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Upper Columbia River Expanded Site Inspection
Mines and Mills
Northeast Washington
Sampling and Quality Assurance Plan

TDD: 01-02-0028

Contract: 68-S0-01-01
June 2001

Region 10

START-2

Superfund Technical Assessment and Response Team Two

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SAMPLING AND QUALITY ASSURANCE PLAN FOR:

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Northeast Washington

TDD: 01-02-0028

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Contract No: 68-S0-01-01

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SAMPLING AND QUALITY ASSURANCE PLAN
UPPER COLUMBIA RIVER MINES AND MILLS ESI
NORTHEAST WASHINGTON

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LIST OF ACRONYMS

Acronym	Definition
ASL	above sea level
bgs	below ground surface
CAS	Chemical Abstracts Service
CD-ROM	compact disk-read only memory
CERCLIS	Comprehensive Environmental Response, Compensation and Liability Information System
cfs	cubic feet per second
CLP	Contract Laboratory Program
CLPAS	Contract Laboratory Program Analytical Services
COC	chain-of-custody
Cominco	Cominco American, Inc.
DQIs	data quality indicators
DQOs	data quality objectives
E & E	Ecology and Environment, Inc.
Ecology	Washington State Department of Ecology
EDDs	electronic data deliverables
EIS	Environmental Impact Statement
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
ESI	Expanded Site Inspection
FASP	Field Analytical Support Project
FOWP	Field Operations Work Plan
gpm	gallons per minute
GIS	Geographic Information Systems
GPS	Global Positioning System
GRC	Gulf Resources & Chemical Corporation
HSP	Health and Safety Plan
IDWs	investigation-derived wastes
LCS	laboratory control sample
LeRoi	LeRoi Company Smelter

LIST OF ACRONYMS (CONTINUED)

Acronym	Definition
MEL	Manchester Environmental Laboratory
mg/kg	milligrams per kilogram
NPDES	National Pollutant Discharge Elimination System
NWI	National Wetlands Inventory
PE	performance evaluation
Pesticides	chlorinated pesticides
PCB	polychlorinated biphenyl
Pintlar	Pintlar Corporation
PM	project manager
PPE	probable point of entry
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QMP	Quality Management Plan
RCW	Revised Code of Washington
RFC	Resource Finance Corporation
RPD	relative percent difference
RSCC	Regional Sample Control Coordinator
SEPA	State Environmental Policy Act
SIS	Sample Information System
SDMS	Site Data Management System
SOPs	standard operating procedures
SOW	Statement of Work
SQAP	Sampling and Quality Assurance Plan
START	Superfund Technical Assessment and Response Team
TAL	Target Analyte List
TDD	Technical Direction Document
TDFs	Tailings Disposal Facilities
TDL	Target Distance Limit

SAMPLING AND QUALITY ASSURANCE PLAN
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION
MINES AND MILLS
NORTHEAST WASHINGTON

TDD: 01-02-0028

1. PROJECT MANAGEMENT

1.1 PROJECT/TASK ORGANIZATION

This section outlines the individuals directly involved with the Upper Columbia River Expanded Site Inspection (ESI) mines and mills sampling and their specific responsibilities. Lines of communication are shown in the Project Organization Chart (Figure 1-1).

1.1.1 Purpose

Pursuant to United States Environmental Protection Agency (EPA) Superfund Technical Assessment and Response Team (START)-2 contract No. 68-S0-01-01 and Technical Direction Document (TDD) No. 01-02-0028, Ecology and Environment, Inc. (E & E) will perform an ESI at the Upper Columbia River properties located in northeast Washington. This ESI will be conducted through a cooperative effort involving START contractor firms, E & E and Roy F. Weston, Inc. (Weston). Weston will conduct sampling activities along the northern reach of the Upper Columbia River and a number of its tributaries. E & E will investigate potential contaminant sources to the northern reach of the Upper Columbia River. EPA has identified approximately 80 mines and mills to be visited and potentially sampled (Table 1-1). Additional mines and mills to be visited and potentially sampled under this ESI will be identified by EPA in an addendum to this Sampling and Quality Assurance Plan (SQAP). Limited sampling will be conducted at mines and mills where potential contaminant source areas are identified. This SQAP outlines the technical and analytical approaches that E & E will employ during the ESI fieldwork.

This document is a combined Field Operations Work Plan (FOWP) and site-specific Quality Assurance Project Plan (QAPP) for field sampling activities. The combined FOWP/QAPP, hereafter called the SQAP, includes a brief project summary; project objectives; sampling and analytical procedures; and QA requirements that will be used to obtain valid, representative field samples and measurements. The SQAP is intended to be combined with information presented in E & E's Quality Management Plan (QMP) for START-2, Region 10. A copy of the QMP is available in E & E's office

located at 2101 4th Avenue, Suite 1900, Seattle, Washington, 98121. Standards contained in the SQAP and QMP will be used to ensure the validity of data generated by E & E for this project. A copy of the site-specific Health and Safety Plan (HSP) will be provided to each field team prior to initiation of sample collection activities and will also be maintained in E & E's Seattle office.

1.1.2 Roles and Responsibilities

The following is a list of key personnel involved in this project, along with their roles and responsibilities.

1.1.2.1 EPA Task Monitor (TM) for E & E

EPA TM Monica Tonel is the overall decision maker and coordinator of the mines and mills sampling activities. The TM reviews and approves the site-specific SQAP and subsequent revisions in terms of project scope, objectives, and schedules. Ensures implementation of site-specific SQAP. The TM is the primary point of contact for general project problem resolution and has approving authority for the project.

1.1.2.2 EPA TM for Weston

EPA TM Mark Ader is responsible for providing technical direction and EPA oversight to WESTON's investigation and sampling activities along the Upper Columbia River and selected tributaries, scheduled for May and June 2001.

1.1.2.3 EPA, Region 10, Quality Assurance (QA) Officer

The EPA QA officer is responsible for reviewing and approving the site-specific SQAP and revisions, and may also conduct assessments of field activities.

1.1.2.4 EPA, Region 10, Regional Sample Control Coordinator (RSCC)

The RSCC coordinates sample analyses performed through the EPA Contract Laboratory Program (CLP) and/or the EPA, Region 10, Manchester Environmental Laboratory (MEL) and provides sample identification numbers.

1.1.2.5 E & E START-2 Project Manager (PM)

The START-2 PM provides overall coordination of fieldwork; provides oversight during preparation of the site-specific SQAP; implements the final approved version of the site-specific SQAP; records any deviations; serves as primary point of contact with the EPA TM; receives CLP/EPA, Region 10, laboratory information from the RSCC; serves as primary START-2 point of contact for any technical problems; and is responsible for the execution of decisions and courses of action deemed appropriate by the TM. In the absence of the START-2 PM, a START-2 alternate project manager will assume the PM's responsibilities.

1.1.2.6 E & E START-2 QA Officer

The START-2 QA officer reviews and approves the site-specific SQAP, conducts in-house audits of field operations, and is responsible for auditing and reviewing the field activities and final deliverables and proposing corrective action for nonconformities if necessary.

1.1.2.7 E & E START-2 Program Manager and EPA Project Officer

Responsible for coordinating resources requested by the EPA TM and for the overall execution of the START-2 program.

1.1.2.8 EPA Investigation and Engineering Unit

The EPA Investigation and Engineering Unit will provide a sampling vessel for sample collection activities. The designated vessel operator is Mr. Doc Thompson.

1.1.2.9 United States Forest Service (USFS)

The USFS has agreed to provide personnel to assist in sample collection.

1.1.2.10 Environmental Services Assistance Team Field Analytical Support Project Team

The Environmental Services Assistance Team, Field Analytical Support Project (ESAT FASP) Team will provide personnel to perform X-Ray Fluorescence (XRF) screening of potential sample locations and to assist in sample collection.

1.1.2.11 MEL or Designated CLP Laboratory

MEL or the designated CLP laboratory will perform the analyses required for the project.

1.2 PROBLEM DEFINITION/BACKGROUND

The EPA has identified approximately 80 mines and mills in northeast Washington to visit and potentially sample in relation to the Upper Columbia River ESI. The field activities will be performed by E & E pursuant to EPA START-2 Contract No. 68-S0-01-01 and TDD No. 01-02-0028. Five of the 80 mines and mills to be visited are currently listed on EPA's Comprehensive Environmental Response, Compensation and Liability Information System (CERCLIS) database. CERCLIS is the database system used by EPA to track its activities at potential hazardous waste sites. Those mines and mills currently identified on the CERCLIS database include the Pend Oreille Mine, Last Chance Mine, Deep Creek Mine, Van Stone Mine and the LeRoi Company Smelter. These sections present a summary of available information discussing the background, operations and characteristics pertinent to the five aforementioned mines and mills.

1.2.1 Pend Oreille Mine

Information presented in this section is based on a review of background information, interviews with the property owner and representatives from various regulatory agencies.

1.2.1.1 Mine Location and Description

This section presents the Pend Oreille Mine location, description, and ownership history.

1.2.1.1.1 Pend Oreille Mine Location

Mine Name:	Pend Oreille Mine
CERCLIS ID No.:	WASFN1002160
Location:	Metaline Falls, Pend Oreille County, Washington
Latitude:	48°52'54.12" N
Longitude:	117°21'35.89" W
Legal Description:	Sections 10, 11, 14, and 15; Township 39N, Range 43E
Property Owner:	Cominco American, Inc. 15918 East Euclid Avenue, P.O. Box 3087 Spokane, WA 99216
Mine Operator:	Cominco American, Inc. P.O. Box 7 1382 Pend Oreille Mine Road Metaline Falls, WA 99153 (509) 446-4516

Property Contact: David Godlewski, Environmental Manager
Cominco American, Inc.
15918 East Euclid Avenue, P.O. Box 3087
Spokane, WA 99216
(509) 892-2584

1.2.1.1.2 Mine Description/Ownership History

The Pend Oreille Mine is a lead, zinc, and silver mine in the Metaline mining district. It is located about 2 miles north of the town of Metaline Falls and approximately 80 miles north of Spokane in northeastern Washington State. The mine is about 11 miles south of the Canadian border and about 15 miles west of the Idaho border. The property is located within portions of Sections 10, 11, 14, and 15; Township 39N; Range 43E (Figure 1-2; Ecology 2000a).

Ownership of the Pend Oreille Mine dates back to 1904, when L.P. Larsen began prospecting in this location. In 1906, the Pend Oreille Mines & Metals Company was incorporated and L.P. Larsen became the president (Lasmanis 1995). The Pend Oreille Mine was owned and operated by the Pend Oreille Mines & Metals Company until 1974, when the Bunker Hill Company acquired the property and operations. The Bunker Hill Company operated the mine and mill until September 1977, when the property was acquired by the Pintlar Corporation (Pintlar), a wholly-owned subsidiary of the Gulf Resources & Chemical Corporation (GRC). In 1988, the Resource Finance Corporation (RFC) obtained an option-purchase agreement from Pintlar, de-watered the mine, and commissioned a feasibility study for mining and milling the East Side Yellowhead deposit. In May 1990, RFC purchased the mine and mill along with 13,000 acres of contiguous mineral holdings in the district (Ecology 2000a). In 1996, Cominco American, Inc. (Cominco) acquired a portion of this property, including the Pend Oreille mine and mill, from RFC (Ecology 2000a).

The Pend Oreille Mine property is included in the 580 acres of Cominco-owned or leased surface lands (including mineral rights) and 980 additional acres of mineral rights in this area (Ecology 2000a). The Pend Oreille Mine is bounded on the west by the Pend Oreille River, on the north and northeast corner by Colville National Forest lands, on the east by privately-owned lands, and on the south by State Route 31 and the Grandview Mine property (USGS 1992a, 1992b).

Existing Pend Oreille Mine facilities on the east side of the river include the mine office building, maintenance shop/blacksmith shop/surface conveyor/portal, coarse and fine ore storage silos, mill, assay office building, and storage facilities (Figures 1-3 and 1-4).

The mine office building is a two-story reinforced concrete frame structure with concrete block walls. The building has 5,500 square feet per floor. The lower floor is used for offices, accounting, and lab facilities; the upper floor is used for storage, a training room, and two offices.

The maintenance shop/blacksmith shop is a 10,500-square foot, 30-foot-high, reinforced concrete building with a flat roof. The conveyor that carries the ore to the coarse ore storage silo is an elevated, steel-frame, integral truss structure supported on steel bents. The shop contains machinery, tools, welding equipment, electrical equipment, lubricants, and chemicals.

Two coarse ore and three fine ore storage silos are on the mine property. The capacity of each ore silo is approximately 1,000 tons.

The mill is a four-story concrete block building with steel interior framework. Mill equipment includes three primary head ball mills, two regrind ball mills, flotation cells, lead and zinc thickeners, stock tanks, a water filter, and a concentrate storage area. The assay office building consists of a lower story used for storage, and an upper story containing laboratory facilities.

Other buildings on the mine property include storage sheds; a powder magazine; a make-up house; a car barn/carpenter shop; a core storage shed; a crusher building; an upper, main, and mill substation; and a compressor house.

A waste rock pile from historic mining activities is located just southwest of the ore storage silos and mine office. Three tailings disposal facilities (TDFs) from historic mining activities are located on the mine property. The Metaline Falls Golf Course and Gun Club is also located within the property boundary, just east of TDF No. 2 (TDF-2; Figure 1-4; Ecology 2000a).

1.2.1.2 Mine Operations and Source Characteristics

The Pend Oreille Mine was one of the earliest, largest, and most productive mines in the Metaline mining district, accounting for over 50 percent of the lead and zinc production in the area (Ecology 2000a).

