canister standard. Vinyl chloride and aromatic hydrocarbons are 500 ppbv in the canister standard.

## 4.0 GC OPERATING CONDITIONS

Operating parameters on the HP5890 PID/ECD are as follows:

- Injection temp: 150°C

- Detector temp: PID/175°C; ECD/300°C

- Column oven temp:

  PID Initial temperature 35°C, for 12 minutes.
  Temperature ramp rate 15°C/minute
  Final temperature 75°C, hold 5 minutes

  ECD 35°C isothermal

- Carrier gas flow: $N_2$, 10 ml/minute

- Makeup gas flow: $N_2$, ECD/40 ml/minute

## 5.0 CALIBRATION

### 5.1 Initial Calibration

- The HP5890 PID and ECD will be calibrated by injecting three different volumes of a canister gas standard containing the following:

| Compound | Approximate Concentration (ppbv) | Instrument Detector Used | Approximate Calibration Range (ppbv) |
|---|---|---|---|
| Vinyl chloride | 500 | PID | 100-500 |
| cis-1,2-DCE | 100 | PID | 20-100 |
| trans-1,2-DCE | 100 | PID | 20-100 |
| Freon 113 | 100 | ECD | 20-100 |
| Freon 12 | 100 | ECD | 20-100 |
| Carbon tetrachloride | 100 | ECD | 20-100 |
| Chloroform | 100 | ECD | 20-100 |
| 1,1,1-TCA | 100 | ECD | 20-100 |
| TCE | 100 | ECD | 20-100 |
| PCE | 100 | ECD | 20-100 |
| Benzene | 500 | PID | 100-500 |
| Toluene | 500 | PID | 100-500 |
| μ-Xylene | 500 | PID | 100-500 |
| o-Xylene | 500 | PID | 100-500 |
| UHP Nitrogen | Balance | --- | --- |

The standard volumes used for the analytes quantitated by the PID are 1.0 mLs, 0.5 mLs, and 0.2 mLs. The standard volumes used for the analytes quantitated by the ECD are 250 μl, 100 μl, and 50 μl.

- A retention time (RT) for each calibrated peak is entered into the integrator used for that compound/detector.

- An RT window of four percent is entered into the integrator for identification of calibrated compounds in the field samples.

- An average response factor is calculated from the three standards described above.

- A relative standard deviation of less than or equal to 30% must be achieved for each analyte or the calibration is repeated.

- The average response factor is entered into the integrator for each compound if the calibration criteria has been met.

**5.2 Daily Calibration and QC Check**

- The daily calibration check standard is analyzed at the beginning of every analysis day that the multi-point calibration is not performed and after the system has been altered or adjusted.

- The calibration check standard is a mixture of the analytes of interest (approximately 40 ppbv of halogenated hydrocarbons and 250 ppbv of aromatic hydrocarbons and vinyl chloride).

- The recoveries of each analyte should be within ± 30 percent of the expected value. If the recovery of one analyte exceeds the ±30 percent criteria but is less than ±50 percent recovery, sample analysis may proceed and all samples analyzed that day containing that analyte must be flagged with a "Q". If more than one component of the calibration check standard does not meet this criteria or if any component exceeds ±50 percent recovery, the standard is reinjected. If the second calibration check standard does not meet the criteria, corrective action must be taken and/or a new multi-point calibration must be performed.

- At the end of the day, a mid-level calibration standard is injected and the percent recovery is calculated for each calibrated compound. If any analyte in the QC check sample varies from the expected value by more than 30 percent, all results are flagged with "Q" for the analyte.

## 6.0 SAMPLE ANALYSES

- A 50 cc glass sampling syringe is delivered to the laboratory with the Field Data Sheet and Analytical Result Data Sheet containing the chain-of-custody documentation.

- The operator signs the chain-of-custody and records the time of sample transfer.

- A 250 microliter aliquot is taken from the 50 cc syringe and injected into the GC/ECD instrument. A 1.0 milliliter aliquot is injected into the GC/PID instrument. The sample identification number, volume of sample injected, date, and run number are recorded in bound logbooks for each instrument.

- If a dilution is necessary to get the analytes within the calibration range, one of two procedures are used. A smaller aliquot of the sample is injected for dilutions up to 10. For dilutions greater than 10, an appropriate aliquot of the sample is mixed with UHP nitrogen in a 50 cc syringe and 250 microliter and 1.0 milliliter aliquots of the mixture are injected into the GC/ECD and GC/PID instruments, respectively.

- After the end of the run, the operator enters the results for calibrated compounds on the Analytical Results Data Sheet.

- Results for any unidentified compounds are calculated using the RF for the calibrated compound closest in retention time to the peak of the unknown compound. The result is entered on the Analytical

Results Data Sheet along with the identity of the closest calibrated compound.

- All analyses are completed within a 4-hour holding time.

- The injection syringe is purged with UHP air or nitrogen between samples and UHP air is injected following high level samples (> 50 ppbv on ECD or > 250 ppbv on PID) to minimize cross-contamination.

## 7.0 BLANKS

### 7.1 System Blank

- A system blank is analyzed daily.

- An injection is made with a 250 microliter aliquot of UHP $N_2$ on the GC/ECD and with a 1.0 ml aliquot of UNP $N_2$ on the GC/PID. The results must be below the reporting limits before field samples are analyzed.

### 7.2 Field Blank

- A field blank (or equipment blank) is analyzed daily.

- A 50 cc sampling syringe is selected at random from the batch cleaned the previous night and given to the field crew to use for the field blank.

- The field blank is run immediately after the system blank by injecting a 250 microliter aliquot. The field blank should be less than 10 ppbv for halogenated compounds and less than 50 ppbv for aromatic compounds.

- If the field blank exceeds these concentrations, the field task leader is notified and another field blank is collected. If the second field

blank does not meet the criteria, all data for that day containing the analyte(s) which did not meet the criteria are flagged with an "FB". Corrective action must be taken before the next sampling day.

### 7.3 Ambient Blank

- A 250 microliter aliquot of ambient air is taken from the on-site lab or a sampling site and analyzed. An ambient blank is collected when contamination from ambient air, either during analysis or sampling, is suspected.

## 8.0 DUPLICATE INJECTIONS

- Every five samples, a duplicate sample is taken from the 50 cc sampling syringe for injection into the GC to monitor analytical reproducibility.

- If the relative percent difference is greater than 30 percent for analytes within the calibration range, the GC system is checked for leaks or plugs and a third aliquot is injected. If the RPD of the third aliquot is greater than 30 percent from either of the first two injections, the next sample is also analyzed in duplicate. If the RPD of those duplicates is greater than 30 percent, corrective action must be taken before sample analysis can proceed.

## 9.0 DETECTION LIMIT STUDY

A detection limit study is performed at the beginning of each soil gas sampling and analysis project by analyzing a low-level mixed standard seven times. The method detection limit is calculated by multiplying the standard deviation of the replicates for each analyte by three. This method is outlined in Appendix B, Part 136 of the Federal Register 40 CFR. Values will not be reported below the detection limit.

## 10.0 SHUTDOWN FOR OVERNIGHT

- The column is cooled to 35° C and the septa are replaced in the injection port of each GC.
- The column is heated to 125° C for overnight bakeout.
- Used 50 cc sampling syringes are disassembled and put in the syringe cleaning oven at 160° C for decontamination.

## 11.0 CORRECTIVE ACTIONS AND PREVENTATIVE MAINTENANCE

### 11.1 Leak-Checking the Sampling Syringe

At the end of the GC run, the 50 cc sampling syringe is checked for leaks or plugs by the following procedure.

- The mininert valve is opened and the plunger is pushed forward to expel soil gas from the needle. This is done in a well ventilated area.
- If excess resistance is encountered the mininert valve and stainless steel needle are inspected for plugs. This is noted in the logbook and on the Analytical Results Summary Sheet for the sample.
- If the mininert valve fits loosely on the 50 cc syringe it is discarded. This is noted on the Analytical Results Summary Sheet.

### 11.2 Changing Injection Port Septum

- The injection port septum is changed at the end of each day to allow overnight conditioning.
- If the RTs of calibrated compounds shift by more than 0.08 and the air peak also shifts, the septum is changed before running any more samples.

## 11.3 Gas Purifying Filter

- If the background signal from the PID/ECD detector suddenly changes and no leaks are found, a new gas purifying filter is installed in the carrier gas line.

## 11.4 Downtime on GCs

- If one of the GCs cannot be calibrated due to an instrument malfunction, an emergency (24 hour) service visit will be requested from U.S. Analytical Instruments.

- If downtime is projected to exceed 48 hours a backup HP5890 ECD/PID from the Radian office will be brought on site.

- A spare 10.6 EV lamp for each PID detector will be on-site to minimize downtime from lamp burnout.

## 12.0 REPORT FLAGS

The following flags will be used on the Analytical Results Data Sheet to qualify data. These flags may be entered by the analyst or a member of the QA staff who reviews the data. A comment is included on the Analytical Results Data Sheet by the analyst if unusual circumstances are encountered or further explanation may be helpful in assessing the quality of the data.

## REPORT FLAG DEFINITIONS FOR DOWNHOLE SOIL GAS

| | | |
|---|---|---|
| FB | = | Detected in field blank |
| B | = | Detected in system blank |
| G | = | Indicates an estimated value due to GC interferences |
| PL | = | Duplicate injection RPD outside control limits |
| NA | = | Not analyzed |
| ND | = | Not detected at specified detection limit |
| E | = | Value exceeds the calibration range |
| Q | = | Lower accuracy than expected evidenced by calibration or QC check standard outside quality control limits |
| Y | = | Value suspect due to plugged or leaking sampling syringe. |
| J | = | Estimated value of tentatively identified compound. |
| PF | = | Duplicate field sample RPD outside control limits. |
| @ | = | Results are less than five times the method detection limit. |

## OU B SOIL GAS ANALYTICAL RESULTS DATA SHEET

Lab ID:_____ Sampling Time:_____
Date:___/___/___ Analyst Initials:_____

| | | | | |
|---|---|---|---|---|
| Injection Number: | | | | |
| Instrument: | | | | |
| Analysis Time: | | | | |
| Injection Volume (μL): | | | | |
| Dilution Factor: | | | | |
| QA Code[1]: | | | | |

[1]QA Code: NS - Normal Sample Result (only one per sample ID per method)
LD - Duplicate Analysis Result (at same dilution factor as NS)

| Compound Detected: | Concentration (ppbv): | | | | RPD (if applicable) |
|---|---|---|---|---|---|
| Freon 12 | | | | | |
| Freon 113 | | | | | |
| Chloroform | | | | | |
| 1,1,1-Trichloroethane | | | | | |
| Carbon tetrachloride | | | | | |
| Trichloroethene | | | | | |
| Tetrachloroethene | | | | | |
| Vinyl chloride | | | | | |
| trans-1,2-Dichloroethene | | | | | |
| cis-1,2-Dichloroethene | | | | | |
| 1,1-Dichloroethene | | | | | |
| Benzene | | | | | |
| Toluene | | | | | |
| m- & p-Xylene | | | | | |
| o-Xylene | | | | | |
| Unknown_____ | | | | | |
| Unknown_____ | | | | | |
| Unknown_____ | | | | | |

COMMENTS:

CHAIN OF CUSTODY:

Released by (Sampler):_____ Date/Time_____/_____

Received by:_____ Date/Time_____/_____

Released by:_____ Date/Time_____/_____

Received by:_____ Date/Time_____/_____

NET NATIONAL ENVIRONMENTAL TESTING, INC.

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# STANDARD OPERATING PROCEDURE

# FOR

# FIELD SCREENING ANALYSIS OF

# SOIL SAMPLES FOR POLYCHLORINATED

# BIPHENYLS (PCB's)

NET Pacific, Inc.

Mobile Laboratory Division

March 6, 1992

NET

NET Pacific, Inc

# FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR PCB's:

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# SOP FOR FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR POLYCHLORINATED BIPHENYLS (PCB's)

## Preface

Polychlorinated Biphenyls (PCB's) were manufactured for many industrial uses until 1976. The same basic properties that made PCB's so useful, their indestructibility, has resulted in widespread and persistent environmental contamination. Assessing and removing PCB contamination always involves laboratory analysis to measure PCB concentrations.

## 1.0 Scope and Application

This field screening method is intended to generate data of known quality in a short time to allow iterative evaluation of field sampling and/or remedial activities as they progress. A single chemist can complete preparation and analysis of up to twenty-five samples per day using this technique. This technique can be used for the analysis of chlordane as well as PCB's (commercial Aroclor mixtures).

## 2.0 Summary of Method

Soil samples are extracted by shaking with acetone. PCB's are then partitioned into hexane, while acetone remains in the aqueous-soil slurry. The hexane extract may be treated with 1:1 $H_2SO_4$, which oxidizes and removes a broad range of potentially interfering co-extracted organic compounds, including elemental sulfur.

Final analysis is by gas chromatography using and electron capture detector. Calibration and quantitation is performed by summing the areas of the four largest peaks for each target Aroclor analyte. The analyst identifies PCB's by pattern recognition; confirmation of identification using a dissimilar GC column is not required.

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# SOP FOR FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR POLYCHLORINATED BIPHENYLS (PCB's)

## 3.0 Safety

General laboratory safety practices require that laboratory personnel wear protective clothing whenever handling environmental samples and/or chemical reagents. Minimum protective clothing includes safety glasses, disposable gloves, long pants and closed-toe shoes. Additional protective clothing to be used when potential for exposure to extremely toxic or corrosive substances exists, includes a lab coat, a face shield, nitrile or other thick, impermeable gloves, and a full-face respirator with acid/organic vapor cartridges.

Mandatory safety equipment includes an eye wash, a first aid kit, a fire extinguisher, and a listing of local emergency service phone numbers posted near the telephone. A box of acid spill mixture is required where acids are used.

All sample manipulations, sample preparations and standard preparations should be performed in a fume hood with a linear velocity of at least 1 ft./sec. The electron capture detector effluent should be routed to a fume hood.

All corrosive and flammable liquids should be stored in cabinets and/or containers designed to contain spills and minimize health and fire danger.

## 4.0 Reagents and Materials

### 4.1 Reagents

4.1.1 1:1 $H_2SO_4$. Prepare by adding volume of concentrated (18N) $H_2SO_4$ SLOWLY to an equal volume of organic-free, deionized water

4.1.2 Pesticide grade acetone

4.1.3 Pesticide grade hexane

4.1.4 Deionized, organic-free (charcoal-filtered) water

## 4.2 Labware

4.2.1 Several each 2 ml, 10 ml, and 25 ml class A volumetric flasks for standard preparations and sample dilutions

4.2.2 Assorted microliter syringes

4.2.3 40 ml glass screw-capped vials (solvent-washed, D.I. water-washed and heated) with teflon-lined caps

4.2.4 12 ml glass screw-capped culture tubes with teflon-lined caps. Caps should be leak-free.

4.2.5 2 ml glass transfer pipets

4.2.6 Rubber pasteur pipet bulbs

4.2.7 Stainless steel scoopulas

# SOP FOR FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR POLYCHLORINATED BIPHENYLS (PCB's)

## 4.3 Equipment

4.3.1 Analytical balance capable of reading to 0.01g.
4.3.2 Mechanical shaker
4.3.3 Electronic timer/stopwatch
4.3.4 Thermometer
4.3.5 Refrigerators for sample storage and extract storage
4.3.6 Portable electronic gas leak detector
4.3.7 Instrumentation

4.3.7.1 A gas chromatograph system equipped with an electron capture detector and liquid autosampler. Integrator and/or data system capable of producing hard copy chromatograms and integration reports listing individual peak areas. The specific system used by NET will be HP GC hardware and PE Nelson chromatography software.

An instrument equipped with dual autosamplers, and/or dual columns and dual detectors will allow simultaneous temperature programmed analyses of standards and/or samples using one GC, thereby increasing instrument throughput.

4.3.7.2 Instrument critical spare parts include autosampler syringes, low-bleed injection port septa, glass injection port liners, 0.8 mm graphite ferrules, and a spare analytical column.

# SOP FOR FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR POLYCHLORINATED BIPHENYLS (PCB's)

## 5.0 Interferences

### 5.1 Laboratory Contaminants

5.1.1 Phthalate plasticizers are the most common laboratory contaminants encountered in the analysis of extractable organics. Any non-teflon plastic or rubber materials may contain potentially leachable phthalates.

5.1.2 Cross contamination of samples and/or standards by other high-level samples and/or standards may result from inadequate cleaning of labware (flasks, scoopulas, or syringes) or careless manipulation of samples. All labware should be soaked in alconox detergent solution, rinsed with D.I. water, and finally rinsed with pesticide-grade hexane after each use. Microliter syringes should be triple-rinsed in each of three successively cleaner hexane wash vials.

Disposable gloves should be worn by the analyst whenever samples or standards are handled, and should be discarded between each sample and/or standard manipulation to minimize the potential for cross-contamination.

# SOP FOR FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR POLYCHLORINATED BIPHENYLS (PCB's)

## 6.0 Procedures

### 6.1 Instrument Operation

6.1.1 Setup and troubleshooting - Refer to manufacturer's equipment manuals for information and instruction as needed.

A portable electronic gas leak detector is the analysts' most valuable tool in GC troubleshooting, and should be used after adjusting any fittings or whenever chromatography problems develop.

6.1.2 Preventive Maintenance

Replacement of injection port septa and rinsing and replenishment of the autosampler solvent rinse vials should occur after each 100 injections.

Clipping of the column, replacement of the glass injection port liner, or detector bakeout may be required more frequently than once per month, but should not be performed when a system is generating acceptable data.

**SOP FOR FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR POLYCHLORINATED BIPHENYLS (PCB's)**

6.1.3 Instrument Conditions

6.1.3.1 Autosampler (HP 7673)

Set to rinse 5x in clean solvent and 2x in sample before each injection.
Set injection volume at 2ul.

6.1.3.2 Gas Chromatograph (HP 5890 II)

Injection port: packed, at 270°C

Column: 30m x 0.52mm, 1.2um film DB-5 or equivalent.

Column oven temperature:

175°C for 2 minutes, ramp at 5°C/minute to 275°C, hold for 5 minutes.

Carrier Gas:

5% methane/argon at 7 ml/minute

ECD detector block temperature: 300°C

Makeup Gas:

5% methane/argon at 20 ml/minute

Detector attenuation: 1

Detector Range:

Adjust to give ca. 200 mv response for largest peak in 2.0 ppm Aroclor 1254 standard.

6.2.3 Continuing Calibration Verification (CCV)

A single level (0.5 or 1.0 ug/ml) standard of one aroclor from the same stock solution as the initial calibration standards should be analyzed at the beginning of each day, after each ten injections, and at the end of each daily analysis sequence. These continuing calibration check standards must meet the criteria for RPD referenced in section 7.3. The aroclor used for the continuing calibration should be the aroclor detected in samples. If aroclors other than the one in the CCV standard is detected in samples, the next CCV should include the aroclor of interest.

Analyses of samples should not continue if CCV criteria are not met until a new multipoint calibration is performed for any aroclor where CCV results are outside specifications. Results for any samples analyzed after the last in-control CCV and before the out-of-control CCV must be flagged as either estimated or false-positive, and reanalysis after corrective action has been taken is necessary if the out-of-control CCV differs by ≥ 50% from the expected response.

6.3 Preparation and Sources of Standard Solutions

6.3.1 Stock standard solutions of individual aroclors in methanol at a concentration of 200 ug/ml, purchased from Supelco, are used for preparation of calibration and CCV standards.

6.3.2 Individual quality control check (QCC) solutions containing aroclors 1242 and 1260, prepared from neat aroclors purchased from Supelco, are used to prepare ICV standards and to spike samples for MS/MSD analyses.

Preparation of standards from neat aroclors requires the use of the analytical balance at the NET Santa Rosa laboratory. Pesticide residue methanol, also from the NET Santa Rosa lab, is used as the solvent for the QCC solution, which is prepared at a concentration of 200 ug/ml using quantitative transfer technique and a 200 ml class A graduated cylinder. Standard preparation notes should include source and lot number for both the neat material and the solvent, balance identification, preparation and expiration dates, exact volumes and masses employed, description of volumetric devices, analysts' signature, and preparation date.

6.3.3 Preparation of Working Standards

Working standards (initial calibration, ICV and CCV) are prepared in hexane using the appropriate methanol stock standard. Low and high level initial calibration standards, and mid level ICV standards should be prepared in the smallest practical volumes. CCV standards, used frequently, may be prepared in larger volumes. Stock solutions and dilution solvent should be allowed to warm to room temperature before aliquoting or volumetric adjustment. Microliter syringes and volumetric flasks are to be used for working standard preparation. Syringes used for standard preparation should be clearly marked and dedicated to that purpose.