Two ore horizons are present at the Pend Oreille Mine; the Josephine and the Yellowhead. The Josephine Horizon is found in the upper 500 feet of the Metaline Limestone, about 1,600 feet below the surface. The Yellowhead Horizon is beneath the Josephine Horizon and is generally found from 1,000 to 2,400 feet beneath the top of the Metaline Limestone (Ecology 2000a).

The primary method for extracting ore in the Metaline mining district, including the Pend Oreille Mine, was underground mining. Historically, most ore extraction was from the Josephine Horizon, which is exposed at the surface at Pend Oreille River. This horizon has been mostly mined out.

Mining in the area by the Pend Oreille Mines & Metals Company was on a small scale during the early years, beginning on the Josephine claim with near-surface deposits on the west side of Pend Oreille River; mining continued here intermittently from 1904 until 1917 (Dings and Whitebread 1965). In 1917, a table concentration mill was constructed and operated for a few months, but was shut down during World War I. There was little activity for the next 10 years.

In 1927, the old table mill was remodeled to a flotation plant which operated on surface ores at 150 tons per day for approximately six months. In 1928, Pend Oreille Mines & Metals Company began additional deeper development of the area; a program of extensive diamond drilling from 1928 to 1930 disclosed promising ore bodies. With a 300-ton flotation mill completed in anticipation of treating this ore, mining resumed in November 1930 and continued intermittently until 1937. The underground workings were developed, with ore hauled to the surface through a network of track haulage drifts and inclined shafts. In September 1937, a power plant on the Pend Oreille River was completed, the mill was overhauled; and production was increased to approximately 600 tons per day (Lambly 1941; Zeigler 1949; Dings and Whitebread 1965).

Subsequent workings on the Josephine Horizon were conducted eastward and downward, crossing under the river in the 1940s. Approximately 14.4 million tons of ore at 1.2 percent lead and 2.7 percent zinc were removed from this horizon between 1938 and 1977 (RFC 1993).

Mining of the Yellowhead Horizon on the east side of the river began in 1972 at about 650 to 900 feet below the Josephine workings. Only 387,000 tons of ore containing 0.5 percent lead and 4.0 percent zinc were removed from this horizon prior to closure of the mine in 1977. Since 1977, there have been no mining operations other than a development program (Lasmanis 1995; Godlewski 2000).

Prior to 1967, tailings from the milling operation-the finely ground sand and silt material remaining after the target minerals have been separated during beneficiation-were disposed of directly into the Pend Oreille River. Beneficiation includes those activities that serve to separate and concentrate the mineral values from waste material, remove impurities, or prepare the ores for further refinement (EPA 2000a). An estimated 3,000 tons of tailings were discharged to the Pend Oreille River each day of milling operations in 1962 (Neale 1962). After 1967, three upland TDFs were used to dispose of the tailings. TDF No. 1 (TDF-1), which is located 2,400 feet northeast of the mill and west of the Metaline Falls Golf Course and Gun Club, covers approximately 24 acres. This facility was used from 1967 through 1974. TDF-2, which is located between the golf course and TDF-1, covers approximately 25 acres; this facility was used during 1974 and 1975. Tailings in these two TDFs are comprised of material from the Josephine Horizon workings (Ecology 2000a).

TDF No. 3 (TDF-3) is located south of the golf course and covers approximately 20 acres. TDF-3 was operated from 1975 until the mine closed in 1977. The tailings in this facility are approximately 40 feet deep at the deepest point and are comprised primarily of material from the Yellowhead Horizon workings (Ecology 2000a). The Pend Oreille River is approximately 3,500 meters west of TDF-3 (Figure 1-4).

Historically, waste rock-the waste material consisting of non-mineralized and low-grade mineralized rock removed from, around, or within the orebody during extraction activities-was stored underground during early operations. From 1950 to 1977, waste rock from both the Josephine and Yellowhead horizons, as well as Ledbetter Slate, was removed from the mine by conveyors from the Josephine Horizon to the surface, and stored in the waste rock pile located 150 feet south and west of the mill. Most of the waste rock has a low pyrite content and consists of carbonate material (limestone and dolomite) removed from above and below the ore horizons that were mined. Some of the waste rock brought to the surface was sold and used locally as road material or in construction from at least the mid-1970s until the early 1990s (Ecology 2000a). The waste rock was used as a road construction base, subbase, and surface material; for private driveways; as parking lot material at Gardner Cave State Park; and as backfill by the county. The total amount of waste rock used for these purposes is unknown. The waste rock pile currently consists of approximately 386,000 cubic yards of waste rock, and covers about 15 acres (Ecology 2000a).

Between 1977 and 1986, Bunker Hill Company, Pintlar, and GRC continued exploration in the area and operated pumps to prevent flooding of the mine by underground water. The mine water was discharged into the Pend Oreille River. The mine was allowed to flood between 1986 and 1988, at which time development ceased (Ecology 2000a).

After purchasing the property in 1990, RFC submitted a proposal to the Washington State Department of Ecology (Ecology) in 1992 to reopen the mine, and submitted a State Environmental Policy Act (SEPA) checklist for the proposed project in February 1993. Ecology reviewed the SEPA checklist and issued a "Determination of Non-Significance" in April 1993. RFC then submitted several applications to Ecology to obtain permits required to reopen the mine. By 1994, RFC obtained four permits (stormwater discharge, dam safety, septic system, and air quality) and was close to receiving two others (National Pollutant Discharge Elimination System [NPDES] and waste discharge) when RFC decided to cancel the project (Ecology 2000a). Cominco currently maintains the following permits: NPDES, Waste Identification, Stormwater General, Ground Water, State Dangerous Waste Conditional Exemption, and Dam Safety (Godlewski 2001).

Hazardous waste from earlier mining operations and stored on the Pend Oreille Mine property were removed from the property during RFC's tenure in 1992 (Ecology 2000a). These wastes included polychlorinated biphenyl (PCB) oil-cooled transformers and capacitors, as well as soils containing PCBs (Ecology 2000a). The data reviewed did not specify waste quantities or disposal locations. Oil, lubricants, light fuels, antifreeze, and herbicides are reportedly the only hazardous materials currently stored on the property. The materials are stored in a shed located between the administrative office and the machine shop.

Cominco acquired the property in 1996, and submitted the document *"Statement of Proposal Pend Oreille Mine, Pend Oreille County, Washington"* to Ecology on January 29, 1998, in order to reopen the mine. Based on the proposal, Ecology determined that an Environmental Impact Statement (EIS) would be required before the mine could be reopened (Ecology 2000a).

Draft and final EIS's have been produced by Cominco regarding reopening the mine. Cominco is currently drafting an engineering plan for the tailings disposal facility. Cominco plans to open the mine within a few of years; Cominco will have to apply for another NPDES permit before opening. Cominco is currently performing construction activities but lead and zinc mining has not been approved. (Hallinan 2001)

1.2.1.3 Mine Area Characterization

This section summarizes previous investigations (Section 1.2.1.3.1), discusses migration/ exposure pathways and targets (Section 1.2.1.3.2), and describes areas of potential contamination (Section 1.2.1.3.3).

1.2.1.3.1 Previous Investigations

Cominco has proposed reopening the Pend Oreille Mine. Proposed activities include extracting ore-bearing rock from underground mine workings and transporting the ore to the surface via the existing conveyor system. Zinc and lead-bearing concentrates would be recovered at the existing on-site mill, with the metal-bearing concentrates shipped by truck to the Cominco Ltd. Smelter in Trail, British Columbia. As required for major actions significantly affecting the quality of the environment (Washington Administrative Code [WAC] 197-11-330) or for any proposed metal mining and milling operation in Washington State (Revised Code of Washington [RCW] 78.56.050), Cominco prepared an EIS document which was issued in draft form on February 24, 2000, and finalized in July 2000. The EIS documents the process used to analyze the Proposed Action, alternatives to the Proposed Action,

environmental impacts, and mitigation measures with regards to extracting ore-bearing rock from underground mine workings and transporting the ore by truck to the Cominco Ltd. Smelter in Trail, British Columbia.

As part of preparing the EIS, testing programs were implemented by Cominco and its consultants to evaluate waste rock and tailings. The following tests were performed on both waste steams:

- EPA toxicity characteristic leaching procedure for Resource Conservation and Recovery Act metals which include arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver;
- Synthetic precipitate leaching procedure;
- Static acute fish toxicity test;
- Whole rock radionuclide analysis;
- Static acid-base accounting;
- Humidity cell test;
- Sub-aqueous column leach test; and
- Tailings liquid analysis.

The results are summarized in the EIS (Ecology 2000a).

The START conducted a visit to the mine on May 18, 2000. The START was accompanied by David Godlewski, the Cominco environmental manager. The mine was accessed by a gravel road off State Route 31, where a gate is open during work hours but closed in the evening to ward off trespassers. The START observed several formerly active buildings previously described and noted one portal on the mine property. Additionally, a waste rock dump area was observed about 150 feet south and west of the mill. No liquid was observed discharging from the waste rock pile. Cominco reported that they currently use the waste rock on roads around the mine and mill (Godlewski 2000). The waste rock pile currently consists of approximately 386,000 cubic yards of waste rock, and covers about 15 acres (Ecology 2000a).

Three tailings disposal facilities (TDF-1, TDF-2, and TDF-3) were observed. There are no containments or berms at any of the tailings disposal facilities. Cominco personnel noted that the TDFs are located approximately 50 feet above groundwater (E & E 2000).

The tailings disposal facility (TDF-1) was reportedly seeded and fertilized by a previous owner, Bunker Hill Company, and is currently covered with vegetation. In the spring of 1998, Cominco installed peripheral trenches along the eastern boundary of the facility to divert water around TDF-1 and to allow the tailings materials to dry. A pool, which is used by bats and other wildlife, has developed

along a portion of the ditch and may form wetland habitat in the future; however, the peripheral trench has reduced much of the water that flows onto TDF-1 and it is likely that most of the wetland habitat associated with TDF-1 will revert to more upland habitat over time (Ecology 2000a). The START estimated the flow rate of the drainage from peripheral trenches into the Pend Oreille River to be approximately 50 gallons per minute (gpm). The maximum tailings depth is estimated at 62 feet, and the volume is estimated at 1,149,721 cubic yards (E & E 2000; Godlewski 2000).

TDF-2 was also seeded and fertilized by the Bunker Hill Company. At the time of the START visit, only half of the TDF-2 area was covered with vegetation, while the other half was covered in tire tracks from suspected recreational use of 4x4 vehicles or motor bikes. The maximum depth is estimated at 20 feet and the volume is estimated at 300,000 cubic yards (Godlewski 2000).

TDF-3 covers approximately 20 acres south of the golf course and was fenced to ward off unauthorized recreational use of the property. The Pend Oreille River is approximately 3,500 meters west of TDF-3, which was apparently never reclaimed or seeded and presently does not support vegetation. There was no surface drainage noted. The maximum depth is estimated at 40 feet. The volume is estimated at 1,290,770 cubic yards.

The START observed wetlands, springs, seeps, and creeks associated with the mine property. There was no evidence of tailings or waste rock deposition in the wetlands, seeps, or creeks. Prior to 1967, the tailings were deposited directly into the Pend Oreille River (Ecology 2000a).

At TDF-2 and TDF-3, the START observed animal tracks and feces as well as tire tracks that appeared to be from 4x4 vehicles or motor bikes. No stressed vegetation or discoloration was noted by the START.

During the visit, the START observed two 10- by 10-foot plastic storage tanks leaking on the ground adjacent to the south side of the mill. There appeared to be a plastic secondary containment, with approximately 4 inches of blue-green standing water. The soil was stained a blue-green copper color.

1.2.1.3.2 Groundwater and Surface Water Migration Pathway and Targets

This section discusses the groundwater and surface water migration pathway and potential targets within the mine's range of influence. The groundwater migration, soil exposure, and air migration pathways are not being evaluated in this ESI (Figures 1-5 and 1-6). The groundwater migration pathway for the Pend Oreille Mine is included in the SQAP based on the sampling scheduled at the well and adit at the mine.

1.2.1.3.2.1 Groundwater Migration Pathway and Targets

The Pend Oreille Mine is underlain by Metaline Limestone, which is overlain by Ledbetter Slate. On the surface above the mine workings, a very large part of the Ledbetter and some of the Metaline is concealed beneath Quaternary lake deposits that range in thickness from a thin veneer to 200 feet or more. This glacial covering is especially extensive east of Pend Oreille River. Most of the carbonate rock is medium-to-dark-gray crystalline dolomite, commonly having a medium- to coarse-grained texture, but some very fine-grained, gray limestone is exposed at many places in both the eastern and western mine workings. Some dolomite is creamy, light gray, or black, and locally the grain size is very fine (Ecology 2000a).

The uppermost 25 feet of the Metaline commonly contains irregular zones of dark-gray to black crystalline limestone, although at other places all rock in this position is dark crystalline dolomite. Black shaley partings and some shale lenses a few inches thick are locally present in these upper rocks. At many places altered rock is in the form of irregularly-shaped masses of dolomite breccia that range widely in size and grade imperceptibly into non-brecciated dolomite. As a very general statement it can be said that the uppermost 150 feet of Metaline Limestone in the Pend Oreille mine area is crystalline dolomite that overlies gray limestone, although at places gray massive limestone is found 50 feet or less below the slate (Ecology 2000a).

Groundwater at the Pend Oreille Mine occurs within both the Paleozoic bedrock formations and the overlying glaciofluvial and glaciolacustrine sediments. An assessment of groundwater flow at the Pend Oreille Mine was based on studies of groundwater in the mine and monitoring wells (Ecology 2000a). The Paleozoic bedrock is composed of carbonate reef deposit sediments.