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# SOP FOR FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR POLYCHLORINATED BIPHENYLS (PCB's)

The minimum volume to be dispensed from a 10 µl syringe should be 2 µl, and only whole microliter aliquots and final volumes follows:

| Desired Standard Concentrations | Aliquot 200 ug/ml stock | Syringe | Final Volume |
|---|---|---|---|
| 0.2 ug/ml IC | 2.0 ul | 10 ul | 2.0 ml |
| 0.5 ug/ml IC,CCV | 25.0 ul | 25 ul | 10.0 ml |
| 2.0 ug/ml IC | 20.0 ul | 25 ul | 2.0 ml |
| 1.0 ug/ml CCV | 50.0 ul | 50 ul | 10.0 ml |
| 1.0 ug/ml ICV | 10.0 ul | 10 ul | 2.0 ml |

### 6.3.4 Storage of Standards

#### 6.3.4.1 Stock Solutions

Stock solutions in methanol should be stored in tightly-capped vials and refrigerated. Solvent meniscus levels must be carefully monitored to avoid evaporation/concentration. Stock solutions can be used for six months from preparation or ampule breakage.

#### 6.3.4.2 Working Standards

Working standards should be stored in tightly capped culture tubes or auto-sampler injection vials and refrigerated. Working standards that have been vialed and injected should be discarded, as evaporation is likely. Working standards can be used for one month from preparation.

### 6.4 Sample Preparation

#### 6.4.1 Subsampling

A representative subsample should be removed from the sample container using a stainless steel scoopula. The exposed surface of the field sample, or portions in contact with the sample container should be avoided. Rocks, twigs, leaves and other debris should likewise be avoided.

The size of the subsample should be the desired sample mass to be extracted, 5 grams, divided by the number of discreet samples to be composited.

The subsample should be placed in a tared 40 ml glass vial. The total sample mass should be recorded in the extraction log after addition of each discreet sample. Masses of individual samples can be calculated by difference.

The analyst must take all steps to maintain sample integrity during subsampling, including using properly cleaned and heated (to remove any solvent) scoopulas, and proper resealing and refrigeration of samples.

# SOP FOR FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR POLYCHLORINATED BIPHENYLS (PCB's)

## 6.4.2 Extraction

5.0 ml of acetone is introduced into the 40 ml vial containing the sample. Using a graduated cylinder, the vial is capped and shaken for at least 5 minutes.

5.0 ml of hexane is then added to the 40 ml vial using a repipettor dispenser. The sample is shaken for 30 seconds.

Approximately 5 ml of organic-free water is added to the vial. The vial is agitated gently for 30 seconds. If a heavy emulsion is evident, centrifugation may help in recovery of the hexane extract.

Transfer the hexane extract into a 12 ml screw-capped vial using a glass pipet, being careful not to transfer any of the aqueous phase. If acid cleanup is necessary to eliminate matrix interferences, add 5.0 ml 1:1 $H_2SO_4$. Cap tightly and shake for 5 minutes. Centrifuge if necessary to assist in recovery of hexane layer. Repeat $H_2SO_4$ treatment if first $H_2SO_4$ cleanup becomes very dark brown or black.

Transfer the cleaned-up hexane extract to a 12 ml screw-capped vial for storage.

### 6.4.3 Sample Receipt and Storage

Samples are introduced into the laboratory along with a chain of custody. The analyst, after inspecting the samples against the chain of custody, receives the samples and signs the chain of custody.

Samples are stored in coolers which contain ice and then may be stored in designated refrigerated areas. Generally, after analysis is complete, the samples are promptly disposed of usually by the client.

## 6.5 Preparation of Matrix Spikes

MS/MSD's are prepared by introducing 50 ul of the .200 ug/ml QCCS into the 40 ml vial containing the 5g subsample before addition of the acetone solvent. Aroclor 1242 and 1260 should alternate as the MS/MSD analyte. The resultant spike level will be 1.0 ppm. Frequency and acceptance criteria for MS/MSD's are given in 7.5.

## 6.6 Documentation

Three bound laboratory notebooks should be kept. The first should contain a record of all standard preparation information, the second a record of all sample preparation information, and the third a log of all instrument runs and any maintenance required.

The standards preparation logbook contains the following information: A sequentially assigned number, type of analyte, lot number, original concentration, any type of dilutions, preparation date, and the analyst's initials.

# SOP FOR FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR POLYCHLORINATED BIPHENYLS (PCB's)

The instrument run log consists of a copy of the daily sequence file from the GC. The information contained will be the following: Sample name, method name, data file and dilution factor. Spiking compounds are identified on the sequence file and cross referenced in the standards preparation log book. Sample weight is always 5.0 grams, so it is not recorded in the log book or sequence file.

## 6.7 Calculations

### 6.7.1 Response Factors

Response factors are generated for each analyte by summing the areas of the four largest peaks:

$$RF = \frac{\text{ng injected}}{\text{total area}}$$

The mean RF for each aroclor for the initial three-point calibration is used to quantitate all QC samples and actual samples

### 6.7.2 Sample Concentration

$$\text{Sample concentration (mg/Kg)} = \frac{\text{analyte detected (ng)}}{\text{sample injected (mg)}}$$

Sample injected = 2 ul x ~5g/5ml = ~2mg

Analyte detected = Total Area x RF

### 6.7.3 Quantitation of a PCB

Quantitation is performed by summing the areas of five or more target peaks for each aroclor analyte. Peaks, which are plus or minus two standard deviations from the mean, can be omitted from the calculation of the average. The analyst identifies pcb's by pattern recognition. confirmation of identification using a dissimilar GC column is not required.

## 7.0 Quality Control

### 7.1 Data Quality Objectives

NET's QC objectives are to conduct analyses in a manner such that the data quality meets the users' requirements. Data quality objectives are measured in terms of being complete, precise, accurate, representative and comparable.

The data quality objectives for field screening of soils for PCB's may be different for different projects. The frequency of and acceptance limits for quality control analyses may be revised to reflect such differences.

### 7.2 Initial Calibration and Initial Calibration Verification Requirements

The response factors (RF's) for each aroclor for the initial three-point calibration must meet criteria for linearity in order for analysis of samples to begin. The criteria for linearity is RSD $\leq$ 20% or correlation coefficient $\geq$ 0.990.

An initial calibration verification standard of aroclors 1242 and 1260, prepared from the QCC solution (second source standard) must be analyzed directly after a new multipoint calibration for 1242 or 1260 is run, and before analysis of samples can begin. The RPD between the mean RF of the multi-point calibration and the RF of the ICV must be ≤ 30% for analysis of samples to begin.

7.3 Continuing Calibration Verification Requirements

A mid-level (0.5 ug/ml or 1.0 ug/ml), same source standard of at least one aroclor must be analyzed at the beginning of each daily sequence, after each twenty (20) injections, and at the end of each daily sequence. The RPD between the mean RF of the multipoint calibration and the RF of the CCV must be ≤ 30% for analysis of samples to continue.

If this criteria is not met, the analyst must take corrective action, which may include instrument maintenance, standards preparation, recalibration, and reanalysis of samples analyzed after the last in-control CCV standard.

7.4 Method Blank Requirements

A method blank must be processed and analyzed each day samples are run, and at a frequency of at least one method blank per twenty (20) samples.

If any analytes are detected above the MDL (ca. 0.1 ug/kg), corrective action must be taken, including identification and elimination of the source of contamination, reextraction and reanalysis of affected samples, and flagging of any suspect data.

7.5 Matrix Spike/Matrix Spike Duplicate Requirements

A matrix spike/duplicate pair must be processed and analyzed at a frequency of at least one pair per twenty (20) samples. The spike should be done with the QCC standard.

Percent Recovery for both the MS and the MSD must be between 50% and 150%.

RPD of the concentrations measured in the MS and MSD must be less than 25%.

If the background concentration exceeds the spike concentration, sample non-homogeneity may lead to a high RPD, and another sample should be selected for MS/MSD analysis.

7.6 Method Detection Limits

Method Detection Limits are determined for each analyte in accordance with 40 CFR, Part 136, Appendix B, Revision 1.11.

The Reporting Limit may not be lower than the experimentally determined method detection limit.

A standard of each analyte at the Reporting Limit should be analyzed at the beginning of the project to demonstrate acceptable instrument response.

# SOP FOR FIELD SCREENING ANALYSIS OF SOIL SAMPLES FOR POLYCHLORINATED BIPHENYLS (PCB's)

## 8.0 References

8.1 "Field Measurement of PCB's in Soil and Sediment Using a Portable Gas Chromatograph," Thomas M. Spittler, Ph.D., USEPA, Region 1

8.2 "Test Methods for Evaluation Solid Waste," USEPA, November 1986. SW-846 3rd Edition, Section 1B

8.3 "Test Procedures for Analysis of Organic Pollutants," Appendix A, part 136, Code of Federal Regulations, 7-1-85 Ed.

8.4 "The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils," USEPA, 9/82

# DATA VALIDATION STANDARD OPERATING PROCEDURE FOR McCLELLAN AFB PROJECTS

## 1.0 PURPOSE

The purpose of this SOP is to define the procedures used to validate analytical data produced for McClellan AFB sampling and analysis projects. The procedures defined include review of field documentation, analytical reports, and project database tables, and assessments data quality through interpretation of quality control sample results.

## 2.0 SCOPE AND APPLICABILITY

### 2.1 Scope

This SOP describes (1) the review of chain-of-custody forms, sample collection master logbooks, field logbooks, field instrument calibration documentation, laboratory reports, raw QC and field sample data (if needed), electronic data transfer files, and QC and field sample data tables and (2) the procedures used to apply the field and laboratory quality control results to assess the validity of the data set and qualify results which do not meet the quality assurance objectives presented in the current McClellan AFB QAPP.

### 2.2 Applicability

The procedures described in this SOP are applicable to McClellan AFB projects where samples are collected and analytical data are produced. The use of these procedures by qualified personnel will assure that sampling, analytical, and reporting errors are minimized, and that any problems which may affect the quality of the data can be identified and addressed. All reports that incorporate analytical results should include an assessment of the quality of the data as described in this SOP.

## 3.0 DATA REVIEW PROCEDURES

Field logbooks, chain-of-custody forms, laboratory reports, and data tables produced from the project database are reviewed for errors or discrepancies as explained below. All problems or errors are entered in a QA logbook for the project. This logbook is accessible to staff involved in the project so that all project management personnel and QA personnel performing data quality audits, systems audits, or data validation can review the status and data quality impacts of any errors which have occurred and monitor corrective actions.

## 3.1 Review of Field Documentation

Field data sheets and master logbooks are checked for completeness and consistency. Date of sampling, field identification, type of sample (normal sample, field duplicate, trip blank, etc.), volume of sample, analysis required, and any comments which may affect the interpretation of the sample results are checked. The number of field duplicates and blanks collected for the program is compared to that specified in the QAPP or project sampling plan. A comparison of chain-of-custody forms and entries in the logbook and electronic master log is performed to ensure that sample information has been transferred correctly and legibly. Documentation of calibration procedures for field instruments and other field parameters is checked for adherence to the specifications in the QAPP, completeness, and consistency.

## 3.2 Review of Analytical Reports

Analytical reports and a copy of completed chain-of-custody form(s) are received from the laboratory. The reports and chain-of-custody forms are compared to ensure that all samples were analyzed as requested. All analytical reports are reviewed for the following items until the reviewer is confident that any systematic errors no longer occur and that random errors are kept to a minimum. At this time, the following entries are checked in at least ten percent of the analytical reports. This list represents the minimum level of review; additional items may be reviewed depending upon the type of laboratory report requested.

- The date sampled agrees with that listed in the sample master logbook.
- The method-specific holding times have been met for sample extraction/digestion and analysis (including second column analysis for GC methods).
- The sample identification number matches that shown in the master logbook.
- The analytes reported agree with the analyte list specified in the current McClellan QAPP.
- The detection or reporting limits meet the limits specified in the current McClellan QAPP and are adjusted for sample dilution.

- Recoveries of laboratory control samples, surrogate, matrix, analytical, and method spikes meet the laboratory quality control limits and the project quality assurance objectives for accuracy (stated in the QAPP or sampling and analysis plan).

- The relative percent difference for laboratory and matrix spike/matrix spike duplicates meet the laboratory quality control limits and the project quality assurance objectives for precision.

- The laboratory review procedures have been followed and are documented (signed by supervisor or laboratory director).

- Results are flagged when necessary and complete, understandable comments are provided.

- The units are correct and consistent.

- The associated reagent blanks are included with the laboratory report and meet the quality assurance objectives.

- The reported concentrations are within the calibration range.

- The confirmation of gas chromatography (GC) analyses is performed as required in the QAPP and results from both analyses are reported and include appropriate report flags.

- All raw data requested from the laboratory (computer printouts, chromatograms, and/or data calculation logbooks, as applicable) are complete, and reported values are consistent with the raw data.

- Disks for electronic transfer of data to database programs include all data files.

## 3.3 Review of Database Tables

Tables of analytical results for the field sample dataset and quality control samples are produced from the project database. The integrity of data stored in the database is verified by comparing approximately 10 percent of the entries to the laboratory reports. A higher percentage of results will be compared if the error rate indicates problems in the electronic data transfer step. The verification continues until the reviewer does not find any more errors.

## 4.0 CORRECTIVE ACTIONS

Corrective actions are required for both random and systematic errors. Systematic errors are reproducible errors introduced by faulty instrumentation, methods, or techniques which affect the quality of a batch or set of data. Random errors are not repetitive and affect individual results. Prompt action is necessary to correct systematic errors and minimize the volume of data which may be affected. The corrective action depends upon the source and extent of the problem. Corrective actions are documented in the QA logbook for the project.

### 4.1 Corrective Actions for Field Documentation Errors

The project director and/or field task leader are notified in a memorandum of any anomalies in the masterlog, chain-of-custody forms, or in field instrument calibration documentation. The project director or field task leader is responsible for making corrections and providing additional training for field task members concerning appropriate procedures and proper documentation requirements. The impacts and recommended corrective actions are also discussed in the project QA/QC Letter or other data quality assessment documentation.

### 4.2 Corrective Actions for Errors on Analytical Reports

Corrective actions for errors in reporting include (but are not limited to) the following.

- An Analytical Request Form (Figure 4-1) or similar form is issued to the laboratory client services representative of the laboratory from the QC reviewer. This form is used for both technical and non-technical errors, both systematic and random. The laboratory is required to submit corrected analytical reports when requested. Examples of technical errors which may be documented on this form are: laboratory control samples exceeding control limits, reporting limits that do not meet the method-specified criteria, samples analyzed beyond the holding time, and reagent blank results of compounds greater than three times the reporting limit. Examples of non-technical errors are: incorrect or inconsistent units, incorrect or missing data flags, inadequate narrative describing a problem, incorrect sample identification number, or incorrect sampling or analysis dates. This type of report is also used to request missing data (sample or associated QC sample results).

Tracking No: _____

## ANALYTICAL REQUEST FORM

TO: PROJECT REFERENCE:

FROM: LAB/SAM REFERENCE:

DATE (Issued):

DATE (Required):

Information Required:
verbally/written (circle one or more)

Corrections Required:
release hardcopy/re-export/document* (circle one or more)

Data Modification Required: ENTER DELETE CORRECT
(Attach all relevant data sheets or floppy disks)

Comments/Response/Explanation:

Verification: Date:

Distribution: Project File, QC File, Analytical Task Leader

* document on company letterhead for Radian files

**Figure 4-1. Analytical Request Form**

- A Corrective Action Report (CAR) addresses systematic technical questions or apparent deviations from the specified analytical procedures and QA objectives (Figure 4-2). This form, or a simile, is completed by the reviewer and submitted to the client services representative at a Radian laboratory. The laboratory must provide a technically valid explanation for the error, and may be required by the project director (PD) to reextract and reanalyze the sample. If sufficient sample volume is not available or the holding time has expired, the PD will decide if resampling and reanalysis are required. Examples of technical errors are: laboratory control samples exceeding control limits, samples analyzed outside the calibration range, calibration checks or reporting limits that do not meet the method-specified criteria, or reagent blanks results of compounds greater than three times the reporting limit.

- Systematic errors originating in the Radian laboratories may be resolved by submitting a Recommendation for Corrective Action (RCA) form (Figure 4-3). RCAs provide a systematic mechanism for documenting problems and corrective actions. The RCAs are issued to the responsible laboratory manager, and successively higher laboratory or corporate managers become responsible if the RCA is not addressed within the requested time period. The RCAs are tracked by the Radian's Corporate QA Group in Austin, Texas.

## 4.3 Corrective Actions for Database Table Errors

Errors in database tables originate from two sources: 1) the electronic data transfer file from the laboratory, or 2) the software used to transfer and report the information in the database. If the electronic file contains errors, a new file is requested from the laboratory. Alternatively, if the error can easily be corrected, the erroneous entry in the database (originating from the electronic export file) can be manually edited. If the error has occurred during data loading or a problem with the database software is apparent, corrective actions are requested. A request form (Figure 4-4) is issued to the database manager describing the erroneous entry or entries and is filed with the database manager. New database tables are generated and reviewed to ensure that corrections have been made.

# Corrective Action Report (CAR)

## Part I Initial Information (Furnished by Originator)

Originator: _____ Urgency Level:

SAM #: _____ Method #: _____ Requires resolution for immediate job ☐

Client: _____ Matrix: _____ Requires resolution for future jobs ☐

Date:

To Person Responsible for Action: _____

**Present Situation Requiring Action:**

Site / Lab: Type: QC Limit ☐ Documentation ☐ System ☐ Other ☐

Date / Time Identified:

Description of Situation: (attach supporting data if available)

**Recommended Corrective Action or Improvement:**

Description: Implemented by:

**Figure 4-2. Corrective Action Report**

0491-078-51

**Part II Reply / Resolution (Furnished by Technical Director)**

Proposed by: Date: CAR #:

Description: Scheduled Implementation:

**Part III Implemented Correction Action (by: )**

Description: Date Implemented:

**Part IV Follow-Up Required: Yes ☐ No ☐ (by: )**

Verified by: Date: Comments:

**Figure 4-2. Corrective Action Report (continued)**

0491-078-52

RESEARCH & ENGINEERING

# RECOMMENDATION FOR CORRECTIVE ACTION

## A. Initial Information

RCA NO: DATE:

ORIGINATOR: APPROVED BY:

ORGANIZATION/INDIVIDUAL RESPONSIBLE FOR ACTION:

URGENCY LEVEL ☐

1 Potential for major data loss or misrelease.
2 Potential for failure to achieve data quality objectives.
3 Suggested improvement.

## B. Problem Identification

SITE/LAB: SYSTEM: DATE PROBLEM IDENTIFIED:

DESCRIPTION OF PROBLEM:

## C. Recommended Corrective Action

DESCRIPTION: IMPLEMENT BY:

## D. Problem Resolution

PLANNED CORRECTIVE ACTION: PROPOSED BY: DATE PROPOSED: SCHEDULED IMPLEMENTATION:

IMPLEMENTED CORRECTIVE ACTION: DATE IMPLEMENTED:

## E. QA Verification

VERIFIED BY: DATE: COMMENTS:

White: Return for Implemented Corrective Action Yellow: Return for Planned Corrective Action Pink: File copy Gold: Originator's Copy

Information Copies Distribution: _____ _____ _____ _____ _____ _____ _____

_____ _____ _____ _____ _____ _____ _____

3 88 26418

**Figure 4-3. Recommendation for Corrective Action Form**

0491-078-58

**REQUEST TO ENTER/DELETE CORRECT McCLELLAN AFB DATABASE DATA**

NOTE: Attach copies of all relevant data sheets or floppy disks.

Requested by: _____ Date:_____

Desired Activity (circle one):

Enter Data  Delete Data  Correct Data

Description of Data and Location of Data (e.g.,attached sheets, floppies,etc.):
Location:_____ Field Sample ID:_____ Laboratory ID:_____

_____

_____

_____

_____

_____

Charge Number: _____

Comments:_____

_____

The requested additions/deletions/corrections are correct on the submitted sheets or electronic media and should be entered (Requestor):

_____ _____ _____
Signature  Title  Date

I have reviewed the data and approve of this request (Project Peer Reviewer):

_____ _____ _____
Signature  Title  Date

The additions/deletions/corrections have been entered and are correct in the database (McClellan AFB Database Manager):

_____ _____ _____
Signature  Title  Date

Please circle:

c: With Attachments
Location or Well File

c: Without Attachments
Project QA File
Project Director
Database Manager

**Figure 4-4. Database Correction Form**

## 5.0 DATA QUALIFICATION AND REPORTING PROCEDURES

Data qualification and reporting are procedures conducted by McClellan project QA staff to assess data quality after all the data have been received from the laboratory and loaded into the database. The QA staff identifies any problems, determines whether field data are affected, and states in the data assessment report how the data may be limited for use (i.e., qualified) in the intended applications. Data qualification and reporting procedures include:

- Evaluation of blank results to identify systematic contamination;
- Statistical calculations for accuracy and precision using appropriate quality control sample results;
- Qualifying data based on the QA objectives specified in the current McClellan QAPP to reflect limitations identified during data review and evaluation of blank, duplicate, and spike results; and
- Preparation of a QC summary report which includes discussions of qualified data, overall data quality in relation to the project objectives, and estimates of completeness in terms of the percentage of valid data.

The QA staff are responsible for ensuring that data quality has been assessed, that data are qualified according to the established QC criteria, and that QC data are reported appropriately. The typical procedures for assessment of blank contamination, accuracy, precision, and completeness are discussed below; however, alternate assessment procedures may be used by qualified QA personnel familiar with the sampling and analytical methods if unforeseen circumstances are encountered. These procedures will be discussed in the individual assessment reports.

### 5.1 Blank Data Assessment

Reagent blank results indicate whether any or all of the contaminants reported in sample results may be attributed to laboratory sources (reagents, glassware, instrumentation).

If any analyte is reported in reagent blanks, the samples associated with the blank, in either the same analytical or extraction batch, may be qualified to indicate that some or all of these analytes may have been introduced from laboratory sources. If the concentrations reported in the associated samples are less than 5 times the blank concentrations, it is likely that some or all of the contamination was introduced in the

laboratory, and the results are qualified to indicate laboratory contamination.

Results for other types of blanks such as equipment, ambient, or trip blanks are assessed individually. The probable source of contamination is identified and associated sample results are qualified as necessary. For example, if equipment blank results show contamination, and the sample collected from the same sampling equipment (or sampling equipment which have been decontaminated using the same procedure) shows the same analyte, sample results will be qualified to indicate a probable level of introduced contamination. The assessment of all types of blank data is discussed in the QA report for the sampling task.

## 5.2 Assessment of Accuracy

Accuracy is a comparison between a measured value and the expected, value. Accuracy is assessed from method spike, matrix spike, and surrogate spike results.

Spike results are reported by the laboratory as percent recovery and are compared to the accuracy objectives presented in the McClellan QAPP. Associated results are qualified for inaccuracy if the spike recovery is outside the project objectives.

Method spikes are spikes of a reference material into a pure matrix. If recovery is outside established limits, samples from the same extraction or analytical batch are qualified. Matrix and surrogate spike results are generally more sample-specific. If matrix or surrogate spike recovery is outside the established limits, results for samples collected from similar conditions and/or handled in the same batch will be examined. Results for analytes similar in chemical structure to a surrogate compound recovered outside the predetermined limits will also be examined. If any results appear to have been affected, those results may also be qualified. The flagged data (and any ensuing or recommended corrective actions) will be discussed in the QA report for the sampling task, and specific limitations such as poor or enhanced recovery for specific compounds will be stated.

Confidence intervals or other statistical measures may be calculated for selected analytes from specified McClellan projects if a series of performance evaluation samples is submitted, a series of method spikes is analyzed, or a large pool of sample or spike duplicate results is available. The results are used to define confidence intervals or experimentally-derived recovery limits for the analytes included in the

PE samples or method spikes, or statistical limits or confidence intervals for precision based upon duplicate results. These measures provide criteria to evaluate historical sampling and analytical performance. These procedures and results will be described in the associated QA or project report.

## 5.3 Assessment of Precision

Precision is a measure of variability between duplicate (2) or replicate (3 or more) analyses. For McClellan projects, precision is calculated for field and laboratory duplicates. By definition, field, or total precision incorporates laboratory precision. Precision is calculated as the relative percent difference (RPD) between duplicate samples or analyses, or matrix spike/matrix spike duplicates. The calculated RPDs are compared to the quality assurance objectives stated in the QAPP. The analyte results for the samples collected or analyzed in duplicate are flagged if they do not satisfy the QA objectives.

An average RPD may be calculated and reported as a measure of overall analytical or sampling precision. If the average RPD of an analyte exceeds the precision objectives specified in the QAPP, that analyte is flagged in associated samples to indicate variability due to poor precision. Associated samples may include samples collected by one sampling team, with one set of equipment, on the same day, or samples processed and analyzed in the same batch. Close evaluation of those results should indicate the most likely source of variability, and corresponding samples will be qualified as warranted.

Occasionally, an RPD cannot be calculated because the analyte in one of the duplicate pair has been quantitated and reported and the analyte is not detected in the other sample of the duplicate pair. If the concentration reported is low (less than 5 times the DL), the greater relative variability near the detection limit is considered the cause for this occurrence and this data would not be qualified to indicate poor precision. A qualifier flag indicating imprecision in the field or analytical procedure would be assigned to data if the concentration reported for one of the duplicate pair was greater than 5 times the detection limit but the analyte was not detected in the other sample of the duplicate pair. Investigation of these data may indicate sample-specific differences or errors, such as inconsistent dilution factors between duplicates, reporting errors, or systematic differences, such as non-homogeneity of soil samples or consistent poor precision at low concentrations. If systematic errors are identified, similar samples may also be qualified.

## 5.4 Assessment of Completeness

Completeness is calculated after QC data have been evaluated, and the results applied to sample results. In addition to results identified as being outside of QC limits established for the method, the occurrence of matrix effects, broken or spilled samples, or samples that could not be analyzed for any other reason are included in the assessment of completeness. The percentage of valid, unqualified results is reported as completeness and is compared to the completeness objective specified in the QAPP.

Completeness is calculated by dividing the number of valid, unqualified results (the possible total number of results minus the number of qualified results and results not reported because of broken or spilled samples, etc.) by the total number of individual analyte results requested.

For example, if from a total of 1,000 individual analyte-measurement results (both detected and nondetected), 30 detected results were qualified and 10 analyte results were not reported because of a spilled sample, the completeness percentage is calculated as follows:

$$\frac{1{,}000 - (30 + 10)}{1{,}000} \times 100 = 96\%$$

If the completeness objective stated in the QAPP is not met, the percentage of types of qualification (field duplicate RPDs, laboratory blank contamination, etc.) are compiled and evaluated to determine if the sampling or analytical procedures are capable of providing the quality of data specified in the QAPP for the sample types, site characteristics, and matrices encountered. The completeness objective may be reevaluated for a specific site or sample matrix because parameters which affect data quality (such as homogeneity of soil, matrix interferences, etc.) are beyond the control of the project personnel. This information can be used in determining further sampling (or analytical) strategies and quality assurance objectives.

## 5.5 Quality Assurance Reports

A report discussing the QC sample results, performance audits, systems audits, and data quality audits is issued at the end of every sampling and analysis task. Quality assurance reports for tasks that involve collecting samples over a long-term period (one quarter or longer) are prepared on a monthly or quarterly basis as defined in the project SOW. The information presented in the quality assurance reports is

incorporated as an appendix into the reports prepared for the task or in separate task QA/QC reports, and follows U.S. EPA format or a format specified in the scope of work for the task.

The report contains an overall summary of data quality for the project or event, which includes a discussion of any systematic errors which may affect the precision or accuracy of all data for a given parameter or method, random errors which affect data quality that were discovered during data review and could not be resolved, a discussion of holding time compliance, and an assessment of completeness. Results of performance, systems, and data quality audits are also summarized in the QA report. Qualified results are presented in the summary section of the report in a table of qualified data, with the reason for qualification, and analytes affected. Qualified data are also flagged in the database so that any tables or reports that include the data indicate that the results are qualified or limited for use. The report includes a detailed assessment of blank, duplicate, and spike results and a comparison of these results to project data quality objectives. Sampling and analytical quality control results are discussed separately so the source of any problems can be identified and appropriate corrective actions can be taken.

The content and format for the QA reports are presented in the following outline:

McCLELLAN AIR FORCE BASE
QUALITY ASSURANCE REPORTS

1.0 Summary of sampling and analytical activity and highlights of quality assurance results

2.0 Measurement data accuracy, precision, and completeness (for each sample matrix and method)

3.0 Results of systems audits

4.0 Results of performance and data quality audits

5.0 Significant quality assurance problems and recommended corrective action

The QC report is reviewed by the project QA officer and other internal QA and project management personnel, and a technical editor prior to external distribution.

## APPENDIX B

### Response to Comments

1. EPA Region IX Comments on Draft QAPP
2. Response to EPA Region IX Comments on Draft QAPP
3. EPA Region IX Comments on Summary of QAPP Revisions and Field Analytical Procedures for OUB RI, 22 August 1991 (Summary)
4. Response to EPA Region IX Comments on Summary
5. DTSC Comments on Summary
6. Response to DTSC Comments on Summary
7. RWQCB Comments on Summary
8. Response to RWQCB Comments on Summary
9. RWQCB Comments on Draft Final QAPP
10. Response to RWQCB and U.S. EPA Comments on Draft Final QAPP
11. U.S. EPA Region IX Comments on Draft Final QAPP
12. Response to U.S. EPA Region IX Comments on Draft Final QAPP

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IX
75 Hawthorne Street
San Francisco, Ca. 94105-3901

9 Dec 1991

Mr. Mario Ierardi
Environmental Management
SM-ALC/EM, Building 250N
McClellan Air Force Base, CA 95652

Dear Mr. Ierardi:

Enclosed are the comments of the Environmental Protection Agency (EPA) to the Installation Restoration Program (IRP) Stage 3 Quality Assurance Project Plan (QAPP) for McClellan Air Force Base (MAFB). The QAPP was reviewed for technical quality from the perspective of utilizing the document as a baseline standard operational procedure (SOP) QAPP for multiple projects and a scope of work (SOW) for new contractors working on MAFB. Because multiple contractors will be working on MAFB, it is very important the QAPP provide a clear framework to collect comparable data of known and defined quality. To provide this framework, two key areas need to be addressed (1) describe the relationship to other documents and (2) data needs based on data quality objectives (DQO) needs to be clarified.

A detailed description of the relationship between the QAPP and companion documents (sampling and analysis plan (SAP), SOW, and other documents) should be provided. This discussion should provide how documents will be amended and the general and specific categories of information that will be included in each document. There should be clear guidance on how new sample collection and analysis techniques will be added to the QAPP and companion documents. A mechanism should be in place so a complete draft QAPP is not issued every time the QAPP needs to be revised or amended.

The DQO's should be clearly defined and associated with different analytical levels. The analytical level selected for a sampling effort would depend on the end use of the data. For example, end uses of the data may range from a screening level characterization that require qualitative indication of contamination to a human health risk assessment that would require quantitative data and rigorous analytical procedures. The quality control measures would be implemented to determine whether the DQO's have been met should be clearly defined.

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The quality control limits for the prime contractor in the QAPP are not the same as those for the subcontractor, providing analytical services for the project, in the same QAPP. The project should have one consistent set of quality control criteria that meet the needs of the project, not the capabilities of different laboratories. The limits should be established to reflect project DQO's and set consistent criteria for each method. The project DQO's will define specific project objectives and insure sampling and analysis will meet the goals of the project with data of comparable quality.

If you have any questions please contact me at (415) 744-2412 or Ramon Mendoza at 9415) 744-2410.

Sincerely,

Lewis Mitani
Remedial Project Manager

enclosure

cc: distribution list

## Distribution List

State of California
Environmental Protection Agency
Department of Toxic Substances
Region I Site Mitigation Unit
ATTN: Mr. Mark Malinowski
10151 Croydon Way, Suite 3
Sacramento, CA 95827-7700

California Regional
Water Quality Control Board
Central Valley Region
ATTN: Mr. Alex MacDonald
3443 Routier Road, Suite A
Sacramento, CA 95827-3098

Planning Research Corporation
Environmental Management, Inc.
ATTN: Mr. Greg Reller
11030 White Rock Road
Suite 190
Rancho Cordova, CA 95670

PTI Environmental Services
4000 Kruse Way Place
Building 1, Suite 220
ATTN: Mr. David Watson
Lake Oswego, Oregon 97035

EPA Comments to
Draft Quality Assurance Plan
McClellan Air Force Base

GENERAL COMMENTS

1. Project Approach and Objectives - The description of data quality objectives (DQOs), project approach, and objectives should be expanded to clarify the quality of the data collected will be appropriate for the end use of the data. Similar to the draft final Operable Unit B sampling and analysis plan (SAP) (see Table 4-2; Radian 1991b), there should be a discussion regarding the levels of analytical testing that will be conducted during the site investigation. The level of quality control should depend on the analytical level required for the end use of the data. The sampling and analysis procedures discussed in the QAPP could be classified under different analytical levels and the appropriate level of quality control implemented. The investigation approach described in the Operable Unit B SAP should be incorporated and discussed in the QAPP.

2. Insufficient Detail - One of the major objectives of the QAPP is to provide guidance to the participants in the Installation Restoration Program (IRP) on the activities required to produce data of known quality. As such, the QAPP must contain enough detail for team members to fulfill this objective; alternatively, the QAPP can reference other documents that will provide this detail. In some instances, this QAPP lacks the appropriate level of detail, particularly in those sections dealing with sample handling, document control, and use of quality control samples.

The discussion of mechanisms for carrying out the quality assurance policies should include information on data that will need to be collected (i.e., what data gaps will be filled) and the investigation efforts that will be initiated to collect those data. This information presently is not included in the text.

3. Relationship of the QAPP to Other Documents - The QAPP does not include a discussion of the hierarchical relationship of the QAPP to other documents for this project, such as the project work plans, SAPs, or health and safety plan. This relationship should be specified in the Introduction section of the QAPP; if specified in another document, that document should be referenced and a summary of the relevant relationships provided. In addition, the QAPP appears to incorrectly refer to other documents such as work plans and SAPs. These documents should be

defined early on in the QAPP and referred to in a consistent manner. Specific comments should be referenced where possible.

4. Quality Assurance for Subcontractors - Generally, the discussion of quality assurance programs for subcontractors should be expanded. If such information is to be included in other documents, such as the SAP, this should be noted in the QAPP.

5. Revisions to the QAPP - The QAPP should include or reference a procedure for updating the QAPP and distributing revised copies to the appropriate individuals.

6. DQOs - The discussion of geologic DQOs did not incorporate the intent of the DQO process as defined in U.S. EPA (1987). The intent of the DQO process is to establish DQOs based on the end uses of the data to be collected. The text notes the use of precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters may not be totally applicable to geologic data. However, a qualitative measurement of some parameters (e.g., representativeness) is possible and should be incorporated into the text as appropriate. For example, a comparison between site data and regional data could be used to evaluate representativeness in this section.

The discussion of steps that will be taken to ensure collected data are as representative, comparable, and accurate as possible would be improved by specifically discussing each step. For example, standardization of data would be accomplished by the use of standard operating procedures (SOP) and field forms. Field forms ensure the correct data are collected. These procedures and forms will help ensure all data are collected using the same methods; thus, the possibility of error will be reduced and data comparability will be increased.

7. Acceptable Tolerances for Hydrological Measurements - The discussion of hydrologic measurements should include acceptable tolerances (e.g., discharge, depth to water, elevation of measuring point). Some of these tolerances are discussed in Stallman (1971). Precision of measurements should be discussed more completely in the QAPP. For example, although specifications for surveyed elevations and locations are generally discussed adequately, other data such as pressure measurements and flow rates need to be discussed more thoroughly.

8. Optional Testing Procedures - In a number of sections, the text identifies optional testing procedures may be used to collect data. The text does not describe the criteria upon which the selection of a particular testing option at a particular location will be based for all testing procedures. This

information is critical to ensure that data collection procedures will produce data that meet the DQOs. A discussion of these criteria should be included or referenced when more than one testing procedure or data collection technique is presented.

9. Decontamination Procedures - In the QAPP, decontamination procedures are discussed in each individual section where sampling or sampling equipment are discussed. It may be less confusing and may provide greater consistency if decontamination is discussed in its own section, and the section is referenced as needed. In addition, it is recommended that all equipment be given a final rinse with ASTM Type II reagent water to wash away any residual methanol or cyclohexane.

10. Loss of Volatile Constituents - To reduce the loss of volatile constituents from soil samples, McClellan should consider putting the soil samples in a methanol solution in the field (as recommended in U.S. EPA 1991a) or taking other measures discussed in U.S. EPA (1991a).

11. Sampling and Purging Wells - The text is not clear what procedures will be used for sampling and purging wells with free product, which are to be tested.

12. Redundancy Among Sections - Many sections of the QAPP contain repetitive information on procedures for decontamination, sample collection, and analysis (see general and specific comments on soil gas sampling). As much as possible, these procedures could be consolidated into separate sections and referenced. This will help limit redundancy, improve the readability, and reduce the potential for procedural inconsistencies among sections. For example, some repetition occurs among Section 5.6.1, Groundwater Sampling Equipment, and Sections 5.6.2 and 5.6.3, Equipment Decontamination Procedures and Field Procedures, respectively. In some cases, the sections are not consistent. For example, in Section 5.6.1, page 72, second paragraph, it is stated that five well volumes will be purged. In Section 5.6.3, page 78, paragraph 2, it is stated a minimum of three well volumes should be purged prior to sampling. For clarity, and to eliminate inconsistencies, the discussion in Section 5.6.1 should be limited as much as possible to equipment; the procedures should be outlined in Section 5.6.3.

13. Soil Gas Sampling - Because soil gas data are being used extensively at Operable Unit B and the other operable units to select soil sampling locations and define potential source areas, it is important to have comparable data produced from similar sampling and analysis methods. The QAPP has separate descriptions of each potential method that could be used to

create a soil gas sampling hole. Each description includes separate discussions of one or more methods that could be used for sample retrieval and analyses. Because there are some inconsistencies between sections (see specific comments) and in order to clarify the sampling procedures and increase the repeatability of the methods, it is recommended that this section be reorganized as described below.

There are two types of soil gas sampling holes described in the text. There are holes that are open to the atmosphere, such as hand auger holes, and driven probes where a discreet sample from a portion of the soil column is collected. The quality and type of sample collected and the end use of the data may be very different for the two types of soil gas sampling holes. It is recommended that the discussion in the QAPP address the difference in data quality between the two types of samples and describe the measures that will be taken to ensure comparability between the sampling methodologies. Although the procedures for creating the holes should be provided, it is recommended that SOPs be developed for collecting and analyzing samples for the two types of holes, independent of how the holes were created. More than one way to collect and analyze the samples could be presented. More detailed information on the quality assurance and quality control (QA/QC) measures (e.g., blanks, duplicates, laboratory procedures) that will be used to measure the PARCC parameters for the different analytical levels of soil gas data collected should be provided. The quality control measures followed will depend on the DQOs and end use of the data.

14. Mobile and Stationary Laboratory Quality Control Measures - The quality control procedures used for mobile laboratories vs. stationary laboratories should be explicitly stated in Sections 7.0 through 15.0. Although some of the procedures for the mobile laboratory are described in the draft final Operable Unit B SAP, they should also be presented in the QAPP so they are applied consistently between operable units.

15. Internal Quality Control - For both the laboratory and field quality control samples, the recommended frequency at which these samples will be taken and analyzed should be described in greater detail in Section 10.0. The frequency should be tied into the DQOs and end use of a given data set. It is recommended a decision tree be developed to guide the use of quality control samples. For example, an equipment blank may be preferred over an ambient blank if equipment is being decontaminated. However, in some circumstances, both types of blanks may be taken. If field screening is being conducted, blanks and/or duplicates could be taken on a less frequent basis than Contract Laboratory Program grade sampling.

16. Inconsistency with Guidance - As much as possible, the format and organization of this document should be made consistent with the latest EPA Region IX guidelines for preparation of QAPPs for Superfund remedial projects (U.S. EPA 1989) (see, for example, specific comments in Section 8.0, Analytical Procedures and Calibration). The QAPP should be reviewed and revised so that it contains all the items recommended in the more recent EPA guidance.

SPECIFIC COMMENTS

17. Table of Contents ,page iii

The page numbers for Sections 10.1 and 10.2 are not consistent with the Table of Contents. This should be corrected.

18. Section 1.0 Introduction, page 1 paragraph 1

A distribution list for official copies of the plan and subsequent revisions should be included in the QAPP immediately after page x of the Table of Contents.

19. Section 1.0 Introduction, page 1, paragraph 1

This section should reference the latest Region IX guidance document for the preparation of QAPPs (U.S. EPA 1989).

20. Section 2.3 Data Collection and Use, page 11, paragraphs 1 and 3

The text of paragraph 1 indicates that, among other types of samples, air samples will be taken at designated sites. It is not clear what type of air samples will be collected and what the purpose of air sampling is. Furthermore, collection of samples is not mentioned in paragraph 3, which describes the detailed work plan for Operable Unit B, even though several volatile organic compounds have been found in soil samples collected from sites in Operable Unit B (page 2-6). The revised QAPP (and/or SAP) should specify the sampling and analysis objectives and strategy for air sampling.