Underground mine workings are in the Paleozoic bedrock. Because underground mine development has occurred primarily in carbonate rocks that are part of the bedrock, groundwater seeps are found throughout the mine below the water table. The mine workings are below the level of Pend Oreille River, allowing for seepage into the mine by both upgradient groundwater from the highlands and from the river.

Currently, all water from the Josephine Horizon flows through ditches and natural fissures, or is pumped to a sump located near the 900-foot level of the mine. The total flow measured from these sources is approximately 80 gpm. Water from the Yellowhead Horizon is directed to one of two sumps (the 440-foot level sump or the 700-foot level sump), and is then pumped to the sump located near the 900-foot level of the mine. Total flow from these two sumps is approximately 220 gpm (Ecology 2000a).

Water from the sump located near the 900-foot level of the mine is pumped to the 1,700-foot level sump, which is then pumped directly into Pend Oreille River (probable point of entry [PPE] 1).

Monitoring well water levels indicate that the groundwater beneath TDF-3 flows from south to north beneath TDF-3 and then flows to the northwest to Pend Oreille River. The groundwater gradient beneath TDF-3 is about 0.02 foot of elevation/distance. The groundwater is principally within the glacial alluvial sediments but can be found in some monitoring wells at the interface between the bedrock and the glacial alluvium. The aquifer beneath the tailings facility is probably unconfined based on Ecology studies (Ecology 2000a).

Information regarding net precipitation in the immediate area of the Pend Oreille Mine is not available. The annual net precipitation factor value reported for Metaline Falls is 27.38 inches (WRCC 2000a).

A domestic water supply exists for workers at the mine and mill; its origin is from a well located approximately 1,000 feet southwest of the main office building. It is also possible that well water quality is influenced by seepage from the adjacent waste rock pile (Ecology 2000a). The mine is apparently not located within 1 mile of any sole-source aquifer areas. There are no documented Washington public groundwater supply wells within 4 miles and there is no data available regarding the presence of public surface water supplies within the Target Distance Limit (TDL; Ecology 2000a). The mine is not expected to be in a wellhead protection area.

1.2.1.3.2.2 Surface Water Migration Pathway

Surface water resources in the vicinity of the Pend Oreille Mine include the Pend Oreille River, creeks that transect the mineralized areas of the Metaline District and provide water to the river, and lakes that are usually found within the glacial sediments and are frequently associated with wetlands. Boundary Dam was constructed across the Pend Oreille River at the United States-Canada border in 1967, causing the Pend Oreille River to back up behind the dam and create Boundary Reservoir. The average water level in the reservoir near the mine is at an elevation of 1,990 feet (Ecology 2000a).

Surface water from hill slopes to the east of the Pend Oreille Mine flows in a westerly direction to the Pend Oreille River. The Pend Oreille River is part of a drainage system that includes parts of Montana, Idaho, Washington, and Canada. The upper reach of the river in Montana is known as Clark Fork, which drains into Idaho and enters Lake Pend Oreille. Pend Oreille River exits the lake and flows into Washington State at Newport.

High river flows occur in May and June during peak snowmelt runoff. The lowest flows occur between July and November. Average annual flow at Newport, Washington, is 25,500 cubic feet per second (cfs). The average July through November flow is 19,100 cfs and the seven-day low flow with a recurrence interval of once every 10 years is 4,500 cfs (Ecology 2000a).

Surface water flows are generated from precipitation in the form of rainfall or snowfall and at locations where groundwater is exposed at the surface. Precipitation at the Pend Oreille Mine averages 27.38 inches annually. The two-year, 24-hour rainfall event for the Pend Oreille Mine area is 2 inches (WRCC 2000a). The drainage area for the waste rock pile at the mine is estimated to be 11.235 acres (USGS 1992a, 1992b).

The Pend Oreille Mine is located within the Metaline Falls watershed. The watershed covers approximately 11,585 acres and is primarily drained by Threemile Creek, which flows year-round and eventually cascades into the Pend Oreille River. Elevations within the watershed range from 1,990 feet at the Pend Oreille River to 6,830 feet in the mountains (Ecology 2000a).

Several creeks transect the mine area. Threemile Creek, which flows year-round and has flow rates of a few hundred gallons per minute or higher depending on the time of year, is located near the northern border of the mine and adjacent to United States Forest Service (USFS)-managed lands (Figure 1-4). Threemile Creek is primarily confined to a single channel along most of its length within the mine area, and forms a large forested wetland used by beavers near the northeastern boundary of the mine (Ecology 2000a).

Two creeks are found near TDF-1. It is unknown if the flow in these creeks is perennial or intermittent. One creek (Creek 1) originates to the east of the mine, near the golf course. Water flows within a well-defined channel along most of its length during most of the year and empties into the Pend Oreille River (Ecology 2000a).

Two seep areas are located near TDF-2 and TDF-3. The water in these seeps appears to originate from hill slopes and from TDF-2. This seepage is collected in diversion channels and routed around and to the base of TDF-1, where it meets with natural seepage. This water then flows downslope (Creek 2) and drains into the Pend Oreille River (PPE 2). Surface water runoff from TDF-1 and TDF-2 drains into installed diversion ditches which drain into the Pend Oreille River (Ecology 2000a).

A third unnamed creek (Creek 3) originates off the property east of the 9th/18th fairway of the Metaline Falls Golf Course. It is unknown if the flow in this creek is perennial or intermittent. It flows approximately 160 feet through a wetland on the edge of the fairway, and for an additional 150 feet on the fairway before flowing underground (Ecology 2000a).

Frog Creek originates approximately 900 feet downslope from TDF-3 as a groundwater spring and seep. Frog Creek derives its water from groundwater flowing beneath the kettle basin that holds TDF-3 and from precipitation; it has a perennial flow. Initially, water from the spring flows into several channels, which then join into a single channel approximately 400 feet downstream from the source. This channel flows downhill until it intersects an access road that leads to TDF-1, then flows northerly along this road, and then westerly under the road into a small pond. The flow rate is estimated by Ecology to be 40 gpm. The creek flows from the pond in a northwesterly direction until surface flow terminates approximately 100 feet downslope from the pond. The creek resurfaces approximately 50 feet downslope from the southern mine access road that parallels the Pend Oreille River. The water collects into two channels and eventually flows into the Pend Oreille River (Ecology 2000a).

Several other creeks are near the mine property. It is unknown if the flow in these creeks are perennial or intermittent. On the east side of the Pend Oreille River, Lime Creek and Slate Creek are found to the north, and Sullivan Creek to the south of the mine. Flume and Beaver creeks are important tributaries of the Pend Oreille River that drain into the river from the west and are also near the mine (PPE 3 from the confluence of Flume Creek to the Pend Oreille River). Surface water runoff from the staged waste rock may drain by sheet flow into Flume Creek which drains into the Pend Oreille River. Since 1992, Cominco has routinely sampled water quality at eight locations, including Sullivan, Flume, Threemile, and Frog Creeks and the Pend Oreille Reservoir upstream and downstream from the mine (Ecology 2000a).

Each of the PPEs described in this report are located along the Pend Oreille River. Approximately 8 miles of the 15-mile TDL are on the Pend Oreille River between the PPEs at the mine and Boundary Dam; 1 mile covers the final section of the Pend Oreille River from Boundary Dam to the Canadian border; and the remaining 6 miles are on the Pend Oreille River in Canada.

Within the 15-mile TDL, it is estimated that 0.043 mile of linear wetland is present. In addition, wetlands were identified by Ecology at five locations on the property (Figure 1-4). These include natural wetlands associated with Threemile Creek, Metaline Falls Golf Course and Gun Club, Frog Creek, and two artificially created wetlands associated with TDF-1. The Threemile Creek wetland is located along Threemile Creek, near the northeastern corner of the mine. It covers approximately 1.5 acres and is comprised of forested and emergent wetland habitat types. Beavers have constructed three dams near the outlet of the wetland, creating a pooled area that is several feet deep. One dam that is in disrepair and two newer beaver-maintained dams block Threemile Creek and provide aquatic bed and emergent wetland habitat for beaver and other wildlife. A beaver lodge is located near the outlet of the wetland

and is constructed of beaver cuttings and soil. Duckweed covers portions of the open water areas, while sedges dominate more shallow areas. Western red cedar and western hemlock (up to 24 inches in diameter at breast height), as well as willow, comprise the edges of the wetland; red osier dogwood is also common along the edges (Ecology 2000a).

Two wetlands are associated with TDF-1. One artificially-created wetland is found along the eastern side of the tailings facility and the base of the adjacent hill slope, and has developed from tailings saturated by runoff from the adjacent hill slope and TDF-2. The wetland covers approximately 2 acres and contains sedges, smartweeds, cat-tails, and scattered willows. In the spring of 1998, Cominco installed peripheral trenches along the eastern boundary of the facility to divert water around TDF-1 and to allow the tailings materials to dry (Ecology 2000a).

A second, small (0.01 acre) wetland is associated with runoff that used to flow to this area from the base of TDF-1, and from water that has collected in the peripheral ditches on the upslope side of TDF-1 and routed around the tailings facility to the base of the facility. Water draining from the base of the facility used to flow down to an old logging road, through a culvert, and under the mine road located adjacent to the Pend Oreille River. The small wetland developed near the intersection of these two roads. Water is currently being diverted into a new channel and no longer flows into this wetland (Ecology 2000a).

A small (0.01 acre) wetland is found between TDF-1 and TDF-2, at the base of the TDF-2 embankment. Natural seepage and seepage from TDF-2 supply water to this wetland (Ecology 2000a).

A wetland is associated with Creek 3 which flows onto the mine property along the eastern boundary of the area and near the 9th/18th fairway on the golf course. The creek originates near State Route 31 and is approximately 160 feet in length from the property boundary to the edge of the golf course fairway. The wetland is approximately 30 feet wide in the vicinity of the creek, resulting in a total wetland acreage of approximately 0.1 acre. The wetland is dominated by nearly complete herbaceous cover of small-fruited bulrush and smartweed; shrubs consisting of willow, thimbleberry, vine maple, currant, and dogwood; and trees comprised of cottonwood, paper birch, and red alder. The creek continues along the fairway for another 150 feet before flowing underground. The creek is bordered by mowed grasses on the fairway (Ecology 2000a).

Springs and seeps located approximately 900 feet downslope from TDF-3 are the source of most of the water for Frog Creek and associated wetlands. Most of the wetland area is associated with the upper portions of the creek, and comprises approximately 1.5 acres (Ecology 2000a).

One fish species present in the Pend Oreille Mine area has been listed as threatened by the federal government; bull trout (*Salvelinus confluentus*), a Federal listed threatened species found in the Columbia River basin distinct population segment, was listed as a threatened species by the United States Fish and Wildlife Service on June 10, 1998. Two bird species present in the Pend Oreille Mine area have been listed as threatened or endangered by the state or federal government. The bald eagle (*Haliaeetus leucocephalus*; a Federal listed threatened species) inhabits areas along the shores of saltwater and freshwater lakes and rivers and typically nests in mature or old growth trees. An active bald eagle nest is located a few miles upstream from the mine along the Pend Oreille River near Sand Creek. The American peregrine falcon (*Falco peregrinus anatum*; a state listed endangered species) typically lives along mountain ranges, river valleys, and coastlines. Peregrine falcons may occasionally fly along the Pend Oreille River near the mine (Ecology 2000a).

Five mammal species in the Pend Oreille Mine area have been listed as threatened or endangered by the state or federal government. Gray wolves (*Canis lupus*; a Federal listed endangered species) have been sighted and confirmed in the Colville National Forest, although these are likely lone dispersing wolves. Camera surveillance has not detected wolves, and no systematic surveys for wolves have been conducted. Grizzly bears (*Ursus arctos*; a Federal listed threatened and state listed endangered species) are present throughout the Colville National Forest, although the number of bears is lower near Lake Sullivan and the mine. A grizzly bear den has been found on the Colville National Forest, east of the mine near the Idaho border, and grizzly bears have been radio-located on both sides of the Pend Oreille River. The mine is adjacent to a designated grizzly bear recovery area, which begins east of State Route 31. The North American (Canada) lynx (*Lynx canadensis*; a proposed Federal listed threatened state listed threatened species) may travel through the mine area; tracks of a lynx were found in the Slate Creek drainage in 1978. The Pacific fisher (*Martes pennanti pacifica*; a Federal listed species of concern and state listed endangered species) is a large, weasel-like animal. There have been unconfirmed sightings in the mine area, and it is believed that the fisher may be present around Sullivan and Slate creeks, as well as Sullivan Lake. The woodland (mountain) caribou (*Rangifer tarandus caribou*; a Federal and state listed endangered species) could potentially enter the mine area, but all woodland caribou that have been previously sighted on the mine area were transplanted animals that had been released nearby and were returning to the release site. There is a woodland caribou recovery area which includes mostly wilderness area east of the mine, within 25 miles south of the United States-Canada border, and further east into Idaho (Ecology 2000a).

1.2.2 Last Chance Mine

Information presented in this section is based on a review of background information, interviews with the property owner and representatives from various regulatory agencies.

1.2.2.1 Mine Location and Description

This section presents the Last Chance Mine location, description, and ownership history.