21. Section 3.5 Primary Task Leaders, page 4, paragraph 1

It is not clear from this discussion who has the primary responsibility for coordinating and tracking corrective action in the field, office, and laboratory. This responsibility should be clarified either by adding this information to this section or by referring to other sections of the QAPP.

22. Section 4.0 Quality Assurance Objectives for Measurements and Geologic Data; Tables 4-1 to 4-12, Quality Control Acceptance Criteria for EPA Methods; and Tables A-1 to A-12, Quality Control acceptance Criteria for EPA Methods, for BC Analytical, Glendale.

In Table 4-4, for EPA Method 8010 laboratory control sample (LCS) spike recoveries appear to cover an unusually broad range considering that a clean matrix is being spiked for a purge and trap analysis. In many cases the LCS recovery limits are wider than those for the matrix spiking compounds. It is suggested these limits be reevaluated, and at a minimum be made consistent with the matrix spiking limits. A rationale for how spike recoveries for a LCS sample can be over 150% or less than 65% and represent adequate method performance should be provided. This is especially true for recoveries over 200%.

The quality control (QC) limits provided by Radian are not the same as those provided by BC Analytical. The project should have one consistent set of QC criteria that reflect the needs of the project, not the capabilities or acceptance criteria of two different laboratories. The limits should be established to reflect project data quality objectives and set consistent criteria for each method.

Project Data Quality Objectives (DQOs) are required to, (1) define specific project objectives and (2) to ensure proposed sampling and analysis will meet the goals of the project. For example, these project objectives may be defined based on the risk assessment (to population or environmental) or designed to provide semiquantitative screening preparatory to further investigation. After the goals are defined, a sampling program may be statistically defined which will meet the project needs with the required degree of confidence.

23. Section 4.6 Geologic DQOs page 20, paragraph 2

The text indicates all field personnel will review geologic samples and cores. Review of these materials by all personnel may not be necessary and will probably not be efficient or cost-effective. The review of samples and cores should be limited to those personnel who actually require the information to perform their duties. In addition, the discussion of steps that will be undertaken to ensure that the data obtained are "representative, standardized, and accurate as possible" is incomplete. A discussion should be included of standardized field forms, soil color charts, SOPs, internal and laboratory QA/QC procedures, notebooks, photographs, and calibration procedures.

24. Section 4.6 Hydrologic Data, page 20, paragraph 3

Paragraph 3 discusses the hydrologic data to be compiled during the site investigation (i.e., water-level measurements and pump discharge rates). Hydrologic data that will be compiled should also include observations made during well installation, including well completion diagrams. The discussion should also note that the choice of equipment used to make hydrologic measurements is a function of data use and the equipment's operating specifications should be sufficient to meet the DQOs related to the data. A table summarizing types of hydrologic data, corresponding DQOs, and the equipment necessary to attain the DQOs is recommended.

25. Section 5.0 Field Procedures, pages 1-136, general comment

Because the location, number, and types of samples to be collected have not yet been determined, the document that will contain this information should be referenced.

26. Section 5.1 Geophysical Techniques, page 1, paragraph 5

The text states surface geophysical surveys will be conducted along transect lines. Station locations and ground elevations should be surveyed as part of the procedures for performing these surveys. The accuracy of surveyed locations and elevations should be included or referenced in the text. The text should include an explanation of how the instruments used to perform the surveys will provide data that meet DQOs.

27. Table 5-1, Water and Soil Sample Storage and Preservation Requirements.

Table 5-1 was inconsistent in its description of containers. For several of the methods where an extraction is required, the container listed is appropriate for soils but not for waters. If both types of samples are to be collected the table should be revised.

28. Section 5.1.1 Borehole Geophysical Investigation Procedures page 2, paragraph 2

The text states that natural gamma logging may also be conducted in boreholes. The criteria for determining if a natural gamma log will be run should be provided or referenced in the text.

The discussion of borehole geophysical survey procedures should include the procedures for storing data (or the SAP should be referenced for this information). The text includes a very

good discussion of the information that will be included on the logs, but no description of how the information will be archived. Presumably, the original field data will be stored in computer files on tape or disk and will be transferred to the project database. Procedures for storing and transferring the borehole geophysical data should be described in the QAPP. This same comment applies to the surface geophysical surveys (Section 5.1.2).

29. Section 5.1.2.2 Magnetometry, page 6, paragraph 3

When conducting a magnetometer survey, a base station should be established at the beginning of the workday, and the work schedule should provide enough time for a return to the base station to collect a calibration reading after measurements are performed at a few stations (Dobrin 1976). This procedure allows for evaluation of instrument drift, which may impact the readings, and should be incorporated into the text.

30. Section 5.2.1 Site Selection, page 6, paragraph 5

The text lists criteria for the selection of monitoring well and subsurface sampling locations. The list of criteria should include the presence and location of receptors such as water supply wells.

31. Section 5.2.1 Site Selection ,page 7, paragraph 2

The text discusses procedures that will be followed prior to drilling to minimize disturbing utility lines during well installation. The text should also include procedures (e.g., magnetometer survey, potholing, reduced drilling rates) to minimize disturbing other unknown buried objects (especially at shallow depths) during drilling.

32. Section 5.2.2 Drilling Preparation, page 8, paragraph 1

Procedures for managing waste materials generated by decontaminating the drill rig should be specified or referenced in the text.

33. Section 5.2.3 Drilling Methods, page 9, paragraph 2

The text discusses the conditions under which the mud rotary method will be used for drilling, but not the conditions under which other drilling methods will be used. To be consistent, the text should include a discussion of the conditions under which each of the drilling methods will be used.

34. Section 5.2.3 Drilling Methods, page 11, paragraph 1

Management and disposal of potentially contaminated drilling mud and cuttings are not discussed in the description of mud rotary procedures; they should be referenced (i.e., Section 5.2.5 of the QAPP). Also, portable pits should be used for containing mud and cuttings during drilling.

35. Section 5.2.3 Drilling Methods, page 12, paragraph 3

The procedures for managing waste materials generated by the bucket auger method should be referenced (i.e., Section 5.2.5 of the QAPP).

36. Section 5.2.4 Sample Collection Methods, page 16, paragraph 3

The text indicates core samples will be placed in boxes and stored. The text should indicate how the boxes will be marked with pertinent information, such as borehole number, sampled interval, date, and box number before leaving the drilling site.

37. Section 5.2.5 Cuttings and Groundwater Disposal (page 17, paragraph 3)

The text indicates that drill cuttings will be monitored for organic vapors. The frequency of measurement or the criteria for determining that frequency should be provided in the text to ensure uniformity of approach. The text should also state that measurements will be recorded in the field notebook or on a field form.

38. Figure 5-3, page 19

The length of the recovered sample or the percent of recovery should be included on the drilling operations form.

39. Figure 5-4, page 20

The location of the measuring point for water level measurements should be described or illustrated on the well completion form.

40. Section 5.2.6 Recordkeeping page 28, paragraph 2

The text notes that at the discretion of the Air Force, lithologic samples will be collected at a minimum of every 5 feet and stored in labeled sample bags. The collection of samples should be determined by investigation goals and DQOs, and the

policies for meeting those DQOs should be contained in the QAPP. If data gaps include lithologic descriptions, samples to fill those data gaps should be collected. Alternatively, sampling strategy could be described in the SAP.

41. Section 5.2.6 Recordkeeping, page 29, second bullet

The text indicates a favorable condition for screen interval selection is adequate penetration into the saturated zone with termination depths being a minimum of 10 to 15 feet deeper than the water table. Consideration should be given to the presence of floating product, which would require the well screen to intersect the top of the water table.

42. Section 5.3.1 Well Installation and Completion, page 33, first line and first bullet

The text indicates that stainless steel centralizers will be set at every 40 feet from the preceding centralizer, without stating where the first centralizer will be installed. Figure 5-10 indicates that the centralizers will be placed at the top and bottom of the well screen and then every 40 feet above the centralizer at the top of the well screen. The text should be clarified.

43. Section 5.3.1 Well Installation and Completion, page 35, paragraph 2

The text discusses procedures for placement of the sand pack around the screen. The procedure should also include maintaining the level of sand above the bottom of the drill string or drive casing to ensure that formation material does not collapse into the borehole. Also, a sounding tape should be used during sand pack installation to monitor the location of the top of the sand pack and to ensure that the level is above the bottom of the drill string or drive casing.

44. Section 5.3.1 Well Installation and Completion, page 35, paragraph 3

The text discusses procedures for ensuring that the well is properly aligned and notes that the procedures "should" be followed. The QAPP should state that the procedures will be followed or provide reference criteria for deciding if they will be followed. Criteria for determining if well alignment meets DQOs should be described, and procedures that will be followed if the casing does not meet those standards should be stated or referenced.

45. Section 5.3.1 Well Installation and Completion (page 35, paragraph 4)

The text states that bentonite slurry will be mixed until it is "viscous." The text should specify the weight of the slurry per gallon of slurry mixture and describe the method for measuring the slurry weight.

46. Section 5.3.1 Well Installation and Completion page 36, paragraph 5

The text indicates that only the elevation and location of the reference point for water level measurements will be surveyed. In addition, the elevation of the ground surface should be measured because it is the reference point for well installation measurements and lithologic samples. The ground surface elevation and other pertinent elevations (e.g., tops of casings) should be noted on the drilling operations form, Figure 5-3.

47. Section 5.3.1 Well Installation and Completion, page 36, paragraphs 4 and 5

In paragraph 4, the text describes procedures that will be undertaken at the end of installation of a monitoring well. The discussion in paragraph 5 of a notch on the top north side of the casing as the reference point for water level measurements should be moved to paragraph 4. Also, the text should note the notch should be placed at the time of well completion to allow for immediate access to the well for water level measurements rather than having to wait for the surveyor. The location of the notch should be documented on the well completion log, Figure 5-4. Whether the measurement will be taken at the top or bottom of the notch should be clarified.

48. Section 5.3.2 Monitoring Well Development, page 41, paragraph 1

The text states that wells will be pumped until pH, conductivity, and temperature parameters have stabilized. The criteria for deciding when stability is reached should be provided in the text of the appropriate section referenced.

49. Section 5.4 Aquifer Test Methods, page 41, paragraph 2

The text notes that a pumping test consists of pumping a well and recording drawdown. The pumping test should also include recovery measurements after the pump is shut off. Recovery measurements serve as a means to check calculations based on the pumping record (especially if pumping rates were variable).

50. Section 5.4.1 Pumping Test Procedures, page 41, last bullet

The text states water generated by the pumping tests will be discharged at the Groundwater Treatment Plant or into the Industrial Wastewater Line. Criteria for selection of the disposal point should be included in the text or a reference should be provided.

51. Section 5.4.1 Pumping Test Procedures, page 42, paragraph 1

The text states data from the data logger will be downloaded to the computer. The data should be reviewed throughout and immediately following the test. If the quality of data is not sufficient to meet the DQOs, then the test should be repeated or an alternate test should be considered. This comment also applies to Section 5.4.2 Slug Test Procedures (page 42, paragraph 5).

52. Section 5.4.1 Pumping Test Procedures, page 42, paragraphs 2 and 3

The text indicates that a pumping test is continued for a minimum of 4 hours. The criteria for determining the maximum duration of a test (i.e., when pumping is shut off) should be described. The pumping should continue as practical until DQOs for the test are satisfied, including sufficient data for analysis.

53. Section 5.4.1 Pumping Test Procedures, page 42, paragraph 2

The text also references instrumentation that will be used to collect and store data from the pumping tests. This instrumentation should be capable of measuring parameters at levels of accuracy and with the precision necessary to meet the DQOs. Examples of these tolerances for aquifer test instrumentation are discussed in Stallman (1971).

54. Section 5.4.1 Pumping Test Procedures, page 42, paragraph 3

The text describes procedures for setting the depth of pressure transducers. The text should also state that the depth of the transducer should be measured before and after the pumping test. The text should also include procedures for selection of a pressure transducer. Because accuracy of a pressure transducer typically varies with the operating range of the transducer (e.g., 0 to 10 pounds per square inch), selection of the

transducer is dependent upon the desired accuracy of the measurements. A discussion should be provided of the desired accuracy of the pressure measurements and what procedures will be followed to ensure that measurements attain that accuracy. The required precision of the readings and the precision of the pressure transducer should also be included. A discussion of the difference between vented and unvented transducer data and interpretation of these data should be provided. In addition, the text should discuss how measurements are to be corrected for variations in barometric pressure (especially if tests are of long duration, such as overnight).

55. Figure 5-13 page 43

The pumping test form does not contain all the information necessary to properly document the performance of an aquifer test. The serial numbers and operating ranges of pressure transducers should be noted. The serial number and description of the data logger also should be included. The serial number of each electrical water level measuring tape should be shown. In addition, specific information that will be included on this form should be described in the second paragraph of page 42 in a manner similar to that for geophysical logging (e.g., page 3 of Section 5.0).

56. Section 5.4.1 Pumping Test Procedures, page 44, paragraphs 3 and 5

The text describes procedures that will be followed to ensure that the data logger is properly operating. The text should also describe procedures to ensure that the pressure transducers are properly calibrated.

57. Section 5.4.1 Pumping Test Procedures, page 44, paragraph 5

The text states that the recovery is "monitored for the specific amount of time." The text should specify what is meant by "specific amount of time." As the well is recovering, the data should be reviewed to determine if sufficient late time data have been collected to conduct permeability calculations.

58. Section 5.4.1 Pumping Test Procedures, page 45, top of page

Procedures for archiving the test data in the project's database should be provided here or included in Section 9.0 of the QAPP.

59. Section 5.4.2 Slug Test Procedures (page 45)

It should be noted the slug bomb should be lowered to below the elevation of the static water level; otherwise, part of the bomb may be above the water table when water levels equilibrate. If this occurs, the volume of the slug removed will be calculated incorrectly.

60. Section 5.5 Sample Storage and Preservation, and Handling (page 46, paragraph 1)

The text refers to EPA-recommended procedures and specifications related to sample container pretreatment, treatment, and decontamination. The source(s) of the EPA-recommended procedures should be specifically cited in the text.

Little detail is provided for the activities discussed in this section. An explanation should be included of how samples will be logged and labeled (e.g., how station locations will be documented), how samples will be stored in the field, what chain-of-custody procedures will be followed, and what sample shipping procedures will be used. Although some of these subjects are covered in other sections of the QAPP (or perhaps in other associated documents), these sections and documents should at least be referenced and briefly summarized here.

61. Section 5.6 Groundwater Sampling (page 54, paragraph 1)

The text notes that field QA/QC samples will be incorporated into the sampling schedule. This schedule, along with descriptions of the sampling procedures, should be included or referenced in the text.

62. Section 5.6.1.3 Sampling Vehicle, page 69, paragraphs 1, 2, and 3

The text should specify how sample unit and purge pump placement procedures will be documented (e.g., recorded on a field form or in the field notebook).

63. Section 5.6.1.3 Sampling Vehicle (page 72, paragraph 2)

The text discusses procedures that will be followed to determine when a well is sufficiently purged. These procedures should also include measurement of pH, conductivity, and temperature, as described in the third paragraph of page 78. It is not clear that pH, conductivity, and temperature will be monitored while purging with the Packer assembly. Monitoring these parameters is recommended because stabilization of these

parameters is important for determining if a sample should be taken and if the well has been purged adequately.

64. Section 5.6.2 Equipment Decontamination Procedures, page 73, paragraphs 1 and 2

Equipment decontamination procedures should also include collection of periodic equipment blanks to check the effectiveness of the decontamination program. These blanks should be included or referenced in the procedures along with a schedule for their collection. It should be clarified that the procedures listed for decontamination are for nondedicated equipment only. It is recommended during decontamination all equipment be given a final rinse with ASTM type II reagent water to wash away any methanol or cyclohexane residue. In addition, if "equipment used to collect water samples for organic analysis is not allowed to come into contact with plastic," then a substitute for plastic should be listed. It is recommended that, at a minimum, clean water be pumped through the interior of the nondedicated submersible pump and other pumps prior to sampling at the next well.

65. Section 5.6.3 Field Procedures (page 73, last paragraph)

Radioactive waste has been reported at McClellan AFB. Groundwater sampling equipment could include instrumentation for measuring radioactivity at sites where radioactivity may be present and should be discussed as appropriate in this section.

66. Figure 5-20 (page 76)

It is recommended that this or some other form have a place to report the thickness of free product.

67. Section 5.7 Surface Water Sampling (page 84, paragraph 1)

The text for this section should include 1) a description of methods and criteria that will be used to determine station positioning and the accuracy of the station position, 2) a discussion of methods that will be used to prevent cross-contamination between sampling stations (e.g., equipment decontamination procedures, sampling from downstream to upstream locations, and sampling upstream of personnel), and 3) a description of personal protection that will be required during the sampling effort and criteria for determining personal protection levels (alternatively, the text can refer to the health and safety plan or SAP for personal protection information).

68. Section 5.7 Surface Water Sampling (page 84, paragraph 4)

The text states that requirements for blanks and duplicates are presented in Section 11.0. The reviewed copy of the QAPP did not contain that discussion. Other QA/QC samples (e.g., trip blanks) may also be required. A discussion of the field QA/QC program for groundwater, surface water, and soil should be included in Section 11.0.

69. Section 5.8 Soil Samples (general comment)

The description of compositing methods to be used in the lab and in the field lack sufficient detail to make the procedures repeatable and comparable. For example, for the laboratory samples, there is no description of how samples will be mixed and split. For the field methods, it is stated that "approximately equal amounts of soil from each location will be included in the composite." It is recommended that procedures be presented to make sure that equal amounts of soil from each sample be composited instead of "approximately equal" amounts.

70. Section 5.8 Soil Samples (page 93, item #5; page 94, item #4)

The text indicates a sleeve is placed in a plastic bag if the caps have the potential to fall off. If the caps fall off, volatile compounds will escape from the sample and the analysis of the sample will not be accurate. Alternative procedures, such as securing the caps to the sleeve with tape, should be proposed to ensure that the DQO is met. Also, standard procedures for taking field head space readings should be discussed in this section as appropriate.

71. Section 5.9 Sediment Sampling (page 98, paragraph 2)

The text should specify the criteria for accepting (or rejecting) a sample and the individual (e.g., field leader) who will be authorized to make the determination. Sample equipment decontamination should also be discussed.

72. Section 5.10 Soil Gas Sampling (pages 100-136)

The introduction to Section 5.10 states the selection of the optimum soil gas sampling technique will depend on the operation being performed and the intended use of the analytical data. In general, the description of the soil gas collection methods does not include the criteria used for selecting a particular collection method. The selection of the appropriate collection method should be based on the DQOs of the soil gas investigation.

The text should refer the reader to the appropriate section of the work plan or SAP for a description of the selection process.

Section 5.10 should refer the reader to a description of the methods for locating the soil gas sampling stations. The text should identify the relative accuracies of the surveyed locations. It should also emphasize the importance of documenting the location of the sampling station and how deviations from the planned locations due to field conditions will be accurately documented.

In addition, the text should describe how soil gas samples will be purged or pumped when collecting canister samples; methods and materials for backfilling boreholes should be specified for every sampling effort. Additional discussion is provided in the General Comments section.

73. Section 5.10 Soil Gas Sampling (page 100, paragraph 3)

The text states surface screening will be performed in a grid pattern. The text should provide information or refer to another section of the QAPP for a description of the surveying methods and grid spacing selection methods. In addition, the text should define the approximate distance for collecting the upwind sample.

74. Section 5.10.2 Shallow Soil Gas Sampling Methods (pages 110-118)

The SOP for each of the methods includes the initiation of the field data sheet/chain-of-custody form. The text should refer the reader to the appropriate section of the QAPP, which contains a description of these forms and the procedures for their completion.

75. Section 5.10.2 Shallow Soil Gas Sampling Methods (page 102, paragraph 1)

The text should include a description of the methods that will be used to prevent clogging of the gas sampling tube by soil particles.

With the exception of the description for the pneumatic method, the other shallow soil gas collection methods do not state that a digging permit is required. An explanation of the procedure to obtain a digging permit should be provided. Presumably a digging permit refers to a utilities check to ensure that a utility is not encountered as a result of the soil gas collection process. All the shallow soil gas collection methods should include obtaining a utilities clearance.

76. Section 5.10.2 Shallow Soil Gas Sampling Methods (page 104, paragraph 1)

The description of the hand auger method should include the anticipated range of penetration.

77. Section 5.10.2 Shallow Soil Gas Sampling Methods (page 104 item #2)

The text should include a description of the methods that will be used to prevent clogging of the sampling tube by soil particles and a description of the method that will be used for withdrawing the sample probe following sample collection. Sample tubing should be discarded between samples/stations. The text should describe methods for sealing the tube in the borehole to ensure collection of an adequate borehole sample that is not impacted by atmospheric air flooding into the borehole. In addition, the method that will be used to backfill the hole should be specified.

78. Section 5.10.2 Shallow Soil Gas Sampling Methods (page 105)

The descriptions of equipment decontamination vary from soap-and-water wash to steam-cleaning to no apparent decontamination of sampling between stations. All equipment that comes into contact with the soil and soil gas should either be decontaminated between sampling stations or disposed of after use. In addition, the text should provide a description of the decontamination and disposal procedures for the glass syringes and septa, respectively. These procedures should be included or referenced; as written, procedures are described only for pneumatic and hydraulic methods.

79. Section 5.10.2 Shallow Soil Gas Sampling Methods (page 106, item #3)

The text should include a description of the probe extraction technique.

80. Figure 5-28 (page 107)

The figure should include specification of the probe tip material that will remain in the soil following extraction of the sample probe.

81. Section 5.10.2 Shallow Soil Gas Sampling Methods (page 108, item #9)

The text should include a description of the sample probe extraction technique.

82. Section 5.10.3 Deep Soil Gas Sampling Methods (page 121, items #B4 and C1d)

The text states that 3 to 5 volumes of soil gas will be purged prior to sampling. The text should provide the calculations for determining tube volumes by the field team.

The text should state that the soil gas samples will be delivered to the laboratory under chain-of-custody procedures and should refer the reader to the appropriate section of the planning documents for a description of those procedures.

83. Section 5.10.3 Deep Soil Gas Sampling Methods (page 122, item #4)

The text should state how frequently the septa will be replaced.

84. Section 5.10.3 Split Spoon Sampling (page 123)

Due to the impacts that wind, rain, and heat can have on organic vapor meter (OVM) readings and because the OVM readings will be used extensively to select soil samples for analysis, it is recommended that a more standard and rigorous method for obtaining soil gas OVM readings be developed. The procedures could include slipping a cap with a hole in it over the end of the sleeve and inserting the OVM tip into the hole to take the reading or taking headspace readings in a plastic bag or a glass jar. Dependable Data When You Need It (U.S. EPA 1990) provides other methods for taking standardized OVM readings.

85. Section 5.10.4 Emission Flux Measurements (page 130, items #2 and #4)

The text states that steady-state emission rates will be recorded. The text should provide criteria to allow the field scientist to determine when a steady state has been achieved.,

86. Section 5.10.4 Downhole Emission Flux Measurement (DHEF) (page 132)

A figure showing the DHEF sampling apparatus is recommended.

87. Section 5.10.4 Downhole Emission Flux Measurements (page 133, item #6)

The text states that the DHEF chamber will be decontaminated if there is visible contamination. However, the sampling chamber may contain contaminants at levels that are sufficient to cause

cross-contamination but that are not visible in the field. The chamber should be decontaminated after use at each sampling station.

88. Section 5.10.5 Recordkeeping (page 135, paragraph 1)

The text lists information that will be recorded on the daily log during the soil gas investigation. The field personnel should also record the air temperature at the surface of the soil at each sampling station. This information will be useful during the analysis of soil gas flux estimates.

89. Section 6.0 Sample Custody (page 1, paragraph 1)

A discussion should be provided of activities that will be implemented to maintain proper chain-of-custody (e.g., physical possession of sample, sample storage in a secured area, etc.).

90. Section 6.1 Documentation Procedures (page 1, paragraph 3)

The procedures, forms, and notebooks used to determine and record the exact location and ambient conditions associated with sample collection, possession, and analysis should be discussed in further detail in this section, or reference should be made to other documents that contain this information. For example, a checklist should be provided containing general information that should be collected for every sampling site (e.g., location, ambient meteorological conditions).

The text should specify where documents relating to sample control (e.g., chain-of-custody forms and field notebooks) will be kept in the field and who is responsible for maintaining such documents.

91. Section 6.1 Documentation Procedures (page 3, paragraph 1)

The text should specify procedures for completing forms and labels. For example, a discussion should be included of how sample identification numbers will be assigned and tracked.

92. Section 6.2 Electronically Generated Chain-of-Custody and Master Log (page 3, paragraphs 1 and 2)

This section should include a discussion of the procedures that will be implemented for checking the accuracy of information entered into the field computer system and for ensuring security of the data once entered.

It is not obvious in the text whether the electronically generated daily chain-of-custody forms are in addition to manually prepared chain-of-custody forms generated in the field. Manually prepared chain-of-custody forms should be generated when samples are transported, not just at the end of each day.

93. Section 6.2 Electronically Generated Chain-of-Custody and Master Log (page 8, paragraph 1)

In order to maintain a proper chain-of-custody and ensure traceability of data, the text should specify who will be responsible for shipping samples and maintaining shipping records.

94. Section 6.3 Sample Handling Procedures (page 8, paragraphs 3 and 4)

If samples must be stored at 4°C, they should be packaged so that the labels do not become waterlogged and paper seals do not come loose. In addition, the ice should be contained in plastic bags to prevent leakage. Ice should not be used as a packing material. The text should be modified to reflect these concerns.

The text does not make clear what sample handling control procedures will be followed by the laboratory to maintain the chain-of-custody and ensure the integrity of incoming samples. For example, this section should specify there will be a designated laboratory sample custodian who is authorized to sign for incoming samples, obtain shipping documents, verify the data and sample custody records, and determine integrity of incoming samples. If this information is covered in another section or in another document, it should at least be referenced.

A discussion of laboratory storage of samples should be included in this section or perhaps in Section 5.5. For example, samples for analysis of volatile organic compounds should be stored separately from other samples. Other documents such as the SAP for the project may be referenced for this information.

A listing of which laboratories will be used for each type of analysis should be included in the final document or referenced as being included in an associated document, such as the SAP.

95. Section 6.3 Sample Handling Procedures (pages 10)

A refrigerator blank is mentioned in the last bullet of page 10. The method and schedule for taking refrigerator blanks should be described.

96. Section 7.3 Field Instrument Calibration (page 1, last paragraph)

This section details field equipment that will require calibration. Field equipment that should also be listed in this section includes pressure transducers, flow meters, and electrical water-level measuring tapes.

97. Section 7.3 Real-Time Portable Organic Vapor Analyzers (OVA) (page 2, paragraph 3)

In paragraph 3, the corrective action for an OVA instrument showing excessive drift is to flag the suspect data. Because sampling and analysis decisions will be made on the basis of these readings, it is recommended that in addition to flagging the data, the OVA be repaired or replaced with a new instrument as soon as possible. Extra OVAs should be kept onsite just for these types of situations.

98. Section 8.0 Analytical Procedures and Calibration

Pages 7 and 8 of U.S. EPA (1989) include a format for presenting the analytical and quality control procedures for each field or laboratory measurement or for each group of analytes to be measured by a single analytical method. This format should be followed for clarity. Most of the items are discussed somewhere in the QAPP, but three important items should be included or referenced in this section: preventive maintenance, data calculations and reporting units, and documentation and deliverables.

The tables which present method detection limits and quantitation limits are confusing. In the text Radian's laboratory's detection limits are presented in Tables 8-3 to 8-16. However, the QAPP indicates Brown and Caldwell Analytical (BCA) will be performing some, if not all the analytical work. BCA's reporting limits are provided in Tables A-13 to A-26. The limits presented by the two organizations do not agree. Since corrective action in some cases (e.g. as a result of blank contamination) is dependent on the defined detection limits, it is important to have clearly defined limits. Further, it is not clear whether the limits in the tables have been evaluated with respect to the needs of the project. The QAPP should be revised and a single consistent set of reporting limits provided which reflect the needs of the project.

99. Section 8.0 analytical Procedures and Calibration, SW8015 Modified, Total Petroleum Hydrocarbons, Luft Field Manual 7/87.

The April 1989 LUFT Field Manual should be referenced.

100. Section 8.0 Analytical Procedures and Calibration, SW8240, SW 8270.

No discussion is presented in the text concerning internal standard calibration, internal standard areas, and associated criteria, although this information is represented in the summary tables. A brief discussion should be added to the text in this area.

101. Section 8.4 Real-Time Portable Field Meters (pages 31-35)

Because multiple types of OVAs and OVMs are proposed for the same end use (i.e., to take similar types of organic vapor readings and make decisions on where soil samples will be collected), it is recommended that procedures be developed to assess the comparability of readings between the instruments.

102. Section 8.6 Analytical Methods for Physical Properties (pages 35-37)

There is no discussion in the QAPP on the QA/QC measures that will be taken to ensure that the quality of the physical testing results meet DQOs. There is no methodology presented for determining porosity or volumetric moisture content. These parameters may be important for assessing the fate and transport of contaminants in both the vadose and saturated zones. These points should be addressed.

103. Section 9.0 Data Reduction, Validation and Reporting (pages 1-8)

It is recommended that the QAPP contain an outline of the project data management scheme that traces the path of the data management tasks, including field information; receipt of data from the field or laboratory; validation and compilation of the data; and use or storage of the final reported data.

This section, or a related section, should include a discussion of how field sampling documentation is tracked with the sample and analytical data. Also, the method by which field data are validated should be discussed. In both cases, other sections of the QAPP or other documents may be referenced, if appropriate.

This section or a related section should include a discussion of procedures for checking data from other (non-Radian Corporation) laboratories.

104. Section 9.1 Data Reduction (page 1, paragraph 2)

A discussion of the percentage of data which will be reviewed and the procedures the laboratory manager will use to determine whether analyses were properly performed and reported should be provided.

105. Section 9.2 Data Transfer and Verification

A discussion should be included of how data received via electronic formats from subcontracting laboratories will be cross-checked against hard copy documents. Will the Radian SAM system be used to report all data including data from BCA or will it be used for only Radian data? If the discussion applies only to Radian, information should be added on BCA.

The text should also clarify whether these are validated, laboratory, or original data. If the data are validated by the subcontract laboratory, a discussion of the validation procedures should be included in this section.

106. Section 9.3 Data Validation (page 4, paragraphs 2 and 3)

It is not clear whether this section is referring to the program QA/QC coordinator or another person, such as the laboratory quality assurance coordinator. The text should clarify this point.

If the analyses are performed by a subcontract laboratory, the text does not specify whether information such as laboratory control charts will be provided. It is also not clear in the discussion whether the data will be validated by the laboratory or the project quality control coordinator. These points should be addressed.

107. Section 9.4 Reporting (page 5, paragraph 2)

An explanation should be provided of how data that are entered into the Installation Restoration Program Information Management System (IRPIMS) will be checked for accuracy and errors. Furthermore, there should be a discussion on how both electronic data and hard copy documents will be backed up.

108. Section 9.4 Reporting (page 6, bullet)

The text should make clear what percentage of calculations and measurements will be checked for errors. Also, a reference should be made to any corrective action procedures that must be followed, such as those provided in Section 15.0 of the QAPP. It

is recommended that an example calculation sheet be included in this section.

109. Section 10.0 Internal Quality Control; Tables 10-1 to 10-9, Summary of Calibration and Internal Quality Control Procedures

It is recommended if surrogate recoveries are not acceptable the sample be reextracted and reanalyzed. Reference to laboratory control sample results does not provide any information on whether a matrix problem or an extraction problem was responsible for the surrogates being out. This is especially true for the gas chromatography (GC) methods where only one or two surrogates are being used.

The use of matrix spike sample as an ongoing calibration check is not recommended. First, calibration standards normally do not go through the sample preparation steps like a matrix spike sample, so the results are not comparable. Second, matrix spike sample recoveries can be affected by the sample matrix which again limits their comparability. The QAPP also does not indicate the continuing and ongoing calibration checks for GC are to be conducted with fewer than the analyte list. If a subset of the original list will be used for the GC methods, as it already is for GC/MS analyses, this should be indicated in the QAPP. Finally matrix spikes are routinely prepared from a second source, while ongoing calibration check sample is normally one of the original calibration solutions. The system for doing ongoing calibration checks needs to be reevaluated.

110. Section 10.1 Analytical Laboratory Quality Control Samples (pages 1-81)

The text does not make clear whether the same quality control procedures will apply to all laboratories, specifically whether similar quality assurance programs are in place in the non-Radian Corporation laboratories. This should be clarified.

111. Section 11.0 Performance and Systems Audits

It is recommended at least one technical systems audit of the subcontractor laboratory be conducted early in the project after a representative number of samples for different analyses have been received. This audit could be conducted by Radian or the Air Force. The QAPP already indicates BCA has been selected as the subcontractor. This would suggest no other technical audits are presently planned to ensure the laboratory is complying with the requirements of the QAPP.

112. Section 11.0 Performance and Systems Audits (page 1, paragraph 3)

The text does not specify when or what custom performance evaluation audit samples will be submitted and analyzed for the sampling and analysis programs. If this information will be included in the SAP, then that document should be referenced. If not presented in the SAP, it should be presented in the QAPP.

113. Section 11.1 Performance and Systems Audits (page 3, paragraph 3)

The type of data quality audits that will be performed for future air sampling should be discussed in this section.

114. Section 11.1 Technical Systems Audit

On the VOC Sampling Systems Audit Checklist for Canister Sampling (Figure 11-2), there should be a provision for checking that the cleaning procedure for the canister is appropriate and that appropriate quality control procedures have been followed.

115. Section 12.2 Maintenance Schedules (page 2, paragraph 2)

A discussion of the schedule for preventive maintenance for laboratory instruments should be included in this or a related section because such a schedule should already be in place. At a minimum, a basic timetable should be included here, or reference should be made to a laboratory quality assurance plan.

116. Section 13.2 Accuracy: Section 15.1, Quality Assurance and Quality Control Reporting Format

These sections contain discussions or references to method spikes, which represent QC samples for which no criteria are presented in Section 4 on Quality Assurance Objectives and no discussion is presented in Section 10.0 on Internal Quality Control. This inconsistency should be resolved.

117. Appendix A

Descriptions of the quality assurance programs in place at the laboratories should be included in this appendix

## REFERENCES


Dobrin, M.B. 1976. Introduction to geophysical prospecting. McGraw-Hill, Inc.

Radian. 1991a. Installation restoration program (IRP) stage 3 quality assurance project plan (draft for McClellan AFB/EM, McClellan AFB, California 95652-5990. Prepared for the U.S. Air Force Center for Environmental Excellence. Radian Corporation, Sacramento, CA.

Radian. 1991b. Installation restoration program (IRP) stage 7 operable unit B remedial investigation sampling and analysis plan (draft final) for McClellan AFB/EM, McClellan AFB, California, 95652-5990. Prepared for the U.S. Air Force Center for Environmental Excellence. Radian Corporation, Sacramento, CA.

Stallman, R.W. 1971. Techniques of water-resources investigations of the United States Geological Survey. Chapter B1: Aquifer-test design, observation and data analysis. U.S. Department of Interior, Washington, DC.

U.S. EPA. 1980. Interim guidance and specifications for preparing quality assurance project plans. U.S. Environmental Protection Agency, Washington, DC.

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U.S. EPA. 1991a. Soil sampling and analysis for volatile organic compounds. Ground-Water Issue. EPA 540/4-91/001. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, DC.

U.S. EPA. 1991b. Management of investigation-derived wastes during site inspections. EPA/540/G-91-009. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC.

# RESPONSE TO EPA REGION IX COMMENTS ON DRAFT QAPP

## GENERAL COMMENTS

### 1. Project Approach and Objectives

Data quality objectives are addressed and presented in the SAP for a project; a general description of the use of DQOs to select the appropriate sampling and analytical procedures is presented in Section 1.0, page 2; and in Section 4.0. DQOs are also referenced in the descriptions of specific field and analytical procedures in Sections 5.0 and 8.0. These are references to the type of DQO the procedure will fulfill, not site-specific DQOs.

Analytical levels have been specified and are described in Section 4.0. The quality assurance objectives in Tables 4-1 through 4-13 and the analytical procedures and reporting limits presented in Section 8.0 are based on the levels as defined for the projects. The investigation approach described in the Operation Unit (OUB) B SAP is applicable to that field program. Because the QAPP is intended for use as a reference document for all field programs performed by Radian at McClellan AFB, the specific OUB information has not been added.

### 2. Insufficient Detail

Detail has been added to QAPP sections following the recommendations in the specific comments provided by EPA. References to other Sections or documents have also been added to ensure that all aspects of field work, documentation, and analysis are addressed and the requirements are clearly presented for field staff using the QAPP as a reference.

### 3. Relationship of the QAPP to Other Documents

A description of the relationship between the QAPP and other project documents has been added to Section 1.0. Consistent reference to site-specific SAPs or other documents has been incorporated.

**4. Quality Assurance for Subcontractors**

Additional detail has been added to Section 1.0 to describe subcontractor compliance requirements. Specifications for subcontractors will be described in detail in each subcontractor scope of work and SOP. The SOPs will be reviewed by project staff to ensure that project objectives can be satisfied.

**5. Revisions to the QAPP**

Procedures for issuing revisions have been added to Section 1.0. A schedule for revisions (i.e., annual) has not been established. Agency representatives will be notified when a revision is planned.

**6. DQOs**

Geologic DQOs have been addressed in more detail, in Section 4.6, following the recommendations and the specific comments provided by EPA.

**7. Acceptable Tolerances for Hydrological Measurements**

Hydrological measurement tolerances are discussed in more detail in Section 4.7, as recommended.

**8. Optional Testing Procedures**

This QAPP was developed for use as a procedural reference for all field projects conducted by Radian at McClellan AFB. This approach was discussed and approved by agency and Air Force representatives in late 1987 and early 1988 when it was first developed. Because this QAPP is not site-specific, the selection process and rationale for use for procedures are presented in the site-or OU-specific SAP. Some detail has been added or was already present in well drilling and field sampling procedures (Sections 5.2 through 5.10), describing applicable conditions for use of the procedure.

## 9. Decontamination Procedures

The description of decontamination procedures has been clarified. Procedures for groundwater sampling equipment are referenced to Section 5.6.2, and soil and soil gas equipment decontamination procedures are described at the beginning of Sections 5.8 and 5.10, respectively. These descriptions have been maintained within each general procedure for easier access by the field teams.

## 10. Loss of Volatile Constituents

Use of methanol to minimize loss of volatile analytes was considered for use during the OUB RI, but was not selected because of concerns about handling large numbers of samples, solvents, and vials in the field. The logistics for sample weighing and solvent handling during sonic drilling operations were determined to be unworkable.

## 11. Sampling and Purging Wells

Free product has not been observed during well sampling activities conducted since 1985. As described in Section 5.6.13, a water level meter capable of measuring hydrocarbon layers is available for use if needed. If anticipated for future work, a procedure for sampling wells with a floating hydrocarbon layer will be included in a revision of the QAPP.

## 12. Redundancy Among Sections

Redundancy has been reduced for decontamination procedures and field sampling procedures. The groundwater sampling procedures presented in Section 5.6 have been reorganized and a clear distinction between the discrete interval sampler system (DIS) and other groundwater sampling procedures is made. The DIS system operation is now presented in Section 5.6.4. The rationale for differences in well purging volumes and measurements are described. A demonstration and comparability study were conducted and the system and its operation were approved for use by agency representatives. Soil gas sampling procedures (Section 5.10) have been consolidated to reduce redundancy.

## 13. Soil Gas Sampling

Soil gas sampling procedures have been consolidated to reduce redundancy and more clearly reflect consistency between procedures. The "open hole" methods were retained because they have been used in past investigations, and current reference is needed for data users. Current methods are the shallow and downhole probe methods, which produce very similar samples, the primary difference being the method used to drive the probes to the desired depths. The sample collection procedures for each type of equipment are detailed in each subsection because the section is used as an SOP. A description of how QC sample requirements will be communicated to field staff has been added at the beginning of Section 5.10, and analytical levels have been incorporated.

## 14. Mobile and Stationary Laboratory QC Measures

The QC procedures for mobile and stationary laboratories are presented by analytical level in Section 8.0. The SOPs for mobile laboratories have been included in Appendix A. Specific calibration, QC procedures and acceptance criteria, and corrective actions for Level 3 analyses are presented in the detailed tables in Section 10.0.

## 15. Internal Quality Control

The frequency requirements for field and laboratory QC samples are presented clearly in Section 10.0. For stationary laboratories, these frequency requirements follow method specifications, with additional Air Force requirements. These are detailed in the tables in Section 10.0. The field QC sample requirements follow the Air Force IRP guidance, and are usually designated in the project SOW. As described in the Section 5.0, Field Procedures, and in Section 10.2, QC samples are assigned by qualified QC staff at the beginning of each sampling event, and included in field task instructions.

**16. Consistency with Guidance**

This Draft QAPP was an update of an existing and approved document. Initial planning and development, including the format, was conducted during early 1988, prior to issuance of the EPA Region IX guidelines. Therefore, major format changes were not made.