1.2.2.1.1 Last Chance Mine Location

Mine Name:	Last Chance Mine
CERCLIS ID No.:	WASFN1002162
Location:	Stevens County, Washington
Latitude:	48°51'59.52" N
Longitude:	117°41'56.39" W
Legal Description:	Section 24, Township 39N, Range 40E, Willamette Meridian
Property Owner:	Vaagen Brothers Lumber Company 565 W 5th Colville, WA 99114
Property Contact:	Mr. Mick Vaagen, Owner 565 W 5th Colville, WA 99114 (509) 684-5242

1.2.2.1.2 Mine Description/Ownership History

The Last Chance Mine is an inactive underground silver, lead, and zinc mine located east of Deep Creek, in Section 24, Township 39N, and Range 40E in Stevens County, Washington (Figure 1-7). The mine is located approximately 5 miles southeast of Northport, Washington. From Northport, the mine is accessed via Colville-Aladdin Northport Road, which runs on the west side of the mine property (Figure 1-8).

During a visit to the mine on May 4, 2000, the START observed a former 60-ton per day gravity flotation mill, a former house or office, and two shafts and one adit approximately 0.25 mile upgradient from the mill. Additionally, there was a large tailings pile surrounding the mill and two waste rock deposits below the adit and shafts. The tailings pile was estimated to be 7,000 cubic yards in volume, and the waste rock deposits were estimated to be 7,300 cubic yards and 125 cubic yards in volume,

respectively. Besides the flotation mill, no other mill equipment remains on the mine property (Huntting 1956).

The mine is currently owned by Vaagen Brothers Lumber Company. Previous owners include the Juniper Lead Company, which owned the mine from 1907 to 1926. Reports indicate that in the period of 1938 the property was owned by Mrs. A. Baker, the widow of owner Al Baker. Last Chance Consolidated Mines, Inc. owned the mine from 1948 to an undetermined date (Huntting 1956). Additional information regarding previous ownership history is not available.

1.2.2.2 Mine Operations and Source Characteristics

The mine was operated from approximately 1904 to 1954, when the mine was shut down (Huntting 1956). The construction of the flotation mill is reported to have begun in 1940. Mine tailings were created as a result of the milling process. No information is available about the chemical processes used at the mill, but it is assumed that mercury amalgamation and/or cyanide leaching processes were used. Contaminants of concern at the mine include heavy metals and acid mine drainage discharging into Deep Creek from the tailings and adit. It is estimated that approximately 7,000 cubic yards of tailings and 7,425 cubic yards of waste rock are present on the mine property.

1.2.2.3 Mine Area Characterization

This section summarizes previous investigations (Section 1.2.2.3.1), and discusses migration/ exposure pathways and targets (Section 1.2.2.3.2).

1.2.2.3.1 Previous Investigations

The START conducted visit to the mine on May 4, 2000. Mr. Vaagen, the property owner, was not present during the visit. The START was accompanied by a Colville Tribe representative, Patti Stone, and its environmental consultant from Fulcrum Environmental Consulting, Inc. The START observed a former mill building. Two shafts and one adit were approximately 0.25 mile upgradient from the mill (Figure 1-9). Additionally, there was a large tailings pile surrounding the mill and two waste rock deposits below the adit and shafts. The tailings pile was estimated to be approximately 7,000 cubic yards in volume, and the waste rock deposits were estimated to be 7,300 cubic yards and 125 cubic yards in volume, respectively. An unnamed creek was observed to run through the middle of the tailings pile for approximately 600 feet. Upstream, a side channel of the unnamed creek also was observed to run through the smaller waste rock pile for approximately 50 feet. The creek water flowing through both

piles did not appear discolored, showed no signs of a precipitate, and did not show evidence of an overabundance of algal growth.

The tailings in the vicinity of the mill appeared to be smaller grained the further downgradient they were deposited from the mill. This may be due to the processing of the ore as it proceeded through the mill. The mill building itself was severely dilapidated, with few remaining walls and the roof structure leaning down to the ground. The foundation of the mill is multi-leveled to accommodate different steps of the milling process. The existence of electrical conduit and junction boxes indicate that there had been some form of electricity on the mine property. Inside the mill building the START observed a small pile of white powder on the ground, approximately 1 cubic yard in volume. The mill is approximately 10 yards from the unnamed creek. It is unknown if the flow in this creek is perennial or intermittent.

From the mill, the adit and the shafts are approximately 0.25 mile upslope along the unnamed creek. Along the slope, between the mill and the adit, there is evidence of mining activity, such as ore buckets and rail tracks. Water was observed flowing from the adit at an estimated rate of 60 gpm. The discharge was not discolored and showed no signs of a precipitate on the wet surfaces. Approximately 3 feet to the south of the adit discharge point there was an area of ponded water that contained an abundance of long fibrous algal growth, which can be indicative of a low pH.

There are two shafts, one located to the north of the unnamed creek and the other located to the south. A dilapidated wooden building was observed on top of the north shaft, which was not further investigated due to safety reasons. The START did not observe any discharge from this shaft. No water was discharging from the south shaft, but a flowing seep was observed approximately 15 feet downgradient of the shaft. This seep is most likely hydrologically influenced by the unnamed creek, which flows approximately 6 feet north of it. The creek forms many channels in this area. In one of the channels, tadpoles were observed by the START.

In the vicinity of the north shaft there is an abandoned road that appears to have been used for the transport of waste rock from the shafts and adit to a dump site. The waste rock was dumped downhill from this road. The waste rock pile created by this dumping was observed by the START to be approximately 0.25 mile long and 25 yards wide. The START did not observe any water discharging from the waste rock or evidence of former water runoff or discharge. The START also observed cow dung on the property, indicating that grazing has occurred on the mine property.

The START observed that the unnamed creek infiltrates the ground approximately 20 yards upgradient from the Colville-Aladdin Northport Road. There was evidence of channels where this creek,

during periods of high flow, has flowed into a drainage ditch along the east side of the road. This drainage ditch flows south, approximately 100 yards to a culvert, which directs the flow beneath the road and discharges it into a field. Once the flow reaches the field, it either infiltrates the ground or discharges to Deep Creek by sheet flow.

1.2.2.3.2 Surface Water Migration Pathway and Targets

This section discusses the surface water migration pathway and potential targets within the mine's range of influence. The groundwater migration, soil exposure, and air migration pathways are not being evaluated in this ESI (Figures 1-10 and 1-11).

Surface water from the adit drains overland for approximately 20 feet to the southwest where it discharges into the unnamed creek (PPE 1) (Figure 1-9). The discharge rate of the adit is estimated at 60 gpm. From this point, the unnamed creek flows to the west for approximately 0.25 mile until it reaches the tailings pile at the mill. The unnamed creek flows through the tailings pile for approximately 200 yards (PPE 2) and then continues to the west, toward Deep Creek. The unnamed creek flows for approximately 0.1 mile from the bottom of the tailings pile into a field where it infiltrates the ground approximately 20 yards upgradient from the road. The START observed dry channels where the creek flows, during high water, to a drainage ditch along the road. Observations show that the water would then flow for approximately 100 yards to a culvert which diverts the water beneath the road into the above-mentioned field. From the field, surface water is expected to flow by sheet flow approximately 0.1 mile into Deep Creek. From PPE 2, the 15-mile TDL extends approximately 0.2 mile along the unnamed creek to its confluence with Deep Creek, and from Deep Creek approximately 6.5 miles to its confluence with the Columbia River. The remaining 8.3 miles of the TDL is along the Columbia River.

The mean annual precipitation at Northport for 1920 through 1999 is 19.46 inches (WRCC 2000b). The two-year, 24-hour rainfall event for the mine area is 1.6 inches (WRCC 2000c). It is expected that the mine lies outside of a floodplain. As stated previously, the area consists of Waits-Rock outcrop complex type soils. This soil is a loam with moderate permeability, rapid runoff, and a high hazard of water erosion (USDA 1982). The upland drainage area of the mine is estimated from a topographic map to be 290 acres (USGS 1992c).

There are 10 surface water intakes within the 15-mile TDL of the mine. Nine of these are private domestic intakes and one is a municipal intake for the City of Northport (Ecology 2000b). The nine domestic intakes are estimated to serve 25 people. This number is based on the average number of persons per household for Stevens County, which is 2.73 (USBC 1990). The closest domestic intake is

located 1.1 miles from the mine. The municipal intake at Northport serves 460 people (EPA 2000b). Surface water is used within the TDL for irrigation of commercial food or forage crops and watering of commercial livestock (Ecology 2000b).

Flow rate data for the unnamed creek and Deep Creek are not available; however, based on visual observations during the START visit, the flow for the unnamed creek was estimated to be less than 1 cfs, and the flow for Deep Creek was estimated to be approximately 187 cfs. The average annual flow rate for the Columbia River, measured at Northport, Washington, is 103,340 cfs (USGS 1998).

No known commercial fisheries exist within the unnamed creek, Deep Creek, or the Columbia River within the 15-mile TDL. Sport fishing occurs along Deep Creek and the Columbia River and subsistence fishing occurs on the Columbia River. Fish catch data is not available for Deep Creek. Sport catch data for the 13.5- to 15-mile section of the TDL on the Columbia River has been estimated at 49.5 pounds of kokanee salmon, 16.6 pounds of rainbow trout, 8.4 pounds of walleye, 4.75 pounds of smallmouth bass, and 7.53 pounds of yellow perch (DOE 1996). Sturgeon have also been caught in this area, but no data is available for this species. Additional sport catch data for the remainder of the 15-mile TDL in the Columbia River is not available.

The bull trout (*Salvelinus confluentus*), a Federal listed threatened species, is known to be present within the 15-mile TDL in the Columbia River (Vail 2000). There are two reported nesting areas for bald eagles (*Haliaeetus leucocephalus*), a Federal listed threatened species, along the Columbia River approximately 1 mile upstream of its confluence with Deep Creek (WDFW 2000). It is assumed that these eagles will utilize portions of the 15-mile TDL for feeding purposes although their nests lie outside the TDL for Last Chance Mine.

It is estimated from National Wetlands Inventory (NWI) Maps that 3.2 linear miles of wetlands exist along Deep Creek within the 15-mile TDL of the mine (USFWS 1994). The Coulee Dam National Recreation Area is within the site's 15-mile TDL (USGS 1992d).

1.2.3 Deep Creek Mine

Information presented in this section is based on a review of background information, interviews with the property owner and representatives from various regulatory agencies.

1.2.3.1 Mine Location and Description

This section presents the Deep Creek Mine location, description, and ownership history.

1.2.3.1.1 Deep Creek Mine Location

Mine Name:	Deep Creek Mine
CERCLIS ID No.:	WASFN1002161
Location:	Stevens County, Washington
Latitude:	48°51'48.9" N
Longitude:	117°42'54.4" W
Legal Description:	Sections 22, 23, and 26; Township 39N, Range 40E
Property Owner:	Richmond Family Trust 3041 Aladdin Road Northport, WA 99114
Property Contact:	Terry Richmond 3041 Aladdin Road Northport, WA 99114 (509) 732-1305

1.2.3.1.2 Mine Description/Ownership History

The Deep Creek Mine is an underground, inactive silver, lead, and zinc mine located west of Deep Creek, in Sections 22, 23, and 26, Township 39N, and Range 40E (Figure 1-12). The mine is located approximately 7 miles south of Northport, Washington, in Stevens County. From Northport the mine is accessed via the Colville-Aladdin Northport Road, which runs on the east side of the property (Figure 1-13).

The mine is currently owned by the Richmond Family Trust. Previous owners include the Northport Mining Company (1919-1921); Anaconda Copper Mining Company (1926-1941); Western Knapp Engineering Company (1941-1944); Jamison-Higginbotham Partnership (1944-1947); Goldfields Consolidated Mines (1947-1964); American Zinc Company (1964-1971); Columbia Resources, Inc. (1971-1979); and Great Basins Petroleum, Inc. from 1979 to the current owner, the Richmond Family Trust (Waddell 1962; McGinn 1979). No information was found regarding other periods of ownership.

1.2.3.2 Mine Operations and Source Characteristics

The mine was operated from approximately 1944 to 1956, when the mine was shut down (Waddell 1962). Details of mining operations prior to 1944 are not available. The maximum depth of

development was to 850 feet below ground surface. According to tabulations in 1958, the following quantities of metals were obtained from a total ore production of 763,307 tons (the following amounts are rounded to two significant figures): 66,000,000 pounds of zinc, 15,000,000 pounds of lead, 24,000 pounds of copper, 36,000 ounces of silver, and 69 ounces of gold (Fulkerson and Kingston 1958). The mine was closed in 1956 due to a combination of factors including a shaft fire, rising mining costs, and declining metal prices (Fulkerson and Kingston 1958). Following the closure, the headframe, hoisting, pumping, and other machinery were removed, and the mine was allowed to flood to within several feet of the shaft collar. Contaminants of concern include heavy metals in the water contained in the mine.

1.2.3.3 Mine Area Characterization

This section summarizes previous investigations (Section 1.2.3.3.1), and discusses migration/ exposure pathways and targets (Section 1.2.3.3.2).

1.2.3.3.1 Previous Investigations

The START conducted a visit to the mine on May 4, 2000. The START observed one shaft and one adit adjacent to the shaft; evidence of five mine buildings on the mine property (including an assay lab, a pump house, and other collapsed unidentified buildings); Mr. Richmond's residence; one fenced concrete pad with an electrical power tower (no transformers were observed on the tower); two ponds with standing water connected by a small stream; one settling pond (dry with fine, clay-like material on the bottom); 15-foot-deep waste rock piles covering approximately 0.33 acre and located adjacent to the entrance road, which is also covered in waste rock (total waste rock is approximately 8,000 cubic yards in volume); and a foundation that may have been the truck-loading area. The waste rock appeared to be quartzite and limestone. Six transformers were noted in the buildings, and there was no evidence of a mill on the property. Drainage from the shaft was clear, had no odor, and was estimated to flow at 20 gpm. The drainage infiltrated within approximately 75 feet of the shaft. The shaft is approximately 0.1 mile from Deep Creek (Figure 1-14).