SPECIFIC COMMENTS

**17. Table of Contents, Page iii**

The Table of Contents has been corrected.

**18. Section 1.0, Introduction, Page 1, Paragraph 1**

The distribution list has been added before the Table of Contents.

**19. Section 1.0, Introduction, Page 1, Paragraph 1.**

The guidance reference has been updated.

**20. Section 2.3, Data Collection and Use, Page 11, Paragraphs 1 and 3.**

There are no currently anticipated plans to collect air samples, so the reference has been deleted. Operable Unit B RI gas phase sampling consists of shallow and downhole soil gas samples.

**21. Section 3.5, Primary Task Leaders, Page 4, Paragraph 1.**

The project QA officer has primary responsibility for coordinating and tracking corrective action. The description of responsibilities in Section 3.2 has been revised to reflect this.

**22. Section 4.0, Quality Assurance Objectives for Measurements and Geologic Data, Tables 4-1 through 4-12, and Tables A1 through A12.**

As recommended by EPA, the approach to QA objectives has been changed to have one consistent set of precision and accuracy criteria for each method, and all data produced by subcontracted laboratories will be evaluated using these criteria. Section 4.0 text has been revised to describe the approach used to establish project QA objectives, and Tables 4-1 through 4-13 present the QA objectives by analytical level and method.

Project DQOs are developed during the site-specific planning process, and are described in the SAP. The DQO's for individual sites or field projects are not included in the QAPP because it is intended as a procedures document for multiple field projects.

**23. Geologic DQOs, Page 20, Paragraph 2**

Text for Section 4.6 has been revised to state that personnel performing geologic logging will review existing cores. Additional detail about field tests and standard forms, and training has been added.

**24. Section 4.6, Hydrologic Data, Page 20, Paragraph 3**

This discussion has been placed in a separate subsection, 4.7. Additional text detail has been added for hydrologic measurements.

**25. Section 5.0, Field Procedures, Pages 1-136, General Comment**

Reference to the SAPs has been added to the beginning of Section 5.0 and each subsection describing field and sample collection procedures.

**26. Section 5.1, Geophysical Techniques, Page 1, Paragraph 5**

The requested detail about sampling sites, surveys, and selection of appropriate procedures has been added to Section 5.1, Page 2, Paragraph 1.

**27. Table 5-1, Water and Soil Sample Storage and Preservation Requirements**

Table 5-1 has been revised to show consistent and complete information.

**28. Section 5.1.1, Borehole Geophysical Investigation Procedures, Page 2, Paragraph 2**

A description of the applicability and criteria for selection of the natural gamma logging technique has been incorporated in Section 5.1.1.2, Page 4, Paragraph 3; and Page 5, Paragraph 1. Data storage procedures have been added to Section 5.1.1, Page 3, Paragraph 1; and Section 5.1.2, Page 6, Paragraph 2.

**29. Section 5.1.2.2, Magnetometry, Page 6, Paragraph 3**

This information has been added to the text in Section 5.1.2.2, Page 7, Paragraph 2.

**30. Section 5.2.1, Site Selection, Page 6, Paragraph 5**

Consideration of receptors has been added to the list of criteria used for the site selection in Section 5.2.1, Page 8, Paragraph 1.

**31. Section 5.2.1, Site Selection, Page 7, Paragraph 2**

A description of equipment and procedures used to clear sites prior to drilling is included in Section 5.2.1, Page 8, Paragraph 2.

**32. Section 5.2.2, Drilling Preparation, Page 8, Paragraph 1**

Soil and cuttings management procedures are presented in Section 5.2.5. References to the section have been added to Section 5.2.2, Page 8, Paragraph 5; and Section 5.2.3, Page 10, Paragraph 4.

**33. Section 5.2.3, Drilling Methods, Page 9, Paragraph 2**

Conditions or criteria for use of drilling methods have been added to Section 5.2.3, Page 11, Paragraph 1 for Hollow Stem Auger; Page 11, Paragraph 2 for Mud Rotary; Page 12, Paragraph 1 for Air Rotary Casing Drive; Page 13, Paragraph 2 for Dual Tube Percussion; Page 15, Paragraph 2 for Bucket Auger; and Page 15, Paragraph 3 for Sonication.

**34. Section 5.2.3, Drilling Methods, Page 11, Paragraph 1**

Reference to Section 5.2.5 has been added to the mud rotary method description in Section 5.2.3, Page 11, Paragraph 2.

**35. Section 5.2.3, Drilling Methods, Page 12, Paragraph 3**

Reference to Section 5.2.5 has been added to the Bucket Auger method described in Page 15, Paragraph 2.

**36. Section 5.2.4, Sample Collection Methods, Page 16, Paragraph 3**

Core identification information has been added to Section 5.2.4, Page 18, Paragraph 3.

**37. Section 5.2.5, Cuttings and Groundwater Disposal, Page 17, Paragraph 3**

A description of the monitoring frequency and documentation for organic vapor measurements has been added to Section 5.2.5, Page 19, Paragraph 3.

**38. Figure 5-3, Page 19**

This information is included in the drilling notes section of the form.

**39. Figure 5-4, Page 20**

The measuring point is notched into the top north side of the well casing at the time of well completion, as described in Section 5.3.1, Page 40, Paragraph 3. This information will be noted on the well completion form.

**40. Section 5.2.6, Recordkeeping, Page 28, Paragraph 2.**

The reference to lithologic sample collection intervals has been revised in section 5.2.6, Page 30, Paragraph 2.

**41. Section 5.2.6, Recordkeeping, Page 29, Second Bullet.**

The third bullet on Page 31 describes screened intervals that intersect the water table.

**42. Section 5.3.1, Well Installation and Completion, Page 33, First Line and First Bullet.**

The text has been revised to indicate that centralizers will be set at the bottom and top of the well screen on Page 35, second bullet.

**43. Section 5.3.1, Well Installation and Completion, Page 35, Paragraph 2**

The text has been revised as recommended for monitoring the top of the filter pack and preventing raising the drill string above the level of the filter pack in Section 5.3.1, Page 37, Paragraph 3, and Page 38, Paragraph 1.

**44. Section 5.3.1, Well Installation and Completion, Page 35, Paragraph 3.**

The text has been revised on Page 38, Paragraph 1.

**45. Section 5.3.1, Well Installation and Completion, Page 35, Paragraph 4**

The weight of the slurry is not important as long as the mixture is correct and the amounts of sand and bentonite added meet or exceed the estimates based on the borehole annulus.

**46. Section 5.3.1, Well Installation and Completion, Page 36, Paragraph 5**

Reference to measuring ground surface elevation has been added to Section 5.3.1, Page 40, Paragraph 4. The surveying occurs after well completion, therefore this information is recorded by the surveyor and provided to project staff at the time of survey rather than on the drilling operations form.

**47. Section 5.3.1, Well Installation and Completion, Page 36, Paragraphs 4 and 5.**

The text has been revised as recommended by EPA. See Page 40, Paragraphs 3 and 4. Figure 5-4 has not been revised because the notch would be insignificant relative to the scale of the figure.

**48. Section 5.3.2, Monitoring Well Development, Page 41, Paragraph 1.**

The stabilization criteria for pH, temperature, and conductivity have been added to Section 5.3.2, Page 44, Paragraph 1.

**49. Section 5.4, Aquifer Test Methods, Page 41, Paragraph 2**

Reference to also measuring water level recovery has been added to Section 5.4, Page 44, Paragraph 2.

**50. Section 5.4.1, Pumping Test Procedures, Page 41, Last Bullet**

The criteria for selecting the discharge point have been added to Section 5.4.1, Page 45, first bullet.

**51. Section 5.4.1, Pumping Test Procedures, Page 42, Paragraph 1.**

The text has been revised to indicate that data review occurs during the tests in Section 5.4.1, Page 45, Paragraph 3, and Page 49, Paragraph 3.

**52. Section 5.4.1, Pumping Test Procedures, Page 42, Paragraphs 2 and 3.**

The text has been revised as recommended. See Section 5.4.1, Page 47, Paragraph 1.

**53. Section 5.4.1, Pumping Test Procedures, Page 42, Paragraph 2.**

The precision and accuracy requirements are referenced to Section 4.7. See Section 5.4.1, Page 45, Paragraph 3, and top of Page 47.

**54. Section 5.4.1, Pumping Test Procedures, Page 42, Paragraph 3.**

The requested information about transducer depth measurements and selection has been added to Section 5.4.1, Page 47, Paragraph 2. Precision and accuracy objectives for hydrologic measurements are presented in Section 4.7, and referenced in Section 5.4.1, Page 45, Paragraph 3 and the top of Page 47. Interpretation of the data occurs during test results evaluation, and use for different types of data are considered at that time.

**55. Figure 5-13, Page 43**

Figure 5-13, Page 46 has been revised to record the serial numbers or equipment numbers of equipment used.

**56. Section 5.4.1, Pumping Test Procedures, Page 44, Paragraphs 3 and 5**

The requested information has been added to Section 5.4.1, Page 47, Paragraph 3.

**57. Section 5.4.1, Pumping Test Procedures, Page 44, Paragraph 5**

The recovery monitoring time period has been specified in Section 5.4.1, Page 48, Paragraph 3.

**58. Section 5.4.1, Pumping Test Procedures, Page 45, Top of Page**

Archiving information has been added to Section 5.4.1, Page 48, Paragraph 4. General data transfer procedures are described in Section 9.0.

**59. Section 5.4.2, Slug Test Procedures, Page 45**

The slug placement description has been revised as recommended in Section 5.4.2, Page 49, Paragraph 2.

**60. Section 5.5, Sample Storage, Preservation, and Handling, Page 46, Paragraph 1**

The text has been revised to include references and some additional detail as recommended by EPA. See Section 5.5, Page 49, Paragraph 4, and Page 56, Paragraph 1.

**61. Section 5.6, Groundwater Sampling, Page 54, Paragraph 1**

Information about QC sample assignment and scheduling has been added to Page 57, Paragraph 2 and references to Sections 10.0 and 13.0 have been added.

**62. Section 5.6.1.3, Sampling Vehicle, Page 69, Paragraphs 1,2, and 3**

Documentation of sampling system placement procedures has been added to Section 5.6.4, Page 84, Paragraph 4.

**63. Section 5.6.1.3, Sampling Vehicle, Page 72, Paragraph 2**

Description and rationale for these procedures are provided in Section 5.6.4, Page 82, Paragraph 2, and Page 87, Paragraph 6 and top of Page 88.

**64. Section 5.6.2, Equipment Decontamination Procedures, Page 73, Paragraphs 1 and 2**

Schedules for equipment blank collection (along with other QC samples) are discussed in Section 5.6, Page 57, Paragraph 2, and in Section 10.0. Description of decontamination of both dedicated and nondedicated equipment is provided in Section 5.6.2, Page 71, Paragraphs 1 and 2. A final rinse and pumping with ASTM II water is impractical for use in the field. Storage of equipment used to sample for organics is discussed in Paragraph 1, Page 71.

**65. Section 5.6.3, Field Procedures, Page 73, Last Paragraph**

Reference to measuring radioactivity has been added to Section 5.6.3, Page 80, last paragraph.

**66. Figure 5-20, Page 76**

Free product has not been observed in monitoring wells at McClellan AFB since routine monitoring began in 1985. Therefore, a separate item on the data sheet is not considered to be necessary. If free product is observed, it will be noted in the comments section of the well purging data sheet, Figure 5-17.

**67. Section 5.7, Surface Water Sampling, Page 84, Paragraph 1**

The information recommended by EPA has been added to Section 5.7, Page 88, Paragraphs 4 and 5, and Page 89, Paragraph 1.

**68. Section 5.7, Surface Water Sampling, Page 84, Paragraph 4**

The initial reference to Section 11.0 was incorrect; the reference is to Section 10.0. This has been corrected in Section 5.7, Paragraph 1.

**69. Section 5.8, Soil Samples, General Comment**

The compositing procedures have been made more specific. See Section 5.8, Pages 94 and 95, Items 5 and 6, Page 98, Items 6 and 7, and Page 100, Items 8 and 9.

**70. Section 5.8, Soil Samples, Page 93, Item 5, Page 94, Item 4**

The caps are available in numerous sizes, allowing ordering sizes that will snugly fit the sleeves; the reference to the caps falling off has been deleted. Reference is added for standard FID/PID measurements described in Section 8.4 in Item 4a, Page 97, Item 5 on Page 99, and Page 103, Item 4.

**71. Section 5.9, Sediment Sampling, Page 98, Paragraph 2**

Detail and reference to the SAP has been added to Section 5.9, Page 104, Paragraph 2

**72. Section 5.10, Soil Gas Sampling, Pages 100-136**

Additional project planning details and reference to the SAP for rationale and selection of procedures has been added to Section 5.10, Page 105, Paragraphs 3,4 and 5, and the top of Page 107. Purging and backfilling requirements are added to Section 5.10.2, Page 112, Item 8; and Section 5.10.3, Page 127, Item D5.

**73. Section 5.10, Soil Gas Sampling, Page 100, Paragraph 3**

The grid spacing rationale is referenced to the SAP in Section 5.10.1, Page 107, Paragraph 2. The location for ambient measurements has been added to Page 107, Item 2.

**74. Section 5.10.2, Shallow Soil Gas Sampling Methods, Pages 110-118**

The datasheet and documentation procedures have been referenced to Section 6.0 for all sampling procedure descriptions. The detailed task instructions and training are also described in Section 5.10, bottom of Page 106, and top of page 107.

**75. Section 5.10.2, Shallow Soil Gas Sampling Methods, Page 102, Paragraph 1**

Detail regarding prevention of clogging the sampling tube has been added to Item 3, Page 111. Explanation of digging permits has been added to Section 5.10, Page 107, Paragraph 1.

**76. Section 5.10.2, Shallow Soil Gas Sampling Methods, Page 104, Paragraph 1**

Hand auger penetration has been added to Section 5.10.2, Page 109, Paragraph 4.

**77. Section 5.10.2, Shallow Soil Gas Sampling Methods, Page 104, Item 2**

Description of sampling tube placement to prevent clogging has been added to Item 3, Page 111. The slide hammer and hand auger holes do not restrict the sampling tubes. Tubing is purged between uses as described in Section 5.10.2, Page 109, Paragraph 1. Sealing of the hole is addressed on Page 111, Item 5a. The backfill requirement is addressed in Item 8, Page 112.

**78. Section 5.10.2, Shallow Soil Gas Decontamination Methods; Page 105**

The soil gas decontamination procedures for all equipment has been consolidated into the description on Page 109.

**79. Section 5.10.2, Shallow Soil Gas Sampling Methods, Page 106, Item 3**

The probe extraction technique has been revised and is Item D2 on Page 117.

**80. Figure 5-28, Page 107**

The disposable tips are steel. This has been added to the text in Section 5.10.2, Page 112, Paragraph 2, and Page 118, Paragraph 5.

**81. Section 5.10.2, Shallow Soil Gas Sampling Methods, Page 108, Item 9**

This method has been consolidated with the hand drive probe method. Probe extraction is addressed in Item D2, Page 117.

**82. Section 5.10.3 Deep Soil Gas Sampling Methods, Page 121, Items B4 and C1d**

Purge time and volume determinations have been referenced in Item B4 on Page 116, and Item B4 on Page 125. Reference to chain of custody and documentation procedures is added to Item C1d on Page 117 and Item C1d on Page 126.

**83. Section 10.3, Deep Soil Gas Sampling Methods, Page 122, Item 4**

Septum replacement is done daily, described in Section 5.10.2, Page 109, Paragraph 1.

**84. Section 5.10.3, Split Spoon Sampling, Page 123**

The procedure has been revised as recommended by EPA on Page 127, Paragraph 2.

**85. Section 5.10.4, Emission Flux Measurements, Page 130, Items 2 and 4**

Steady state criteria have been added to Section 5.10.4, Page 135, Item 5.

**86. Section 5.10.4, Downhole Emission Flux Measurement, Page 132**

A figure was not available. The procedures described in the text clearly describe placement and operation of this equipment.

**87. Section 5.10.4, Downhole Emission Flux Measurements, Page 133, Item 6**

Decontamination procedures have been revised in Item 6, Page 138.

**88. Section 5.10.5, Recordkeeping, Page 135, Paragraph 1**

Surface temperature has been added to the list of recorded information on Page 140.

**89. Section 6.0, Sample Custody, Page 1, Paragraph 1**

Sample custody and security descriptions have been added to Section 6.0, Page 1, Paragraph 3

**90. Section 6.1, Documentation Procedures, Page 1, Paragraph 3**

Additional detail and references for sample collection documentation and maintenance have been added to Section 6.1, Page 2, Paragraphs 1 through 3.

**91. Section 6.1, Documentation Procedures, Page 3, Paragraph 1**

Sample collection documentation and labeling are addressed in Section 6.1, Page 2, Paragraphs 2 and 5, and the top of Page 5.

**92. Section 6.2, Electronically Generated Chain-of-Custody and Master Log, Page 3, Paragraphs 1 and 2**

Detail regarding training, data entry, checks, and security are addressed in Section 6.2, Page 5, Paragraph 3

**93. Section 6.2, Electronically Generated Chain-of-Custody and Master Log, Page 8, Paragraph 1**

Responsibility for shipping and maintaining shipping and chain-of-custody records is described in Section 6.0, Page 1, Paragraph 3, Section 6.2, Page 5, Paragraph 3, and Page 9, Paragraph 1.

**94. Section 6.3, Sample Handling Procedures, Page 8, Paragraphs 3 and 4**

Sample handling recommendations are addressed in Section 6.3, Page 10, bullet 4 and bullet 1 on Page 11, Blue Ice is an option for shipment, noted on Page 10, Paragraph 2. Use of ice must be maintained as an option.

General Laboratory Sample Control and Storage procedures have been added on Page 11.

Laboratories performing analyses for a field project are designated in the SAP, SOW, and/or task instructions, as discussed in Section 6.2, Page 9, Paragraph 2.

**95. Section 6.3, Sample Handling Procedures, Page 10**

Refrigerator blanks, if performed, are analyzed according to laboratory procedures, included in Section 6.3, Page 11, bullet 5, under Laboratory Sample Control Procedures

**96. Section 7.3, Field Instrument Calibration, Page 1, Last Paragraph**

Calibration procedures for water level meters has been added to Section 7.3, Page 2, Paragraph 1. Calibration procedures for transducers and flowmeters or pumps have been added to Section 7.3, Page 6.

**97. Section 7.3, Real-time Portable OVAs, Page 2, Paragraph 3**

Availability of backup instruments is noted in Section 7.3, Page 2, Paragraph 1, and Page 2, Paragraph 3.

**98. Section 8.0, Analytical Procedures and Calibration**

The current document format was previously reviewed and accepted in the first issuance of this document. As discussed under general comment 16, this document was developed beginning in 1988, prior to issuance of the EPA IX guidelines. Preventive maintenance is addressed in Section 12.0, data calculations and reporting units, documentation, and deliverables are addressed in Section 9.0.

A consistent set of reporting limits for each analytical level, method and matrix has been established, and is presented in Tables 8-3 through 8-17.

**99. Section 8.0, Analytical Procedures and Calibration**

The Luft manual issued in October 1989 is referenced under Method SW8015 on Page 17, Paragraph 3

**100. Analytical Procedures and Calibration, SW8240 and SW8270**

The use of internal standards has been added to Paragraph 3 for each method on Pages 21 and 24.

**101. Section 8.4, Real-Time Portable Field Meters, Pages 31-35**

A description of procedures to improve consistency and comparability has been added to Section 8.4.1, Page 31, Paragraph 3.

**102. Section 8.6, Analytical Procedures for Physical Properties, Pages 35-37**

Field duplicates will be collected as designated for all analytical methods, and the method-specific QC requirements will be followed for physical properties analyses. There are no current plans for porosity or volumetric moisture measurements; these will be added to future updates of the QAPP if needed.

**103. Section 9.0, Data Reduction Validation, and Reporting, Pages 1-8**

Project data flow procedures have been added as Section 9.2, Pages 2 and 3. Transfer and verification of field information are discussed in Section 6.0, and in Section 9.2, Page 2, Paragraph 3 and top of Page 3.

Procedures for laboratory data review are described in Section 9.2, Page 2, Paragraph 2, in Section 9.3, Page 5, Paragraph 2, and in Section 13.0 and the Data Validation SOP in Appendix A.

**104. Section 9.1, Data Reduction, Page 1, Paragraph 2**

Most laboratories plan for 100%, data review by the supervisors. Data review is documented during laboratory systems audits, as stated in Section 9.0, Page 1, Paragraph 1.

**105. Section 9.2 Data Transfer and Verification**

Verification of electronic data transfer is described in Section 9.2, Page 2, Paragraph 2, and Page 3, Paragraph 3, and in Section 13.0 and the Data Validation SOP in Appendix A.