Access to the mine is restricted by a gate with a lock; the gate was unlocked at the time of the visit. No signs of livestock grazing were observed, and no terrestrial sensitive environments were noted. Approximately 2,000 cubic yards of waste rock have been used for road construction.

1.2.3.3.2 Surface Water Migration Pathway and Targets

This section discusses the surface water migration pathway and potential targets within the mine's range of influence. The groundwater migration, soil exposure, and air migration pathways are not being evaluated in this ESI (Figures 1-15 and 1-16).

Surface water from the adit and shaft drains overland northeast to two ponds (connected by a small stream and spanning approximately 50 feet in length), then north to a dry settling pond with fine sediment (approximately 40 feet in width by 70 feet in length). From these areas, the surface water flows southeast to a swampy area before entering Deep Creek (PPE). From the field, surface water from the swampy area is expected to sheet flow approximately 300 feet into Deep Creek. The discharge rate is unknown. From the PPE, the 15-mile TDL extends along Deep Creek approximately 7 miles to its confluence with the Columbia River. The remaining 8 miles of the TDL is along the Columbia River.

The two-year, 24-hour rainfall event for the mine area is 1.6 inches and the average annual precipitation is 27.38 inches (WRCC 2000b). It is expected that the mine lies outside of a flood plain. As stated previously, the area consists of Waits-Rock outcrop complex. This soil is a loam that has a low infiltration rate, rapid runoff, and a high hazard of water erosion (USDA 1982). The upland drainage area of the mine is estimated from a topographic map to be 78 acres (USGS 1992c).

There are 10 surface water intakes within the 15-mile TDL of the mine. Nine of these are private domestic intakes and one is a municipal intake for the City of Northport (Ecology 2000b). The nine domestic intakes are estimated to serve 25 people. This number is based on the average number of persons per household for Stevens County, which is 2.73 (USBC 1990). The closest domestic intake is located 1.1 miles from the mine property. The municipal intake at Northport serves 460 people and is located 3.5 miles from the mine (EPA 2000c). Surface water is used within the TDL for irrigation of commercial food or forage crops and watering of commercial livestock (Ecology 2000b).

Flow rate data for Deep Creek are not available; however, based on visual observations during the START visit, the flow for Deep Creek was estimated to be approximately 84,000 gpm or 187 cfs. The average annual flow rate for the Columbia River, measured at Northport, Washington, is 103,340 cfs (USGS 1998).

No known commercial fisheries exist within Deep Creek or the Columbia River within the 15-mile TDL. Sport fishing occurs along Deep Creek and the Columbia River and subsistence fishing occurs on the Columbia River. Fish catch data is not available for Deep Creek. Sport catch data for the 13.5- to 15-mile section of the TDL on the Columbia River has been estimated at 49.5 pounds of Kokanee salmon, 16.6 pounds of rainbow trout, 8.4 pounds of walleye, 4.75 pounds of smallmouth bass,

and 7.53 pounds of yellow perch (DOE 1996). Sturgeon have also been caught in this area, but no data are available for this species. Additional sport catch data for the remainder of the 15-mile TDL in the Columbia River are not available.

The bull trout (*Salvelinus confluentus*), a Federal listed threatened species, is known to be present within the 15-mile TDL in the Columbia River (Vail 2000). There are two reported nesting areas for bald eagles (*Haliaeetus leucocephalus*), a Federal listed threatened species, along the Columbia River approximately 1 mile upstream from its confluence with Deep Creek (WDFW 2000). It is assumed that these eagles utilize portions of the 15-mile TDL for feeding purposes, although their nests lie outside of the TDL for Deep Creek Mine.

It is estimated from National Wetlands Inventory maps that 3.2 linear miles of wetlands exist along Deep Creek within the 15-mile TDL of the mine property (USFWS 1994). The Coulee Dam National Recreation Area is within the mine's 15-mile TDL (USGS 1992d).

1.2.4 Van Stone Mine

Information presented in this section is based on a review of background information, interviews with the property owner and representatives from various regulatory agencies.

1.2.4.1 Mine Location and Description

This section presents the Van Stone Mine location, description, and ownership history.

1.2.4.1.1 Van Stone Mine Location

Mine Name:	Van Stone Mine
CERCLIS ID No.:	WAD980834808
Location:	Stevens County, Washington
Latitude:	48°45'38.19" N
Longitude:	117°45'23.61" W
Legal Description:	Section 33, Township 38N, Range 40E
Property Owner:	Equinox Resources (Washington) Inc. c/o CT Corporation System 111 Eighth Avenue 13th Floor New York, NY 10011 (212) 894-8570

Property Contact: Mano River Resources Inc.
890 West Pender Street, Suite 600
Vancouver BC
V6C 1K4 Canada
(604) 689-1700

1.2.4.1.2 Mine Description/Ownership History

The Van Stone Mine is located approximately 25 miles north of Colville, Washington. The mine lies within the Onion Creek drainage basin (Figures 1-17 and 1-18). Mining activities are conducted in the areas highlighted on the figures, not just the area listed as the Van Stone Mine on the original topographic map. Van Stone Mine is an open pit lead and zinc mine with a beneficiation plant (used to process ore) on-site. The property has been owned by a number of companies over the years. The mine area was first discovered by George Van Stone and Henry Maylor in 1920. Following World War II, Asarco Incorporated produced 3.44 million tons of ore from an open-pit mine and discovered a new deep deposit. Following Asarco's sale of the property, both Callahan Mining Corporation and Equinox Resources Ltd. completed favorable feasibility studies of the mine and Mano's wholly-owned subsidiary, Equinox, reactivated the 1,000-ton-per-day open pit mine and concentrator in the early 1990s. Low zinc prices caused the closure of the operation in 1993 (Mano River Resources, Inc. 2001). Additional information regarding previous ownership history is not available.

The main asset of the property is a mineral deposit containing a drilled resource of 570 million pounds of zinc and 90 million pounds of lead with an overall potential of more than three times this amount. The deposit is located on over 1,400 acres. There is a fully developed open-pit mine with an operational 1,000-ton-per-day concentrator. The mine and concentrator were refurbished and in operation from 1991 through 1993. All production facilities and related infrastructure are presently on standby for a rapid reactivation to full capacity (Figures 1-18 and 1-19).

1.2.4.2 Mine Operations and Source Characteristics

Tailings created from the beneficiation plant were discharged in a slurry to the lower tailings pond. The upper tailings pond was only used as a standby pond in 1993. The old tailings were reworked and a plastic liner was placed on the tailings to receive the new tailings. The water from the tailings was pumped back to the beneficiation plant. This eliminated the discharges that historically drained to Onion Creek. The old dike on the upper tailings pond still showed signs of breaks that allowed releases to the creek in 1993 (SAIC 1993).

Equinox excavated drainage ditches up-slope of the tailings ponds to divert surface runoff around the ponds. Water that accumulated in the active pit was pumped to the inactive south pit where it infiltrated. Equinox was not permitted to discharge any water from the mine or tailings to surface water bodies.

Prior to Equinox's operation, approximately 3,000,000 tons of ore was processed to generate the tailings underlying the current tailings ponds.

The chemicals used to process the ore are limited. A portion of these may have been carried with the tailings to the tailings pond where the water was pumped back to the plant. The chemicals include flocculants, copper sulfate, quicklime, methyl amyl alcohol, zinc sulfate, sulfuric acid, sodium hydroxide, isopropanol, sodium sulfide, butyric acid, 2-ethylhexanol, and conditioners.

1.2.4.3 Mine Area Characterization

This section summarizes previous investigations (Section 1.2.4.3.1), and discusses migration/ exposure pathways and targets (Section 1.2.4.3.2).

1.2.4.3.1 Previous Investigations

Information regarding previous investigations was not obtained prior to the writing of the SQAP.

1.2.4.3.2 Surface Water Migration Pathway and Targets

This section discusses the surface water migration pathway and potential targets within the mine's range of influence. The groundwater migration, soil exposure, and air migration pathways are not being evaluated in this ESI (Figures 1-20 and 1-21).

Surface water from the tailings pile drains overland to Onion Creek (PPE) to the Columbia River. The lower tailings pond dimensions are approximately 1,200 by 1,200 by 10 feet. The upper tailings pond dimensions are approximately 400 by 1,200 by 10 feet. The average flow rate for Onion Creek is 110 cfs. The average flow rate for the Columbia River, measured at Northport, Washington, is 103,340 cfs. From the PPE, the 15-mile TDL extends along Onion Creek approximately 8 miles to its confluence with the Columbia River.

The mean annual precipitation at Colville for 1917 through 1951 is 16.96 inches (WRCC 2000d). The two-year, 24-hour rainfall event for the mine area is 2 inches (WRCC 2000b). It is expected that the mine lies outside of a flood plain. The area consists of coarse-textured soils with high infiltration rates. The upland drainage area of the upper tailings pond is estimated from a topographic map to be 44 acres.

The upland drainage for the remainder of the mine is estimated from a topographic map to be 130 acres.

It is unknown whether surface water drinking water rights exist on Onion Creek.

No known commercial fisheries exist within Onion Creek or the Columbia River within the 15-mile TDL. Sport and subsistence fishing occurs along the Columbia River.

It is estimated from National Wetlands Inventory maps that 14.7 linear miles of wetlands exist within the 15-mile TDL of the mine (USFWS 1994).

1.2.5 LeRoi Company Smelter

Information presented in this section is based on a review of background information, interviews with the property owner and representatives from various regulatory agencies.

1.2.5.1 *Smelter Location and Description*

This section presents the LeRoi Company Smelter location, description, and ownership history.

1.2.5.1.1 LeRoi Company Smelter Location

Smelter Name:	LeRoi Company Smelter
CERCLIS ID No.:	WAD988507323
Location:	Northport, Stevens County, Washington
Latitude:	48°55'15" N
Longitude:	117°46'15" W
Legal Description:	Section 4, Township 39N, Range 40E
Property Owner:	Mr. Steve Frazier SSF Building Materials 117 Park Road Northport, Washington 99157
Property Contact:	Mr. Steve Frazier 1964 E. Hawthorne Ave Colville, Washington 99114 (509) 684-4311

1.2.5.1.2 Smelter Description/Ownership History

The LeRoi Company Smelter (LeRoi), approximately 32 acres, is located just northeast of the town center of Northport, Washington, along Highway 25. The city of Northport is located along the east bank of the Columbia River approximately 7 miles south of the United States-Canadian border in Stevens

County. The Northport-Waneta Road borders the property along the south and east. Highway 25 defines the western boundary of the smelter. The Burlington Northern Railroad (formerly the Spokane Falls and Northern Railroad) runs parallel to the Columbia River and designates the northern property boundary. The Columbia River is located approximately 200 feet north of the LeRoi property. Residential homes are located west of the smelter. Smelter Hill is located directly east of the LeRoi property, elevation 2,026 feet above sea level (ASL). Silver Crown Mountain is south of the property, elevation 2,943 feet ASL. A city park, approximately 10 acres in area, is located northwest of the smelter property along the Columbia River, approximately 50 feet from the smelter (Figure 1-22; URS 1993).

The smelter encompasses a total area of 32 acres, including an old smelter and an inactive lumber operation. The smelter is accessed from an unpaved road east of Highway 25 located on the south end of the property. This road also provides access to the city park. The property is not paved. Grasses and poplar trees grow throughout the northern portion of the property. A hill, possibly the former smelter tailings pile, with an approximate slope of 10 degrees is located on the north end of the property, resulting in an elevation change across the property of 50 feet. Drainage appears to flow from northeast to southeast to the Upper Columbia River (Figures 1-23 and 1-24; URS 1993).

Stone foundations and old brick walls from the former smelter facility remain on the property. Most of these remains are located on the northern portion of the property. One of the three original smokestacks is still standing. This remaining stack was reported as the second largest stack, being only half the size of the largest stack. It stands approximately 75 feet high and has a maximum width of approximately 10 feet (URS 1993).

The vacant lumber operation is located on the southern third of the property. The majority of the structures used for the lumber operation were constructed over 20 years ago. Two lumber mill structures, an old wigwam burner, and an old log sawmill were identified on the property and are reported to be no longer in use and in a state of disrepair (URS 1993).

The former smelter buildings, which are no longer standing, included the furnace building (130 feet high, 100 feet wide, 700 feet long), the roaster building (90 feet high, 150 feet wide, 500 feet long), and the crusher and ore building (90 feet high, 100 feet wide, 600 feet long; URS 1993).

In the 1890s, a flurry of mining activities evolved in northeastern Washington and southern British Columbia. In 1892, D.C. Corbin, owner of the Spokane Falls and Northern Railroad, built a rail line to reach the city of Northport, then consisting of a lumber mill and several tents. The railroad tracks were located adjacent to the LeRoi smelter, which at that time was owned by Mr. Corbin. In 1896, Mr. Corbin donated the property to the LeRoi Mining and Smelting Company for the construction of the

Breen Copper Smelter. The smelter location was chosen because the area contained large quantities of materials necessary for smelting, such as limerock for flux (URS 1993).

The smelter began treating copper and gold ores transported from the Rossland Mine located in British Columbia, Canada. In 1901, the LeRoi Smelting Company operations reorganized as the Northport Smelting and Refining Company. By 1908, it was one of the largest smelters on the West Coast, processing 500 tons of ore per day. In 1909, the smelter closed because of competition from the Consolidated Mining and Smelting Operations smelter in Trail, British Columbia (URS 1993).

During World War I, the government demand for lead encouraged the Northport Mining and Smelting Company to reopen and process the lead ores that had been discovered at Leadpoint, Washington, approximately 9 miles east of Northport. In September 1914, Jerome Day purchased the smelter and renovated it to accommodate lead ores. On March 5, 1921, the government curtailed its lead purchases. A few months later, the smelter closed and never reopened. After the smelter closed in 1921, the American Smelting and Refining Company purchased the smelter. The company removed the smelting equipment and transported it to a smelter located elsewhere. The company left the dismantled smelter inactive (URS 1993).

Between 1921 and 1953, the inactive property was owned by J.D. Harms. Between 1953 and 1969, a lumber mill went into operation on the property. JB&T Lumber is the first known lumber mill company to have operated on the property. In 1975, Cecil Frazier purchased the property and operated the lumber mill. In 1985, Steve Frazier, son of Cecil Frazier, purchased the property and business and operated a mill under the name SSF Building Materials which is currently vacant (URS 1993; Frazier 2001).

1.2.5.2 Operations and Source Characteristics

The Breen Copper Smelter operated from 1896 until 1901. The initial smelter operations were rudimentary and involved releases of large quantities of pollutants. The tellurium ore was more difficult to process; however, it contained high enough amounts of copper and gold to make the process worthwhile. Tellurium is naturally occurring and belongs to the same family of elements as sulfur and selenium. Because of the tellurium, the ore had to be burned or heated to release the minerals. The burning released high amounts of sulfur dioxide into the air (URS 1993).

The ore was processed by heap roasting, which involves open burning of the raw ore prior to placing it in a mineral filtration furnace. The heap roasting process produced a disagreeable sulfur odor;

the local citizens termed the burning piles "stink piles." Local farmers believed that the heap roasting process was poisoning the nearby soils (URS 1993).

A slag brick platform was used for the initial burning, or heap roasting, of the ore. The ore was piled on the brick platform to an approximate depth of 4 feet. Cord wood was then stacked on top of the ore pile and ignited. The tellurium in the ore would be vaporized during this process, thus freeing the gold and copper for smelting. The location of this brick platform is where the Northport city wells are currently located (URS 1993).

Because gold is heavy, it settled to the bottom of the furnace and formed a gold matte. After the gold accumulated to a thickness of 14 inches, the furnace was shut down. Once the furnace and materials cooled, the sides of the furnace were removed to gain access to the gold matte, which was then pried from the furnace and cut into pieces before being loaded into boxcars and shipped to a gold refinery (URS 1993).

The lead smelter used a process more sophisticated than that used in the copper and gold process of the previous decade, although a large quantity of sulfur (approximately 30 tons per day) was still being discharged into the air. This emission was reportedly considered tolerable by the residents. Filters for the smokestacks were added later (URS 1993).

In the days of the copper and gold smelter, two large steam engines, fueled by coal, provided power. Both flywheel steam engines were hooked onto one long line shaft. On the other end of the line shaft, a dynamo produced 10,000 volts of electricity prior to being boosted by a generator that provided up to 100,000 volts. Once the smelter reopened to process lead ores, a high-voltage line from Canada supplied the power, and the steam plant was shut down (URS 1993).

After 1921, the abandoned and dismantled smelter remained inactive. The town of Northport demolished the buildings for the usable brick. One building retained enough walls to provide an ice-skating rink during the winter. The railroad was abandoned and the tracks were salvaged. By 1929, only half of the upper Stevens County population remained and the entire upper Stevens County was suffering great economic hardship (URS 1993).

Smelting operations produced a tailings waste referred to as slag. The slag was usually placed in piles near the smelter for temporary or permanent disposal. Historical photographs indicate possible tailings piles located on the northeast portion of the LeRoi property. The slag piles could be covered by topsoil and vegetation. The exact location or process of disposal of the slag piles is unknown.

Operations on the property in 1993 involved a lumber mill. It was one of the largest businesses in Northport, employing from 18 to 25 people. The entire property was used for the mill. The southern

half of the property held the main lumber operations. The northern half of the property, which contains the smelter remnants, was used to store lumber products and old metal parts including cars, piping, and roofing.

The lumber mill processed mostly cedar wood from rough-dimension lumber into exterior siding and exterior paneling. The mill process included cutting the wood, drying the cut wood, and shipping it. Mill operations were run on propane. All water used for mill operations was obtained from the city water supply. The mill did not discharge to or collect water from the Columbia River (URS 1993).

The scrap wood materials, including sawdust, were sent to Kettle Falls for the Kettle Falls Water Power Company, which burned the material for energy. Originally, the lumber mill burned the scrap wood on the property inside a wigwam burner. Although the on-site burning has been eliminated for years, the wigwam burner was observed on the property. No wood treatment or chemical use is reported in the past mill operations (URS 1993).

1.2.5.3 Smelter Characterization

This section summarizes previous investigations (Section 1.2.5.3.1), discusses migration/ exposure pathways and targets (Section 1.2.5.3.2), and describes areas of potential contamination (Section 1.2.5.3.3).

1.2.5.3.1 Previous Investigations

In July 1992, the Washington State Department of Health outlined potential activities to be undertaken in response to community health concerns in the Northport area including epidemiologic reviews, rates of hospitilizations for ulcerative colitis and Chron's Disease, reviewing list of suspected pollutants for links with reported disease, and proposing Northport for air, soil, and biological sampling.

A preliminary assessment and a site inspection were performed by URS Consultants, Inc., on behalf of the EPA in 1993. The analytical results for antimony in the surface soil samples from the LeRoi property indicated concentrations exceeding Washington State Model Toxics Act Method B soil cleanup levels. The analytical results for arsenic and lead in the surface soil samples from the LeRoi property indicate concentrations exceeding Washington State Model Toxics Act Method A soil cleanup levels (URS 1993).

The Washington Department of Ecology (Ecology) sampled soils and slag piles at the LeRoi Smelter in 1997. A report for this sampling event is unavailable; however, preliminary results showed levels of two to three orders of magnitude above background. Concentrations of up to 1,010 milligrams

per kilogram (mg/kg) of arsenic, 337 mg/kg of cadmium, 33,400 mg/kg of copper, 20,200 mg/kg of lead, and 1,750 mg/kg of zinc were found (Gregory 2000b). Ecology had planned to conduct a groundwater investigation at the LeRoi Smelter property. However, due to availability of resources, this has not been pursued (Gregory 2000a).

According to the Stevens County Assessor's Office, the property was sold to KES Contracting, Inc., 1252 Bay Avenue, Trail, British Columbia, in June 1999.

1.2.5.3.2 Surface Water Migration Pathway and Targets

This section discusses the surface water migration pathway and potential targets within the mine's range of influence. The groundwater migration, soil exposure, and air migration pathways are not being evaluated in this ESI (Figures 1-25 and 1-26).

The LeRoi smelter is located along the upper Columbia River in northeastern Washington. This area is fairly dry, with an average annual precipitation of 20.29 inches and a 2-year 24-hour precipitation of 1.2 inches. Precipitation accumulates in the form of snow during the winter months (URS 1993).

Most surface water runoff would travel directly north from the southern half of the property and northwest from the northern half of the property to the Columbia River approximately 90 to 200 feet from the northern boundary, with an elevation decrease of approximately 50 feet. The property is not located within the 100-year floodplain (URS 1993).

The Columbia River is the only surface water body identified within 15 miles downstream of the LeRoi property. The river flow and elevation are controlled by several dams located both upstream in Canada and downstream in the United States. The nearest dam is located approximately 15 miles upstream. The first downstream dam, Coulee Dam, is located approximately 130 miles downstream. The 130-mile stretch along the Columbia River from Coulee Dam to Northport is considered Lake Roosevelt.

Twelve surface water intakes are located 15 miles downstream of the LeRoi property along the Columbia River. One surface water intake (Permit #4638), approximately 15 miles downstream, is used for both domestic and irrigation purposes. The remaining surface water intakes are used for irrigation with the exception of one intake used for mining operations. No other surface water intakes were identified (URS 1993).

Fisheries identified along the Columbia River within the 15-mile TDL consist of only nonanadromous species. No anadromous fish are found above the Chief Joseph Dam in Bridgeport, approximately 100 miles downstream from Northport.

1.2.6 Areas of Potential Contamination

Sampling at the mines and mills to be visited under the Upper Columbia ESI will be conducted at those areas considered to be potential contamination sources and PPEs. Table 1-1 lists the mines and mills to be visited and potentially sampled (Figure 1-27). Under this ESI, potential contamination sources include but are not limited to:

Potential Sources:

- **Tailings Piles/Ponds.** Background information indicates that tailings piles or ponds are present on many of the mines and mills. Probable potential contaminants of concern are Target Analyte List (TAL) metals including mercury. Tailings piles/ponds with a total volume less than 1,000 cubic yards will not be sampled.
- **Waste Rock Piles.** Background information indicates that waste rock piles may be present at adit and shaft elevations. The waste rock at these elevations has not been processed. Probable potential contaminants of concern are TAL metals. Waste rock piles with a total volume less than 1,000 cubic yards will not be sampled.
- **Mill Soil Areas.** Evidence of mills has been documented at many of the mines and mills. Expected mill processes include concentration, flotation, and/or mercury amalgamation. Probable potential contaminants of concern include TAL metals including mercury, and PCBs. Mill soil samples will also be analyzed for chlorinated pesticides (pesticides) as these results are provided as part of the analytical method; the Weston field team is also collecting samples for pesticide/PCB analysis.
- **Mine Adits.** Adit discharge is a potential source of contamination. Probable potential contaminants of concern include TAL metals including mercury.

Proposed sample locations for the Pend Oreille Mine are identified in Figure 1-28.

Potential Targets:

- **Pend Oreille River.** Past discharges from the Pend Oreille mine and mill and ongoing discharges from the adit and the tailings piles may be impacting the surface water and sediments in the Pend Oreille River. Probable potential contaminants of concern are TAL metals including mercury and pesticides/PCBs.
- **Tributaries on the Pend Oreille River.** Past discharges from mines and mills and ongoing discharges from adits and tailings piles may be impacting the surface water and sediments in tributaries flowing into the Pend Oreille River. Probable potential contaminants of concern are TAL metals including mercury and pesticides/PCBs.
- **Groundwater Within 4-Mile Radius.** Groundwater at the Pend Oreille Mine will be sampled and analyzed for TAL metals including mercury.

A groundwater sample and up to two soil samples will be collected at a park adjacent to the LeRoi Company Smelter if access is granted to the property. The samples will be analyzed for TAL metals including mercury. The Cleveland Mine and Mill, assigned to E & E under TDD 01-01-0035, will

be visited during the Upper Columbia River ESI field event. Up to 6 surface soil and/or water samples, including background sample(s), will be collected following the guidelines presented in this SQAP. The procedures outlined in Sections 2 and 3 of this SQAP will be followed for the mines and mills.

1.3 PROJECT/TASK DESCRIPTION AND SCHEDULE

This section provides the project description (Section 1.3.1) and schedule (Section 1.3.2).

1.3.1 Project Description

This section defines the objectives and scope for performing the Upper Columbia River ESI activities at the mines and mills. The main goals for the ESI activities are as follows:

- Collect and analyze samples to help characterize the potential sources discussed in Section 1.2.6;
- Determine off-site migration of contaminants; and
- Document a threat or potential threat to public health or the environment posed by the mines or mills.

1.3.2 Schedule

The schedule for implementing the Upper Columbia River ESI mine and mill sampling is intended to be used as a guide. Adjustments to the implementation dates and the estimated project duration may be necessary to account for variable unforeseen or unavoidable conditions that the field team may encounter. Examples include inclement weather, difficulties in accessing a sampling location, or additional time needed to complete a task. Any significant schedule changes that arise in the field will be discussed with the TM at the earliest possible convenience. The proposed schedule of project work is as follows:

Activity	Start	Complete
Mobilize	6/17/01	6/17/01
Sample Collection Activities	6/18/01	6/29/01
Laboratory Receipt of Samples	6/22/01	7/03/01
Demobilize	7/30/01	7/30/01
Receipt of Data from EPA or CLP Laboratory (3 weeks from receipt)	7/13/01	7/24/01
Data Validation for all Laboratory Data (3 weeks from receipt)	8/03/01	8/14/01
Write Project Report (6 weeks)	7/27/01	9/14/01
Target Project Completion Date	Not Applicable	10/01/01

1.4 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The project data quality objectives (DQOs) are to provide valid data of known and documented quality to characterize sources, determine off-site migration of contaminants, and document any threat(s) or potential threat(s) to public health or the environment posed by the mines and mills. The DQO process to be applied to this project will follow that described in the EPA (1994b) document, *Guidance for the Data Quality Objectives Process.*

1.4.1 DQO Data Categories

All samples collected under this SQAP will be analyzed using definitive analytical methods. All definitive analytical methods employed for this project will be methods approved by the EPA. The data generated under this project will comply with the requirements for this data category as defined in *Data Quality Objectives Process for Superfund* (EPA 1993).

1.4.2 Data Quality Indicators (DQIs)

DQI precision, accuracy, representativeness, comparability, and completeness goals for this project were developed following guidelines presented in the EPA *Guidance for Quality Assurance Project Plans*, EPA QA/G-5 Final, Appendix D.

The basis for assessing each of the elements of data quality is discussed in the following subsections. Section 2.5 presents the QA objectives for measurement of analytical data and QC guidelines for precision and accuracy. Other DQI goals are included in the individual Standard Operating Procedures (SOPs) in Appendix A and in the Laboratory Statement of Work (SOW).