**106. Section 9.3, Data Validation, Page 4, Paragraphs 2 and 3**

Responsibilities for data validation procedures have been clarified in Section 9.1, Page 1, Paragraphs 2 and 3, and Section 9.3, Page 5, Paragraphs 1 and 2 for laboratory and project validation, respectively.

**107. Section 9.4, Reporting, Page 5, Paragraph 2**

The IRPIMS data checks are described in Section 9.4, Page 7, Paragraph 2. Archiving procedures have been added to Section 9.4, Page 9, Paragraph 2.

**108. Section 9.4, Reporting, Page 6, Bullet**

All engineering and field calculations are checked by the originator and a reviewer. This has been clarified in Section 9.4, Page 7, Bullet 1.

**109. Section 10.0, Internal Quality Control; Tables 10-1 to 10-9, Summary of Calibration and internal QC Procedures**

Re-extraction and reanalysis are corrective action options presented in the methods. The Laboratory Control Samples (LCS) are extracted control samples and provide an indication of preparation procedure performance. The use of matrix spike samples as ongoing calibration checks has been deleted from all tables. The subset of analytes used for calibration checks is presented in the tables in Section 4.0. Distinction between second source and independently prepared spike and calibration solutions has been added.

**110. Section 10.1, Analytical Laboratory Quality Control Samples, Pages 1-81.**

Clarification for adherence to the analytical specifications has been added to Section 10.1, Page 1, Paragraph 2.

**111. Section 11.0, Systems and Performance Audits**

Systems audits for subcontracting laboratories are addressed in Section 11.0, Page 1, Paragraph 2, and Page 3, bullet 5.

**112. Section 11.0, Performance and Systems Audits, Page 1, Paragraph 3**

Performance evaluation samples are discussed in Section 11.0, Page 2, Bullet 2, and Page 3, Bullet 1.

**113. Section 11.1, Performance and Systems Audits, Page 3, Paragraph 3**

There is currently no plan for air sampling, as discussed under comment 20. Therefore, no information about air sampling audits has been added.

**114. Technical Systems Audit, Figure 11-2**

Canister cleaning procedures are part of the laboratory procedures. Canisters must be analyzed and meet acceptance criteria for total non-methane hydrocarbons before they are released for field use. Blank analysis results may therefore, be requested by project staff for QC review. This information is not included as part of the field audit.

**115. Section 12.2, Maintenance Schedules, Page 2, Paragraph 2**

Reference for laboratory instrument maintenance procedures and schedules has been added to Section 12.1, Page 2, Paragraph 1.

**116. Section 13.0, Accuracy: Section 15.1, Quality Assurance and Quality Control Reporting Format**

The method spike reference was for old terminology, and has been updated to Laboratory Control Sample in Sections 13.2 and 15.1.

**117. Appendix A**

Appendix A contains copies of SOPs for non-standard and field methods, and the project QA SOP for data validation. Laboratory QA manuals are available upon request, and have been reviewed as part of the laboratory selection and audit procedures.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IX
75 Hawthorne Street
San Francisco, Ca. 94105-3901

RECEIVED
NOV 0 8 1991
Ans'd.....

31 October 1991

Mr. Mario Ierardi
Project Manager
SM-ALC/EM, Building 250N
McClellan AFB, CA 95652-5990

Dear Mr. Ierardi:

Please find enclosed the comments of the Environmental Protection Agency (EPA) to McClellan Air Base (MAFB) Summary of QAPP Revisions and Analytical Procedures for OU-B Field Work.

The incorporation of the comments may be included in the basewide Quality Assurance Project Plan (QAPP) for the Installation Restoration Program (IRP) Stage 3 for MAFB. The basewide QAPP is currently being reviewed by EPA.

If you have any question please contact me at (415) 744-2412.

Sincerely,

Lewis Mitani
Remedial Project Manager

cc: distribution list

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EPA Comments to
Summary of QAPP Revisions and Analytical Procedures
for OU B RI Field Work
McClellan Air Force Base

Section 1.0 Sonication Drilling Procedures, Responsibilities.

1. Some of the duties described for the field team appear to overlap. Due to the projected rapid rate at which samples will be retrieved using the sonication drilling techniques, it may be useful to describe, the specific duties of the field personnel as soil and other samples are collected. For example, on pages 7 and 8 the procedures for collecting soil samples could be annotated to describe the individuals that will be responsible for conducting each of the tasks. Some of these procedures may have to be developed in the field on a trial basis, an addendum to the revised QAPP should reflect current operational responsibilities.

2. The procedure used to cool the drilling apparatus should be described.

Section 2.0

3. Page 7. Standard Operational Procedures (SOP) for taking photoionization detector (PID) readings should be developed because field conditions (e.g. wind, rain, heat) can affect readings. This is especially important because the PID readings are one of the primary indicators being used to decide whether soil samples will be analyzed. To confirm the vertical extent of soil contamination has been defined, it is recommended samples from beneath the last obviously contaminated sample be taken where practicable.

4. Page 7 Bullet 3. Recommend the guidance on using PID readings to select sample intervals be clarified and simplified. It is not clear how a PID reading of 50 ppmv or greater was selected as a criterion for determining whether a soil sample will be analyzed. This criterion implies a 50 ppmv reading has some significance relative to the levels of contamination detected using analytical testing methods.

5. It is also stated in bullet three that "samples may be collected from intervals with PID readings of 20 to 49 ppmv if the readings are 10 times greater than other readings in the boring".

This statement should be clarified so it can be determined when samples will be collected. Using the factor of ten, soil with a PID reading of between 20 and 49 ppmv "may be collected" if all other PID readings in the boring are between 2.0 and 4.9 ppmv. Recommend samples be collected from the boring where the highest where the highest PID reading is recorded even if the highest reading is less than 20 ppmv and other PID readings are less than a factor of 10.

6. Page 7, Bullet 6. If there is an indication of contamination in a stepout boring the boring should be screened and sampled as described in the other bullets. It should be clarified whether the criterion specified in bullet 6 is for stepout borings where there is no indication of soil contamination.

Section 4 Field Laboratory Procedures

7. Page 10, Bullet 2. It should be clarified how "all unidentified peaks greater than 100 ug/kg will be quantitated using the response factor for the calibrated compound with the nearest retention time". It will be difficult to determine whether an unidentified peak is above 100 ug/kg unit the quantification is completed. Recommend tentatively identified compounds (TIC) be flagged as such.

8. Page 10, Bullet 3. It should be stated how the method detection limit will be calculated.

9. Page 10, Bullet 4. It is recommended EPA SW-846 guidance be used to conduct the calibration. There is no reference to linear regression procedures for multi-point calibration in EPA SW-846.

10. Page 11, Bullet 1. It is recommended a minimum frequency for conducting multi-point calibrations be specified in the QAPP.

11. Page 11, Bullet 3. The procedures that will be followed if all analytes are not below the laboratory-derived detection limit for a reagent blank should be specified.

12. Page 11, Bullet 4. In bullet 4 it is stated "either matrix spike/matrix spike duplicate or surrogates and laboratory duplicates be analyzed at a frequency of five percent (the surrogates will be added to all samples, blanks, standards)". As specified in EPA SW-846, it is recommended matrix spike/matrix spike duplicate paris be analyzed at a frequency of five percent with surrogates added to all samples, blanks, and standards.

13. Page 11, Bullet 6. It should be clarified under what conditions it would not be possible to analyze a system blank.

14. Page 12. There is a discussion of the number of samples which will be sent to a fixed laboratory for analysis by EPA Method 8240. It is unclear how this strategy applies to data gathering activities for a remedial investigation. The final plan should include a detailed discussion of the samples which will analyzed by the fixed laboratories. In addition, the final plan should discuss the end use of all analytical data and quantitative QC objectives for all methods.

Section 5.0 Sample Collection/Distribution Rationale, Data Comparability.

15. To the extent possible, we recommend 100 percent of the soil samples collected for analysis by one method from one site or cluster of sites be sent to a single laboratory. It is stated in bullet 1, 90 to 100 percent of sample from one site will be sent to a single laboratory. If only 10 percent of the samples from one site are sent to a different laboratory it may be difficult to determine whether there is a statistically measurable difference in the reported values between laboratories. It is recommended procedures be developed to specify how a set of samples from a single site will be split between laboratories if required due to work load or other problems.

## RESPONSE TO EPA REGION IX COMMENTS ON SUMMARY

**Section 1.0, Sonication Drilling Procedures, Responsibilities**

**1. Field Team Responsibilities Overlap**

There is no overlap in the responsibilities of the field team, with the exception of core barrel breakdown and packaging by the technicians. The supervising rig geologist performs tasks 3 through 9 in the sampling procedure described on Pages 7 and 8.

**2. Sonication Apparatus Cooling Procedures**

The current sonication rig procedure generates heat at levels similar to a hollow stem auger rig. The heat is dissipated by the subsurface deposits and air.

**SECTION 2.0**

**3. Page 7, Standard Operating Procedures for PID Operation**

Standard procedures for PID calibration and operation are presented in Sections 7.3 and 8.4 of the QAPP. A core segment cover with a PID sampling port has been designed and is in use to standardize PID readings under various weather conditions. Step 7 addresses collection of samples beneath obviously contaminated zones.

**4 & 5 Page 7, Bullet 3, PID Readings Used for Sample Selection**

The criteria for sample selection of >50 ppm or greater than 10 times the observed PID readings in the boring are based on previous PID screening data collected for coring and sampling programs at McClellan AFB. These criteria have significance for the site. See the response to DTSC general comment 1 for rationale.

**6. Page 7, Bullet 6, Sample Selection**

This criterion is applied in addition to the initial selection criteria in bullets 1 through 5. All borings are subjected to sampling steps 1 through 8 on pages 7 and 8.

**Section 4 Field Laboratory Procedures**

**7. Page 10, Bullet 2**

Because the field laboratory is using surrogate standards at a specified concentration for each sample, the height of the surrogate peak can be used to estimate the concentration of the unidentified peak. If it is near or greater than 100 $\mu$/kg, the unidentified peak will be quantitated relative to the nearest calibrated analyte, and will only be reported if the concentration is greater than or equal to 100 $\mu$/kg. Tentatively identified compounds are flagged with a "J". This is described in the SOPs for field analysis presented in Appendix A.

**8. Page 10, Bullet 3**

The method detection limits will be calculated according to the U.S. EPA procedure described in Appendix B to Part 136 "Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.1," 40 CFR 136, 1984. The MDL is defined as the standard deviation of the seven replicate results times the students' t value for the 99% confidence level with n-1 degrees of freedom.

**9. Page 10, Bullet 4**

Section 7.4.2.2 of SW-846 describes two ways of calibrating. One way is to prepare a calibration curve for each analyte using area responses and mass injected. The widely-accepted procedure used to define the calibration curve (or line) is through calculation of the slope and intercept using the linear regression equation.

10. **Page 11, Bullet 1**

A minimum frequency for conducting multi-point calibrations is not specified in SW-846. However, calibration checks are analyzed daily (and QC checks every ten samples) to assure that the multipoint calibration is still valid.

11. **Page 11, Bullet 3**

If any analyte in the reagent blank is greater than the reporting limit, the reagent blank will be reanalyzed. Corrective action will be taken if necessary to reduce blank contamination to acceptable levels. No field samples will be analyzed until a reagent blank that meets this criterion has been analyzed .

12. **Page 11, Bullet 4**

Section 4.0 on Page 9 explains that some QC requirements have been relaxed for Phase I screening data. These are defined as Level 2 analyses in Section 4.0 of the QAPP. The substitution of surrogates and laboratory duplicate analyses for the MS/MSD requirement in SW-846 is one of these alterations. MS/MSD results are used to determine analytical precision and accuracy in the sample matrix. Surrogate recoveries are used to measure analytical accuracy for the sample matrix, and duplicate analyses are used to measure analytical precision. Thus both precision and accuracy can be evaluated using either alternative.

13. **Page 11, Bullet 6**

A system blank cannot be analyzed following samples with high concentrations if the samples are loaded on an autosampler and analyzed without immediate analyst review (i.e., overnight). In the event that cross-contamination is indicated, the sample(s) following high-level samples will be reanalyzed to verify the results.

**14. Page 12, Sample Selection for Fixed Laboratory Analysis**

This phase of the RI is intended to identify sites requiring further sampling and analysis. The subset of samples selected for confirmation analyses will provide information to support the higher number of screening analyses performed in the field. This approach is most cost-effective, and was discussed with agency and Air Force representatives, and was approved prior to beginning field work for the OUB RI.

**15. Section 5.0, Sample Collection/Distribution Rationale, Data Comparability**

During the course of the RI, 100% of samples from a site will be sent to one laboratory unless laboratory problems or field schedules will not allow this to occur. In the event samples will have to be split among laboratories, comparability has been addressed by requiring adherence to the same protocols, laboratory audits, and review of method performance data for each laboratory and method. If a laboratory has fulfilled the project requirements stated in the QAPP, verified by these reviews, analyses will be approved.

DRAFT

# DTSC COMMENTS ON THE OU-B RI QUALITY ASSURANCE PROJECT PLAN REVISIONS

JKAF

## I. GENERAL COMMENTS:

The QAPP should provide rational for using a PID reading of 50 ppm, or greater than 10 times reading from previously screened cores, as screening criteria.

The QAPP should discuss calibration of the PID using contaminants most like to be found during the field work. Efforts must be made to make the PID screening process as precise as possible.

There is no detailed description of the standards used to calibrate the instruments. Are descriptions of the standards in the standard operating procedures?

Six criteria and an eleven step sampling process have been described in the QAPP revisions. Sampling personnel must have a clear understanding of the prioritization of the sampling criteria and sampling process. A decision logic flow chart could prove helpful in refining and following the sampling process.

## II. SPECIFIC COMMENTS

| Pg. | Pgph. | Comment |
|---|---|---|
| 2 | 1 | During the Sonication drilling demonstration at McClellan AFB on August 9, 1991, use of an unlined sampler/core barrel resulted in the sampler becoming tightly jammed in the drill string. Apparently, the sampler separated slightly to allowing sand to wedge between the sampler and the drill string. The drill string had to be removed to allow for sample retrieval. The QAPP should specify use of samplers that are rigid enough to prevent jamming or describe protocol that will be followed to prevent jamming. |
| 2 | 3 | Please clarify the last sentence regarding the 140-pound hammer and use within the drill stem. |
| 7 | 1 | A maximum soil boring depth should be provided in the event that contamination persist to the water table. |
| | | Bullet 1. The Department concurs with the RWQCB comment regarding use of a minimum pre-selected depth of 10-15 feet (not 5 feet) below the potential contaminant release depth. |

DRAFT

| Pg. | Pgph. | Comment |
|---|---|---|
| 7 | 1 | Bullet 2. The QAPP should be amended to specify that "observed waste or obvious contamination" includes PID responses. |
| | | Bullet 6. Sampling criteria for adjacent and stepout borings should be the same criteria used for all other borings. Additional criteria and sampling depths, as described, will be based on previously detected contamination in adjacent borings. |
| 8 | | Item 4. Requires hand coring of an acquired core. If no lateral support is provided the material may crumble causing a loss of volatiles. The volatile results should be considered low if sleeves are not provided for the initial core and a split spoon sampler is used. Will hand coring be possible if hard material (40-50 blows/6 inch penetration) is encountered? Add a note that if odors exist, the need for respiratory protection will be closely evaluated. |
| | | Item 6. Selection of the sampling intervals should be based on evidence of waste, discoloration, odors and/or lithology (fine-grained units). |
| | | Item 7. Step number seven must be reconciled with the second criteria identified on page 7. Samples should be collected and analyzed where visible signs of contamination exist or where PID readings indicate contamination. Nearby fine grained units should also be sampled if evidence (visual, PID, odors) of contamination is present. Sampling of fine-grained material should be subjugated to positive indications of contamination. |
| | | Item 8. The sampling depth interval for the "finest-grained layer" should account for the depth of contamination in nearby borings. |
| | | Item 9. The QAPP should specify a minimum number of samples to collect, not the "maximum." |

DRAFT



| Pg. | Pgph. | Comment |
|---|---|---|
| 9 | 1 | The borehole abandonment procedures should include completion logs specifying actual amounts of ingredients used, grout weights, placement methods, calculated volumes, actual volume placed, and other pertinent information. |
| 10 | | Bullet 1. Modifications of previously approved standard operating procedures should be submitted for review and comment. |
| 11 | | Bullet 1. Describe what action will be taken if check standards results are outside of 20-30% window. |

III. SECONDARY COMMENTS

| Pg. | Pgph. | Comment |
|---|---|---|
| 7 | 1 | Bullet 2-5. Describe why the term "Representative sample" is used in place of "Sample". |

## RESPONSE TO DTSC COMMENTS ON SUMMARY

### I. GENERAL COMMENTS

1. The PID screening criteria of >550 ppm, or greater than 10 times the other boring readings is based on PID screening data from previous McClellan AFB coring and sampling programs. PID readings less than 50 ppm have not been associated with soils having greater than 10 to 20 ppb of volatile organic compounds, and most associated soil samples were less than 10 ppb.

2. PID calibration procedures are described in Section 7.3 of the QAPP, and use of isobutylene in air, as recommended by the manufacturer. Based on instrument response to this gas, contaminants such as benzene will be conservatively measured. Also, isobutylene is non-hazardous.

3. The standard calibration gases are described in Section 7.3 of the QAPP.

4. The sampling procedures and sample selection criteria were communicated to field staff during training dry-runs, and have been systematized as the RI field work has progressed. Adherence to the procedures and criteria have been evaluated during field audits conducted for the OUB RI drilling effort.

### II. SPECIFIC COMMENTS

**Page 2, Paragraph 1, Sonication Drilling Sampling Equipment**

The sampling core barrels were redesigned/modified to eliminate the jamming problem observed. The core barrels currently in use do not exhibit this problem.

**Page 2, Paragraph 3, Drive Hammer Use.**

The reference to use of a drive hammer inside the drill stem is an alternative configuration to drive the core sampler into the ground.

**Page 7, Paragraph 1, Maximum Soil Boring Depth**

The maximum depth of soil borings will be 100 feet, which is 5 to 8 feet above the water table in OU B. This was stated in the OUB SAP (Radian, 1991).

**Page 7, Paragraph 1, Bullet 2, Observed Waste or Contamination Criteria**

The sample selection criteria are presented in the OUB SAP (Radian, 1991). Observed waste or obvious contamination is a separate criterion for sample selection. It is possible that high PID responses will not occur with wastes that contain metals or semivolatile compounds.

**Page 7, Bullet 6, Sampling Criteria for Adjacent Borings**

The criteria stated in Bullet 6 will be used in addition to the initially stated criteria, as recommended.

**Page 8, Item 4, Hand Coring**

Lateral support will be used if cores to be sampled crumble or are unconsolidated. This has been an infrequent occurrence. Hard material in the cores has not interfered with sample collection from the cores. Health and safety procedures presented in the Radian Health and Safety Plan (Radian 1991) are followed during all field activities.

**Page 8, Item 6, Sampling Interval Selection**

Lithology is listed as a selection criterion in Items 8 and 9.

**Page 8, Item 7, Sampling Interval Selection**

Sampling of fine grained units is subjugated to other indicators. That is the reason this criterion is step 7, and not step 3 or 6.

**Page 8, Item 8, Sampling Interval Selection**

This item is specifically added here to account for contamination in nearby borings.

**Page 8, Item 9, Number of Samples**

The reference should be to the minimum number of samples to be collected. This is being done in the field.

**Page 9, Paragraph 1, Borehole Abandonment**

Separate abandonment logs will not be prepared. The abandonment information will be included on the drilling log forms.

**Page 10, Bullet 1, SOPs for Modified Analytical Methods**

The SOPs for field analytical methods have been included in Appendix A of the QAPP.

**Page 11, Bullet 1, Corrective Actions for Check Standards**

If the daily check standards do not meet the acceptance criteria, a second check will be analyzed. If specifications still cannot be met, instrument maintenance and recalibration are required. This is documented in the SOPs in Appendix A.

## III. SECONDARY COMMENTS

**Page 7, Paragraph 1, Bullets 2-5**

All samples collected for environmental analysis should be representative of the matrix in the interval sampled. One cannot expect representativeness in analytical data without representative samples.