1.4.2.1 Representativeness

Representativeness is a measure of the degree to which data accurately and precisely represent a population, including a sampling point, a process condition or an environmental condition. Representativeness is the qualitative term that should be evaluated to determine that measurements are made and physical samples collected at locations and in a manner resulting in characterizing a matrix or media. Subsequently, representativeness is used to ensure that a sampled population represents the target population and an aliquot represents a sampling unit. This SQAP will be implemented to establish representativeness for this project. Further, all sampling procedures detailed in the SQAP will be followed to ensure that the data will be representative of the media sampled. The SQAP describes the sample location, sample collection and handling techniques to avoid contamination or compromise

sample integrity, and proper chain-of-custody. Additionally, the sampling design presented in the SQAP will ensure that there are a sufficient number of samples and level of confidence that analysis of these samples will detect the chemicals of concern, if present.

1.4.2.2 Comparability

Comparability is the qualitative term that expresses the measure of confidence that two data sets or batches can contribute to a common analysis and evaluation. Comparability with respect to laboratory analyses pertains to method type comparison, holding times, stability issues, and aspects of overall analytical quantitation. The following items are evaluated when assessing data comparability:

- Determining if two data sets or batches contain the same set of parameters.
- Determining if the units used for each data set are convertible to a common metric.
- Determining if similar analytical procedures and quality assurance were used to collect data for both data sets.
- Determining if the analytical instruments used for both data sets have approximately similar detection levels.
- Determining if samples within data sets were selected and collected in a similar manner.

To ensure comparability of data collected during this investigation to other data that may have been or may be collected for each property, standard collection and measurement techniques will be used.

1.4.2.3 Completeness

Completeness is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not rejected through data validation. The requirement for completeness is 95% for aqueous samples and 90% for soil/sediment samples.

The following formula is used to calculate completeness:

$$\% \text{ completeness} = \frac{\text{number of valid results}}{\text{number of possible results}}$$

For any instances of samples that could not be analyzed for any reason (holding time violations in which resampling and analysis were not possible, samples spilled or broken, etc.), the numerator of this calculation becomes the number of valid results minus the number of possible results not reported.

For this investigation, all samples are considered critical. Therefore standard collection and measurement methods will be used to achieve the completeness goal.

1.4.2.4 Precision

Precision measures the reproducibility of 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 the measurement of the variability associated with duplicate (two) or replicate (more than two) analyses. The laboratory control sample (LCS) determines the precision of the analytical method. If the recoveries of the analytes in the LCS are within established control limits, then precision is within limits. In this case, the comparison is not between a sample and a duplicate sample analyzed in the same batch. Rather, the comparison is between the sample and samples analyzed in previous batches.

Total precision is the measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations. Field duplicate samples and matrix duplicate spiked samples shall be analyzed to assess field and analytical precision, and the precision measurement is determined using the relative percent difference (RPD) between the duplicate sample results.

The following formula is used to calculate precision:

$$RPD = (100) \times \frac{(S1 - S2)}{(S1 + S2)/2}$$

where:

S1 = normal sample value

S2 = duplicate sample value

1.4.2.5 Accuracy

Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systemic error. It 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 and standard. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS to a control limit. For volatile and semivolatile organic compounds (not applicable for this ESI), surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed. Analysis of performance evaluation (PE) samples shall also be used to provide additional information for assessing the accuracy of the analytical data being produced.

1.5 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

Special training requirements or certifications are required for this project including the 40-hour Hazardous Waste Operations and Emergency Response class and annual refreshers. Health and safety procedures for E & E personnel are addressed in the E & E site-specific HSP. As previously discussed (Section 1.1.1), this document is maintained in E & E's Seattle, Washington, office and will also be provided to each field team. Included in the plan are descriptions of anticipated chemical and physical hazards, required levels of protection, health and safety monitoring requirements and action levels, personal decontamination procedures, and emergency procedures.

1.6 DOCUMENTATION AND RECORDS

This document is meant to be combined with information presented in E & E's QAPP (E & E 2001a) for the START-2, Region 10. This information is covered in this SQAP by the SOPs found in Appendix A and the CLP laboratory SOW. A copy of the START-2 QAPP is available in E & E's Seattle, Washington, office. Standards contained in the SOPs, the START-2 QAPP, and the QMP will be used to ensure the validity of data generated by E & E for this project.

Following E & E's receipt of all analytical data including sediment and surface water sample results generated by Weston and data from the Phase 1 and Phase 2 mines/mills field events, an ESI report summarizing project findings will be prepared by the E & E START-2 PM. Project files, including work plans, reports, analytical data packages, correspondence, chain-of-custody (COC) documentation, logbooks, corrective action forms, referenced materials, and photographs will be

provided to the EPA TM at the close of the project. Furthermore, a compact disk-read only memory (CD-ROM) containing the project photographs and reports will be provided.

E & E will assemble and fully document a digital data set including all project sampling, analysis and observation data. This digital data will be made available in a Microsoft-Access format. E & E will transfer this data set and documentation to EPA, or if requested, to any other EPA contractor, and shall insure that any data transferred is received in an uncorrupted, comprehensible and usable format. Specific data deliverable elements are presented below.

Data

A summary description of the tables, the sources of information, and other comments are provided below.

Field-Info

The field information table contains all sample collection related information. A Microsoft-Access application (Sample Information System, SIS) will be used to input and store the data. The SIS provides the user with "smart" data input forms that will only allow for the entry of acceptable data field values. For each sampling event, the SIS will be updated to reflect the new samples collected. Once entered, the information will be checked and corrected where necessary. The table structure is presented below.

Field Name	Type	Size	Description
Sample-num	Character	10	Sample Number
Station	Character	10	Station Identifier
Date	Date	8	Sample Date
Time	Numeric	4	Sample Time (24 hour clock)
Sampler	Character	25	Person name
Matrix	Character	6	Sample Matrix – (i.e. soil boring, groundwater, sediment)
Water Depth	Numeric	5.1	Depth of water at sediment sample
Description	Character	40	Sample Description
Comments	Character	40	Comments

Location

The location table contains sample location coordinate information. The sample locations will be determined using three Trimble Pro-XR Global Positioning System (GPS) units. E & E personnel have been trained and have utilized these units in similar projects. For each day or half-day in the field that GPS sample location data is to be collected, the GPS user will create a single file that contains the locations of each sample station. A unique station label will be entered for each sample location. This unique station identifier will be used to link the "Location" table with the "Field-Info" table. This information will be downloaded from the GPS unit and imported into the "Location" table of the Site

Data Management System (SDMS). All locational data for this project will be stored in decimal degrees, and will be referenced to the NAD 27 horizontal datum. Differential corrections will be made real-time. The table structure is presented below.

Field Name	Type	Size	Description
Station	Character	10	Station Identifier
X-Coord	Numeric	12.6	X-Coordinate, Decimal Degrees
Y-Coord	Numeric	12.6	Y-Coordinate, Decimal Degrees

Lab Analytical

The Lab Analytical table will hold all of the sample analysis results provided by each laboratory analyzing samples. The integrity of each data file received from the labs will be checked and verified. Once the files are received, they will be appended into the SDMS Lab Analytical table. The "Sample-num" field will be used to link the "Lab Analytical" table with the "Field-Info" table. The table structure is presented below.

Field Name	Type	Size	Description
Sample-num	Character	10	Sample Number
Lab-id	Character	10	Laboratory Sample Identifier
Method	Character	25	Analytical Method used
L-Matrix	Character	10	Laboratory Matrix
Cas-num	Character	15	Chemical Abstracts Service (CAS) number
Analyte	Character	40	Analyte Name
Result	Numeric	12.6	Analysis result
Qual	Character	6	Sample qualifier
Quantitation-Limit	Numeric	12.6	Sample quantitation limit
Units	Character	10	Result units
Date	Date	8	Date Analyzed
Lab	Character	40	Lab name

For any Geographic Information Systems (GIS) produced maps, E & E shall provide the maps to EPA in hard copy and digital image (i.e. JPEG) formats.

Table 1-1

MINES AND MILLS TO VISIT AND POTENTIALLY SAMPLE
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION
NORTHEAST WASHINGTON

County	Mine/Mill Name	LAT			LONG			S	T	R	River System(s)
Stevens	Morning	48	44	24.36	117	38	46.68	4	37N	41E	South Fork Deep Creek, Deep Creek
Stevens	Burrus	48	43	26.76	117	39	43.92	8	37N	41E	South Fork Deep Creek, Deep Creek
Stevens	Copper King	48	46	51.60	117	39	12.24	20	38N	41E	South Fork Deep Creek, Deep Creek
Stevens	Sierra Zinc (Blue Ridge)	48	46	28.20	117	40	06.24	20	38N	41E	South Fork Deep Creek, Deep Creek
Stevens	Magma	48	46	00.48	117	38	25.08	28	38N	41E	South Fork Deep Creek, Deep Creek
Stevens	Calhoun	48	55	09.84	117	35	28.68	2	39N	41E	North Fork Deep Creek
Stevens	Bechtol	48	51	57.60	117	35	39.48	23	39N	41E	Deep Lake, Deep Creek
Stevens	Thompson	48	52	09.84	117	35	03.84	23	39N	41E	Deep Lake
Stevens	Tyee	48	51	27.36	117	37	12.00	27	39N	41E	Deep Lake
Stevens	Farmer	48	50	5928	117	37	17.40	34	39N	41E	Deep Lake
Stevens	Maki	48	50	45.60	117	36	00.00	35	39N	41E	Deep Lake
Stevens	Lead trust	48	53	49.92	117	33	31.68	7	39N	42E	Unnamed Creek, Republican Creek, North Fork Deep Creek
Stevens	Electric Point	48	52	56.28	117	32	29.04	17	39N	42E	Unnamed Creek, Republican Creek, North Fork Deep Creek
Stevens	Keystone	48	52	53.04	117	31	43.68	17	39N	42E	Unnamed Creek, Republican Creek, North Fork Deep Creek
Stevens	Lucky Four	48	52	51.6	117	32	33.00	17	39N	42E	Unnamed Creek, Republican Creek, North Fork Deep Creek
Stevens	Gladstone	48	53	12.48	117	32	35.16	18	39N	42E	Unnamed Creek, Republican Creek, North Fork Deep Creek
Stevens	Admiral Consolidated	48	55	52.2	117	34	28.6	36	40N	41E	Hartbauer Creek, North Fork Deep Creek
Stevens	Anaconda	48	56	53.5	117	33	24.5	25	40N	42E	Unnamed Creek, Cedar Lake, North Fork Deep Creek
Stevens	Red Top	48	56	35.88	117	33	52.20	25	40N	42E	Hartbauer Creek, North Fork Deep Creek
Stevens	Evergreen	48	56	57.48	117	33	55.80	30	40N	42E	Hartbauer Creek, North Fork Deep Creek
Stevens	Lucile	48	57	01.08	117	3	12.24	30	40N	42E	Hartbauer Creek, North Fork Deep Creek
Stevens	A & C	48	44	30	117	52	27	2	37N	39E	Unnamed Creek, East Fork Bruce Creek, Bruce Creek, Dry Lake
Stevens	RJ	48	43	54.84	117	52	31.80	3	37N	39E	East Fork Bruce Creek, North Fork Bruce Creek, Dry Lake
Stevens	Uncle Sam	48	43	57.72	117	52	33.96	3	37N	39E	East Fork Bruce Creek, North Fork Bruce Creek, Dry Lake
Stevens	Silver Trail	48	45	09.36	117	52	56.28	33	38N	39E	North Fork Bruce Creek, Bruce Creek, Dry Lake
Stevens	New Leadville	48	44	08.52	117	52	33.60	3	39N	39E	East Fork Bruce Creek, North Fork Bruce Creek, Dry Lake
Pend Oreille	Sullivan	48	52	36.84	117	22	55.56	16	39N	43E	Wetlands, Flume Creek
Stevens	Clara	48	59	19.32	117	36	16.2	10	40N	41E	Unnamed Creek, Cedar Creek, Canada
Stevens	Lakeview	48	57	51.84	117	32	57.12	19	40N	41E	Joe Creek, Joe Lake, East Fork Cedar Creek, Cedar Creek, Canada
Stevens	Jackson	48	57	35.64	117	34	08.04	24	40N	41E	Joe Creek, Joe Lake, East Fork Cedar Creek, Cedar Creek, Canada
Stevens	Roosevelt	48	59	22.20	117	32	58.56	7	40N	42E	East Fork Cedar Creek, Cedar Creek, Canada

Table 1-1

MINES AND MILLS TO VISIT AND POTENTIALLY SAMPLE
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION
NORTHEAST WASHINGTON