STATE OF CALIFORNIA GEORGE DEUKMEJIAN, Governor

CALIFORNIA REGIONAL WATER QUALITY CONTROL BOARD—
CENTRAL VALLEY REGION
3443 ROUTIER ROAD, SUITE A
SACRAMENTO, CA 95827-3098

4 September 1991

Ms. Elaine Anderson
Environmental Management
SM-ALC/EMR
Building #250-HH
McClellan Air Force Base, CA 95652-5990

***QUALITY ASSURANCE PROJECT PLAN (QAPP) REVISION FOR OPERABLE UNIT B REMEDIAL INVESTIGATION, MC CLELLAN AIR FORCE BASE***

Thank you for the submittal of the subject plan revision. Regional Board staff has reviewed the proposed revisions and presents the following comment:

1. Page 7, Sample Selection Criteria. It is stated that the sampling will continue to the pre-selected total depth of the boring or to a depth interval 5-feet below the greatest discharge depth at the location or to the base of observed waste or contamination, whichever is greater. For sumps, tanks, and pits, and similar waste units pre-selected depths should be at least 10-15 feet below the bottom of the potential contaminant release depth. Sampling is generally not possible immediately beneath such units if they are still operating, and a certain vertical distance is necessary to allow horizontal transport of pollutants. In addition, will PIDs or similar instruments be used to determine the base of "observed waste or contamination"? Volatile organic contamination can be difficult to detect by simple visual observations.

If you have any questions regarding this matter, please call me at (916) 361-5626.

ALEXANDER MACDONALD
Project Engineer

AMM

Enclosure

Mr. Lewis Mitani, U.S. Environmental Protection Agency, San Francisco
Mr. Mark Malinowski, Dept. of Toxic Substances Control, Sacramento

SEP 06 REC'D

## RESPONSE TO RWQCB COMMENTS ON SUMMARY

1. **Page 7, Sample Selection Criteria**

The reference to 5 feet was a typographical error; 15 feet is the planned depth for sampling below observed discharge points. PID readings, visual observations, and presence of chemical odors are all used to select samples. These criteria were presented in bullets 3 and 4 on Page 7.

STATE OF CALIFORNIA — PETE WILSON, Gov.

CALIFORNIA REGIONAL WATER QUALITY CONTROL BOARD—
CENTRAL VALLEY REGION
3443 ROUTIER ROAD, SUITE A
SACRAMENTO, CA 95827-3098
PHONE: (916) 361-5600
FAX: (916) 361-5688

9 April 1992

Mr. Mario Ierardi
Environmental Management
SM-ALC/EMR
Building #250-HH
McClellan Air Force Base, CA

***QUALITY ASSURANCE PROJECT PLAN, INSTALLATION RESTORATION PROGRAM STAGE 3, MC CLELLAN AIR FORCE BASE***

Thank you for the submittal of the subject plan. Regional Board staff has reviewed it and presents the following comments:

1. Section 4.0, Table 4.4. This table contains the QA/QC objectives for Level 3 measurements in water. The listed methods are primarily the SW8000 series methods. The methods for analyzing water and wastewater samples are developed by the Environmental Protection Agency and for the listed SW8000 series the correct methods should be the EPA Method 600 series. Cyanide should be analyzed for by EPA Method 325, arsenic by Method 206, lead by Method 239, selenium by Method 270, and the ICPES by the Method 200 series.

2. Section 4.0, page 22, paragraph 1. It is stated that the transducers will be calibrated and checked for precision, accuracy, and comparability of pressure readings using hand-held, water-level probes, at the beginning and throughout the duration of the aquifer test. What are the acceptable levels of precision, accuracy, and comparability?

3. Section 8.0. This section deals with analytical methods and calibration. As in Comment 1, above, the Solid Waste analytical methods are not the appropriate methods for the analysis of water and wastewater. In addition, the following reporting limits are too high and are currently lower at on-going projects at McClellan:

   Cadmium (0.005), chromium (0.05), cooper (0.05), nickel (0.15), zinc (0.02), lead (0.005), mercury (0.001), the EPA 601 series (0.0005 to 0.010), TPH (1.0), and EPA 602 series (0.0005 to 0.004). All in mg/l.

   Nearly all EPA 601 and 602 constituents should have detection/reporting limits in the 0.5 µg/l range. TPH should be near 50 µg/l.

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4. Section 16. This section deals with site management and refers specifically to Radian Corporation and the roles its personnel will be taking in the site management activities. This section should be general in nature, and should not be specific to any one contractor working at McClellan. At this time Jacobs and $CH_2M$-Hill are also performing major tasks at McClellan.

If you have any comments regarding this matter, please call me at (916) 361-5626.

ALEXANDER MACDONALD
Project Engineer

AMM

cc: Ms. Katherine Moore, U.S. Environmental Protection Agency, San Francisco
Mr. Mark Malinowski, Dept. of Toxic Substances Control, Sacramento

## RESPONSE TO CALIFORNIA REGIONAL WATER QUALITY CONTROL BOARD COMMENTS

COMMENT 1, Section 4.0, Table 4-4

COMMENT SUMMARY: Question about whether it is appropriate to use SW-8000 series analytical methods for aqueous samples.

Response:

The use of SW-846 methods for analysis of aqueous samples rather than the EPA 600 and 200 series methods has gained general acceptance in the technical community over the past several years. The SW-846 methods were designed for solid waste samples, but are applicable to aqueous samples. The SW-846 methods are analogous to the EPA 600 and 200 series methods. The Air Force IRP Handbook, which is used to specify analytical methods acceptable for sample analysis for all IRP sites has required use of these methods for both solid and aqueous samples since May, 1989. The primary reason for selecting SW-846 methods is the additional level of QC required; the use of five-point rather than three-point calibrations, matrix spike/matrix spike duplicate analyses, and other QC protocols provide additional information useful for data validation and assessment. These methods were included and accepted in the first issue of QAPP that was approved and finalized in May, 1990.

COMMENT 2, Section 4.0, Page 22, Paragraph 1.

COMMENT SUMMARY: Acceptable transducer calibration, precision, accuracy, and comparability levels should be included.

Response:

See responses to EPA comments on Response 7, 53 and 54, and 56.

COMMENT 3, Section 8.0

COMMENT SUMMARY: Reporting limits for metals, TPH, SW8010/601 and SW8020/602 are higher than other current projects at McClellan.

Response:

The reporting limits presented in the QAPP have been established as project objectives to ensure comparability of data reported from multiple laboratories, and are the Maximum Allowable Quantitation Limits for IRP sites IRP Handbook, May 1991). Previous versions of the QAPP have presented the reporting limits based on individual laboratory capabilities, but this approach results in data which are not comparable

between laboratories or over time. Therefore, based on EPA comments on the Draft QAPP to establish overall project objectives, a single set of reporting limits will be used for all laboratories performing sample analyses under this QAPP. The levels presented are considered reasonable to provide information needed to assess site conditions.

**COMMENT 4, Section 16.**

COMMENT SUMMARY: Site management activities specific to Radian personnel and not general for all contractors performing work at McClellan AFB.

**Response:**

A basewide QAPP will be developed and the site management discussion will encompass multiple contractors by using a more general format, or will include information for each contractor.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION IX
75 Hawthorne Street
San Francisco, Ca. 94105-3901

April 3, 1992

Mr. Fran Slavich
Remedial Project Manager
Environmental Management
SM-ALC/EMR
Building #250-HH
McClellan Air Force Base, CA

Dear Mr. Ierardi:

We have reviewed the draft final document entitled, "Operable Unit B Quality Assurance Project Plan (QAPP) for McClellan Air Force Base." The Air Force's responses to our comments on the draft document are adequate. However, the following comments remain.

The Air Force's approach of using the QAPP to develop the standard operating procedures (SOPs) and Quality Assurance Objectives (QAOs) for each analysis method, associating the method with an analytical level, and requiring all laboratories to follow the SOPs and meet the QAOs appears to be reasonable. Discussion of site-specific DQOs would be in the individual sampling and analysis plans (SAPs) for the operable units (OUs). Because the analytical level for the sampling procedure or analytical method will be specified in the QAPP, it will be possible to determine whether the analytical method will meet the site-specific DQOs specified in the SAP.

However, if this approach to defining DQOs is taken, it is strongly recommended that DQOs for the basewide investigation be developed. The remedial objectives from OU to OU should not change substantially across the base. Therefore, a document that defines program DQOs should be prepared and used to refine and provide consistency to the specific DQOs and QAOs that will be described in the QAPP and SAPs. It is difficult to see how site-specific DQOs can be consistently defined and implemented if program DQOs have not been defined.

EPA has raised concerns regarding the soil sample selection criteria presented in the OU B QAPP. The Air Force response addresses only part of the concern expressed. If the 50 parts per million volume (ppmv) photoionization detector (PID) readings do have "significance" relative to analytical measurements, this

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conclusion should be supported with data. In addition, in the Air Force response to the comment it is stated that a PID reading of less than 50 ppmv has not been associated with soils having greater than 10 to 20 ppb of volatile organic compounds. It is recommended that the significance (or lack of significance) of volatile concentrations of 10 to 20 ppb be supported.

Also, it was stated in the Air Force's response summary that "samples may be collected from intervals with PID readings of 20 to 49 ppmv if the readings are 10 times greater than other readings in the boring." It is recommended that this criterion be supported or replaced with a criterion that is more flexible and can account for site-specific conditions. As stated previously, it is recommended that a sample be taken where the maximum PID reading was detected in the borehole.

Additional technical comments are enclosed. If you would like clarification of these comments, please contact me at (415) 744-2407.

Sincerely yours,

Katherine Moore

Katherine Moore
Remedial Project Manager

Enclosure

cc: Mark Malinowski, DTSC
Alex MacDonald, RWQCB
Greg Reller, PRC
Dave Watson, PTI

Enclosure

Additional Technical Comments on Draft Final OU B QAPP

The comments are referenced by the number assigned to the response in the draft final QAPP.

## Response 7.

The original EPA comment noted that acceptable tolerances for hydrological measurements should be provided in the text along with a reference for those tolerances. The response indicated that the discussion of hydrological tolerances was expanded in the QAPP. That discussion is inadequate because it does not ensure that the accuracy at which hydrologic data will be collected will meet DQOs. For example, the text notes that flow meters will be capable of reading 1 gallon per minute of flow. This specification fails to provide a level of accuracy (e.g., ±10 percent) that will meet DQOs. The following tolerances have been considered adequate for hydrological investigations by the U.S. Geological Survey (Stallman 1971):

- Control-well discharge (±10 percent)
- Depth to water measurements with an electric sounder (±0.01 feet)
- Distance from control well to each observation well (±0.5 percent)
- Synchronous time (±1 percent of time since control effected)
- Elevations of measuring points (±0.01 feet)
- Vertical distance between measuring point and land surface (±0.01 feet)
- Total depths of all wells (±1 percent)
- Depth and length of screened intervals of all wells (±1 percent)
- Diameter, casing type, screen type, and method of construction of all wells (nominal).

DQOs should be reviewed relative to these recommended tolerances and the tolerances should be accepted or modified as required based on project needs.

## Response 10.

The original EPA comment indicated that soil samples should be placed in a methanol solution in the field (as recommended by EPA guidance) to reduce loss of volatile constituents. The response to the comment indicates methanol was considered but was ruled out

because it was considered impractical for use in the field. At a minimum, it is recommended that duplicate soil samples be placed in a methanol solution at a test location, and the results from the analyses of the duplicate samples be evaluated to determine whether loss of volatile constituents due to sampling procedures is a major concern at the base.

## Response 44.

The response to the EPA comment concerning corrective actions in case of misalignment of the casing indicated that the text had been revised to address the concerns. The text in the draft final QAPP notes that in the event of casing misalignment, steps (unspecified in the text) will be taken to align the casing. It is recommended that these "steps" be specified and be designed to ensure that the bentonite well seal remains undisturbed. It is unclear how the bentonite well seal will remain intact if the casing is moved to align it. Therefore, it is recommended that the alignment be done before placement of the bentonite seal.

## Responses 53 and 54.

EPA comments 53 and 54 concerned selection of instrumentation for monitoring water levels during aquifer testing. If water level measurements are required to ±0.01 feet, a pressure transducer with a stated accuracy of ±0.2 percent full scale as discussed on page 47 (Section 5.4.1) would be inadequate if its range exceeds approximately 2 psi. These limitations should be considered in choosing testing equipment. See comment on Response 7 concerning tolerances during hydrological investigations.

## Response 56.

EPA's comment noted that procedures should be incorporated in performing pumping tests to ensure that the pressure transducers are properly calibrated after being placed in the well. The response indicated that the information was added to Section 5.4.1, Page 47, Paragraph 3. The information provided in the text does not completely address the concerns that the transducers be tested and operating prior to use. It is recommended that after equilibrium is established, the transducer be moved up or down a known distance and the resulting change in pressure noted. This change should be compared to the measured distance moved to ensure the pressure transducer is operating properly.

## Response 64.

The EPA comment was on equipment decontamination procedures. In the response, it is stated that "a final rinse and pumping with ASTM II water is impractical for use in the field." However, the EPA comment did not call for pumping ASTM II-grade water through the nondedicated pumps. Instead, it was recommended that "at a

minimum, clean water be pumped through the interior of the nondedicated submersible pump and other pumps prior to sampling at the next well location." In addition, EPA recommended that "all equipment be given a final rinse with ASTM type II reagent water to wash away any methanol or cyclohexane residue." This final rinse should be practical, since the present procedures for decontamination described on page 71 of the draft final QAPP call for this type of rinse prior to the methanol or cyclohexane rinse. It should also be clarified why and how the decontamination procedures are different if the ambient temperature is below 45°F.

## Response 102.

This EPA comment concerned lack of QC on analytical parameters for physical properties, and lack of porosity and volumetric moisture content analyses. It is stated in the response that there are no current plans for porosity or volumetric measurements. However, the SAPs for OU C1 and OU A discuss the need for these analyses. As stated in the original EPA comment, these parameters will be needed to understand contaminant fate and transport now and under cleanup scenarios. Therefore, it is recommended that procedures for conducting these analyses be provided in the QAPP.

## RESPONSE TO EPA REGION IX COMMENTS

**General Comments**

**PARAGRAPH 3, Page 1, Cover Letter**

COMMENT SUMMARY: There is still a need for a document presenting program Data Quality Objectives for the basewide investigation to ensure consistency in the remedial objectives from OU to OU.

**Response:**

A basewide QA Program Plan will be developed to address Data Quality Objectives and ensure consistency in RI/FS activities across McClellan AFB.

**PARAGRAPH 4, Page 1, and Paragraph 1, Page 2, Cover Letter**

COMMENT SUMMARY: Data showing significance of photoionization detector (PID) readings relative to analytical measurements should be provided to support use of 50 ppmv photoionization detector readings as soil sample selection criteria. The criterion for collecting samples from intervals with PID readings less than 50 ppmv should be supported with data, or made more flexible to account for site-specific conditions. Samples should be collected from the core interval with the maximum PID reading.

**Response:**

Data from OUB RI sample analyses indicate that there is no correlation between field PID readings on the core and concentration of VOCs in soil samples. The text regarding soil sample selection criteria has been changed to state that a soil sample will be collected for analysis from the core intervals in each borehole that have the highest PID readings above ambient levels. All other sample selection criteria will remain unchanged.

**Technical Comments**

**RESPONSE 7**

COMMENT SUMMARY: Acceptable tolerances for hydrological measurements should be provided in Section 4.0. DQOs should be reviewed relative to the USGS recommended tolerances presented in Stallman (1971), and modified as required based on the project needs. The tolerances presented in Stallman are listed.

**Response:**

The text will be changed to include the tolerances presented in Stallman (1971). However, for aquifer tests the precision of measurements of water level changes is more important than the accuracy of each individual measurement because the change in water levels over time is used in analyzing the data. Therefore, the use of transducers with an accuracy of $\pm$ 2 percent is adequate.

**RESPONSE 10**

COMMENT SUMMARY: The use of methanol preservation for volatile constituents in soils should be further evaluated by conducting a comparison of preserved and unpreserved duplicate samples at a test location.

**Response:**

As stated, the use of methanol was considered and ruled out because of logistical problems in handling the total volume of samples and sampling rate projected for the OUB field effort. Other considerations when evaluating the usefulness of this method for Phase 1 of the OUB RI were the increased detection limits caused by sample dilution that would have been greater than the reporting levels established for this program, and the decision to use a field VOC laboratory for rapid analysis of soil samples. Nearly all field laboratory analyses have been performed within 6 hours of sample collection on samples disturbed as little as possible, minimizing the potential for loss of volatiles. The analysis of samples for VOCs in off-site laboratories has indicated a probable loss of volatiles, but the increased detection limits that would result from the dilution factor associated with the methanol preservation are often greater than the observed losses. A further evaluation of these results is being conducted as part of QC and data analysis activities. Changing procedures at this point in the Phase 1 investigation raises questions about the comparability of the data, and is not recommended. A comparison study between the field analysis, off-site analysis, and methanol preservation may be conducted and used for post-RI sampling efforts.

**RESPONSE 44**

COMMENT SUMMARY: Steps used to align bent casings should be specified and designed to ensure that the bentonite well seal remains undisturbed. The alignment should be done before placement of the bentonite seal.

**Response:**

The text has been changed to state that the casing will be tested for alignment prior to emplacement of the bentonite seal. Any misaligned casing will be straightened or replaced prior to emplacing the seal.

**RESPONSES 53 and 54**

COMMENT SUMMARY: Instrument tolerances of ± 0.2 percent full scale for water level measurements may not be adequate to monitor water levels within ± 0.01 feet for some instruments; this should be taken into account when selecting equipment. Refer also to Comment/Response 7.

**Response:**

The use of the data must be considered when determining the needed accuracy and the appropriate equipment for any field work to be performed. The stated accuracy of the transducers is adequate for the methods of analysis used to determine aquifer parameters. The use of transducers and data loggers discussed in the QAPP allow precise measurements of water levels for the planned data uses. Also see Response 7.

**RESPONSE 56**

COMMENT SUMMARY: Pumping test procedures should include more information regarding pressure transducer calibration after being placed in the well and equilibrium is established. The transducer should be moved up or down a known distance, and compared with the measured distance to ensure proper operation.

**Response:**

The recommended procedure has been included in the text in Section 5.4.1.

**RESPONSE 64**

COMMENT SUMMARY: The original comment recommended pumping clean water through the interior of non-dedicated submersible pumps prior to sampling the next well. rather than use of ASTM type II water, stated in the Air Force response as impractical. The final rinse with ASTM type II water after solvent rinses for all sampling equipment should be included to remove any solvent residues, as recommended in the original comment. The specification for the use of hexane rather than cyclohexane for the nonpolar solvent rinse when temperatures are below 45 degrees F should be clarified.

**Response:**

The term "clean" water is vague and unspecific; the use of potable water, purchased distilled water, or water from other sources may contain low levels of contaminants. The procedure for decontamination of submersible pumps specifies that the steam or high pressure hot water rinse be forced through the pumps and around intake and check valves to clean the interior of the pump. This step is considered to be sufficient to rinse away or volatilize remaining contaminants or residues. An additional consideration is that the submersible pumps are only used in deeper wells which require up to several

hundred gallons of water to be purged, and there are one-way check valves to prevent backflow into the well being purged. Therefore, the potential for introducing contaminants into samples collected from the wells purged using this equipment is minimized. It is still considered unnecessary and impractical to pump water through the submersible pumps at the surface as part of the decontamination procedure because of the necessity to either carry large volumes of "clean" water with the sampling truck or van, or to return to a central location between wells to perform this operation, and because of the additional time and labor required. The probability of cross-contamination by following the current procedure is very low, as indicated by historical QC data collected for the GSAP program over the past several years; field blank contamination has not been traced to this source.

The recommendation for a final rinse with ASTM type II water to remove any residue remaining after the solvent rinses has been added. The text has been modified to note that hexane is used when ambient temperatures are below 45 degrees F because the cyclohexane freezes at these temperatures and does not evaporate. The use of hexane is minimized because of health and safety concerns for the field samplers.

**RESPONSE 102**

COMMENT SUMMARY: Porosity and volumetric moisture content measurements are included in the SAPs for OU C1 and OU A; they should be included in the QAPP because they are needed to understand contaminant fate and transport and to evaluate cleanup scenarios.

**Response:**

This document currently references procedures planned and in use for the OUB RI; however, the procedures for porosity and volumetric moisture content will be added to section 8.6 as recommended.

The text will be modified to state that:

Porosity of a soil sample will be determined from the relation:

$$\text{Total porosity} = 1 - \frac{\text{dry bulk density}}{\text{particle density}}$$

Dry bulk density of samples will be determined by ASTM Method 854. A particle density of 2.65 $g/cm^2$ will be assumed for the soils beneath McClellan AFB. This particle density is a reasonable value for the quartz-feldspar-clay-mica soils typically encountered.

Volumetric water content of a soil sample will be determined from the relation:

$$\text{Volumetric fraction } (cm^3/cm^3) = \frac{\text{dry bulk density soil}}{\text{bulk density water}} \times \text{weight fraction water } (g/g)$$

The bulk density of the soil will be determined by ASTM Method 854. The bulk density of water will be assumed equal to 1 $g/cm^3$. The weight fraction of water will be determined by ASTM Method 2216-80.