County	Mine/Mill Name	LAT			LONG			S	T	R	River System(s)
Stevens	Myeerah	48	59	26.16	117	27	14.76	11	40N	42E	Unnamed Creek, Jubilee Creek, Canada
Stevens	United treasure	48	59	21.48	117	27	51.12	11	40N	42E	Unnamed Creek, Jubilee Creek, Canada
Stevens	Frisco Standard	48	59	34.80	117	26	39.48	12	40N	42E	Unnamed Creek, Jubilee Creek, Canada
Stevens	Scaman	48	57	30.24	117	32	24.00	19	40N	42E	Joe Creek, Joe Lake, East Fork Cedar Creek, Cedar Creek, Canada
Pend Oreille	Red Top	48	59	52.08	117	11	02.76	1	40N	44E	Overland, Lead Creek, Canada
Pend Oreille	King Tut	48	59	21.48	117	11	28.32	11	40N	44E	Overland, Cedar Creek, Canada
Pend Oreille	Blue Bucket	48	43	26.76	117	39	43.92	8	37N	41E	Overland (1,700 ft) (@ RM 30)
Pend Oreille	Metaline	48	50	32.64	117	23	24.00	32	37N	41E	Overland (@ RM 30)
Pend Oreille	Sterline	48	50	10.32	117	23	29.04	32	37N	41E	Unnamed Creek (@ RM30)
Pend Oreille	Pend Oreille	48	52	54.12	117	21	36.00	15	39N	43E	Overland (@ Deadmans Eddy)
Pend Oreille	Josephine	48	52	48.00	117	22	15.96	16	39N	43E	Overland (@ Deadmans Eddy)
Pend Oreille	Yellowhead	48	52	59.88	117	22	14.16	16	39N	43E	Overland (@ Deadmans Eddy)
Pend Oreille	Oriole	48	51	36.72	117	24	46.44	19	39N	43E	Linton Creek (@ RM 29.25)
Pend Oreille	West Contact Mine	48	57	07.92	117	24	56.88	19	39N	43E	Linton Creek (@ RM 29.25)
Pend Oreille	Washington	48	52	05.52	117	22	27.12	21	39N	43E	Overland (@ RM 27)
Pend Oreille	Bella May	48	50	53.16	117	24	16.20	29	39N	43E	Unnamed Creek, overland through Metaline (@ RM 30)
Pend Oreille	Lehigh No. 1	48	51	02.52	117	24	14.04	29	39N	43E	Unnamed Creek, through Metaline (@ RM 29)
Pend Oreille	Diamond R.	48	51	12.60	117	25	19.20	30	39N	43E	Unnamed Creek, Linton Creek (@ RM 29.25)
Pend Oreille	Lehigh No. 2	48	51	31.68	117	24	52.20	30	39N	43E	Linton Creek (@ RM 29.25)
Pend Oreille	Hanley	48	59	06.72	117	21	00.72	10	40N	43E	Overland
Pend Oreille	Lead Queen	48	58	46.56	117	19	37.20	11	40N	43E	Overland (3,546 ft),
Pend Oreille	Z Canyon	48	58	41.52	117	20	34.08	11	40N	43E	Overland
Pend Oreille	Cliff	48	57	27.72	117	21	16.56	22	40N	43E	Upper Lead King Lake, Lower Lead King Lake, Everett Creek
Pend Oreille	Hoage	48	56	57.48	117	21	18.00	22	40N	43E	Upper Lead King Lake, Lower Lead King Lake, Everett Creek
Pend Oreille	Lead King	48	56	16.44	117	21	13.32	27	40N	43E	Lower Lead King Lake, Everett Creek
Pend Oreille	Lucky Strike	48	55	49.80	117	19	51.24	35	40N	43E	Overland
Pend Oreille	Lead Hill	48	58	12.72	117	11	49.56	14	40N	44E	Slate Creek
Stevens	Gold Bar	48	41	39.84	118	00	30.24	22	37N	38E	Unnamed Creeek (@ RM 711)
Stevens	Van Stone	48	45	38.16	117	45	23.76	33	38N	40E	Onion Creek (@ RM 730)
Stevens	Hope & Twin Cabins	48	53	09.96	118	01	37.92	7	39N	38E	Unnamed Creek, Squaw Creek (@ RM 721.5)
Stevens	Homestake	48	52	06.96	118	01	18.84	19	39N	38E	Unnamed Creek, Hope Creek, Fifteen Mile Creek (@ RM 721.5)

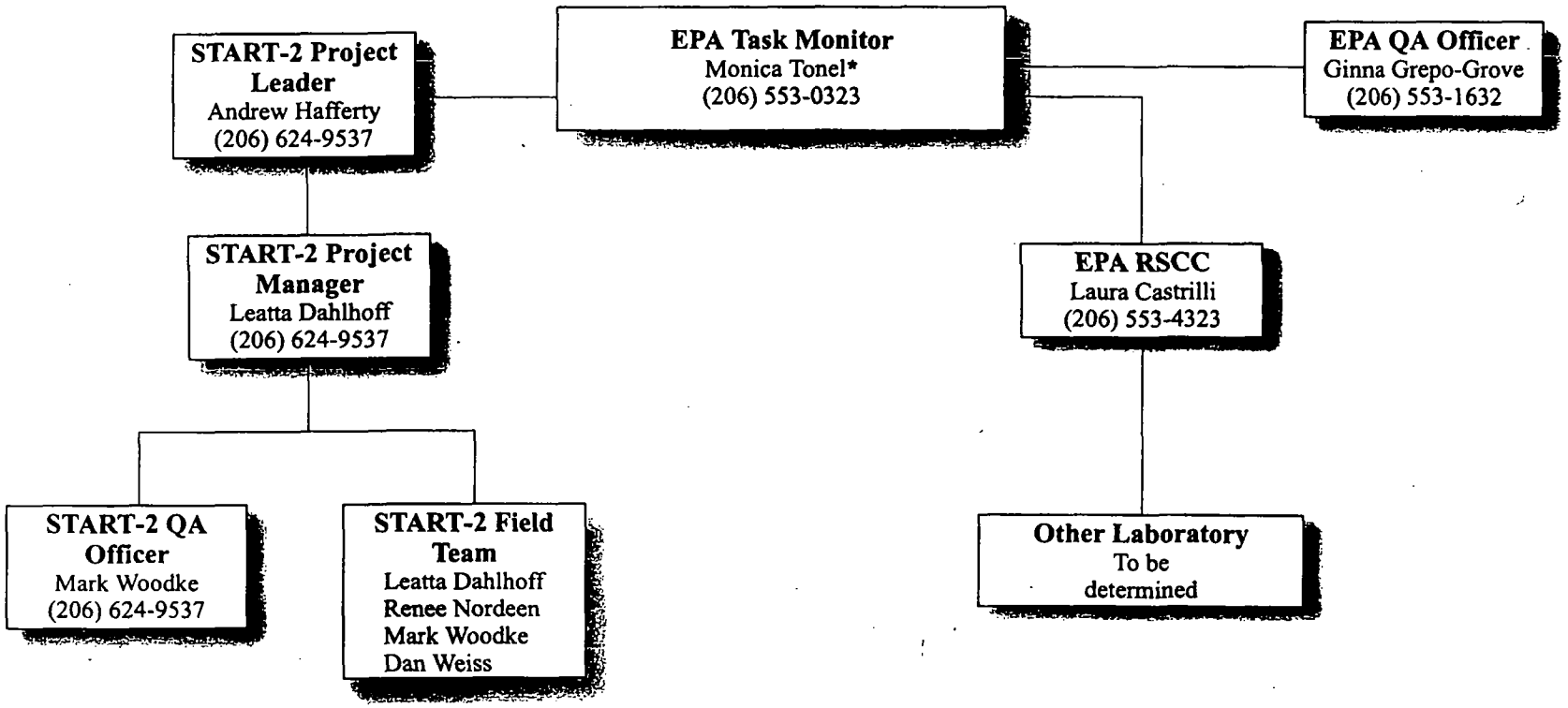
Table 1-1

MINES AND MILLS TO VISIT AND POTENTIALLY SAMPLE
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION
NORTHEAST WASHINGTON

County	Mine/Mill Name	LAT			LONG			S	T	R	River System(s)
Stevens	Lottie	48	51	53.28	118	01	15.24	19	39N	38E	Unnamed Creek, Fifteen Mile Creek (@ RM 721.5)
Stevens	Minorca	48	51	44.28	118	01	44.40	19	39N	38E	Unnamed Creek, Fifteen Mile Creek (@ RM 721.5)
Stevens	Antelope	48	52	04.80	118	01	05.88	19-20	39N	38E	Unnamed Creek, Hope Creek, Fifteen Mile Creek (@ RM 721.5)
Stevens	Mountain View	48	54	49.32	117	49	49.44	3	39N	39E	Unnamed (intermitent) Creek (@ RM 735)
Stevens	Black Rock	48	53	16.44	117	54	25.56	7	39N	39E	Deep Creek (@ RM 737)
Stevens	Bullion	48	53	16.44	117	54	25.56	7	39N	39E	Unnamed Creek, Rattlesnake Creek (@ RM 727.5)
Stevens	Silver Crown	48	54	33.84	117	46	40.80	5	39N	40E	Overland, through Northport (@ RM 734)
Stevens	Scandia	48	52	32.88	117	43	08.04	23	39N	40E	Deep Creek (@ RM 737)
Stevens	Great Western	48	52	06.24	117	41	48.48	24	39N	40E	Deep Creek (@ RM 737)
Stevens	Last Chance	48	51	59.40	117	41	56.40	24	39N	40E	Deep Creek (@ RM 737)
Stevens	Deep Creek	48	51	48.96	117	42	54.36	26	39N	40E	Deep Creek (@ RM 737)
Stevens	Lone Star	48	50	27.60	117	42	59.76	35	39N	40E	Deep Creek (@ RM 737)
Stevens	Hubbard	48	55	15.96	117	52	03.72	32	40N	39E	Unnamed Creek, Flat creek (@ RM 721.5)
Stevens	Coyote	48	55	44.76	117	48	54.72	35	40N	39E	Unnamed (intermittent) Creek (@ RM 735)
Stevens	Northport Smelter (LeRoi Company Smelter)	48	55	23.16	117	46	02.28	33	40N	40E	Overland (@ RM 735)
Stevens	Sterrett	48	55	33.24	117	45	45.36	33	40N	40E	Overland (@ RM 735)
Stevens	Melrose	48	56	44.52	117	38	45.96	28	40N	41E	Tom Bush Creek, overland (1,100 ft) (@ RM 742.5)
Stevens	Iroquois	48	57	06.12	117	32	22.92	30	40N	42E	Unnamed Creek, Squaw Creek (@ RM 721.5)

Key:

E = East.
Lat = Latitude.
Long = Longitude.
N = North.
R = Range.
RM = Road Mile.
S = Section.
T = Township.



KEY:

* Approving Authority

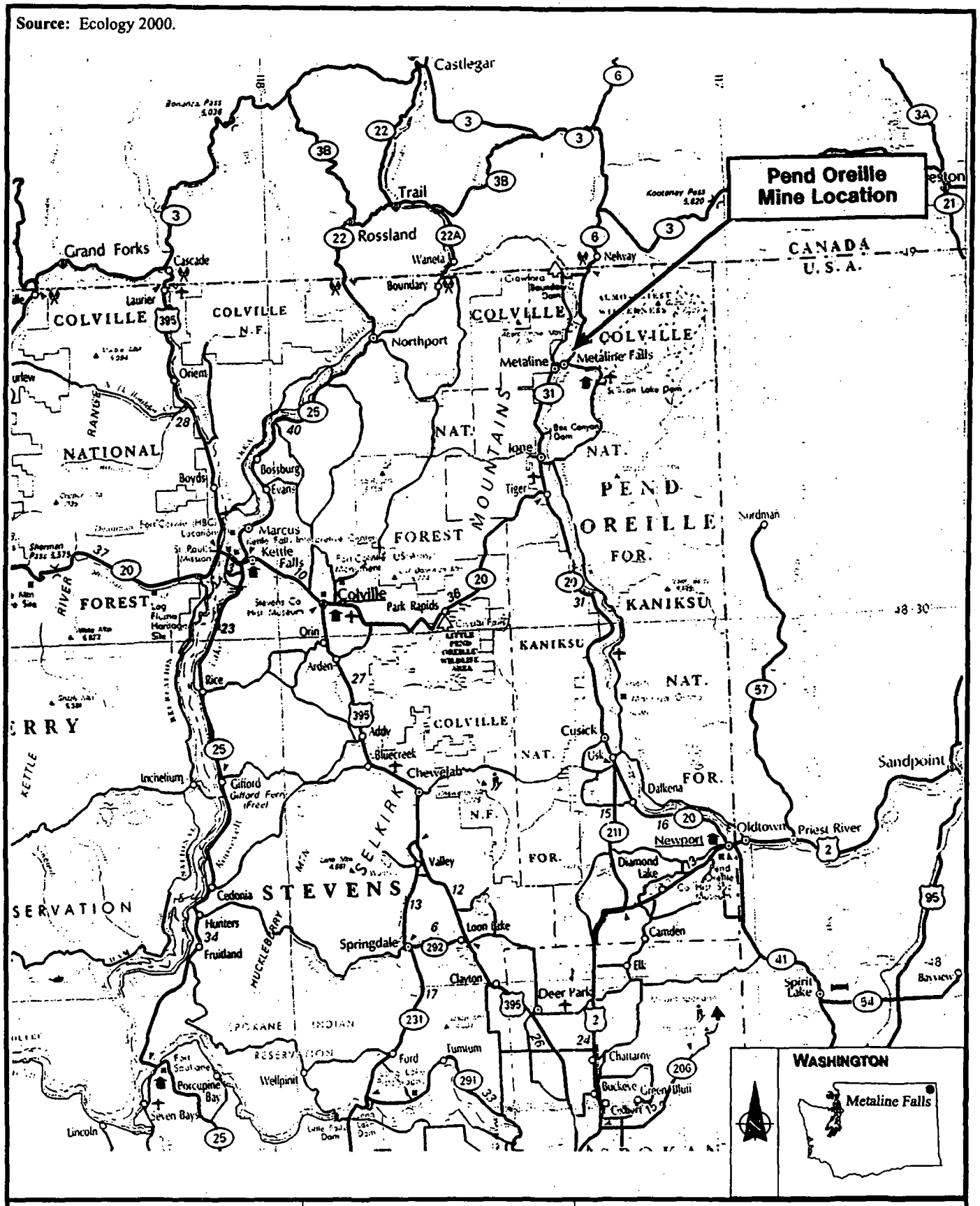
ecology and environment, inc.
International Specialists in the Environment
Seattle, Washington

UPPER COLUMBIA RIVER ESI
MINES AND MILLS
Northeast Washington

Figure 1-1
PROJECT ORGANIZATION CHART

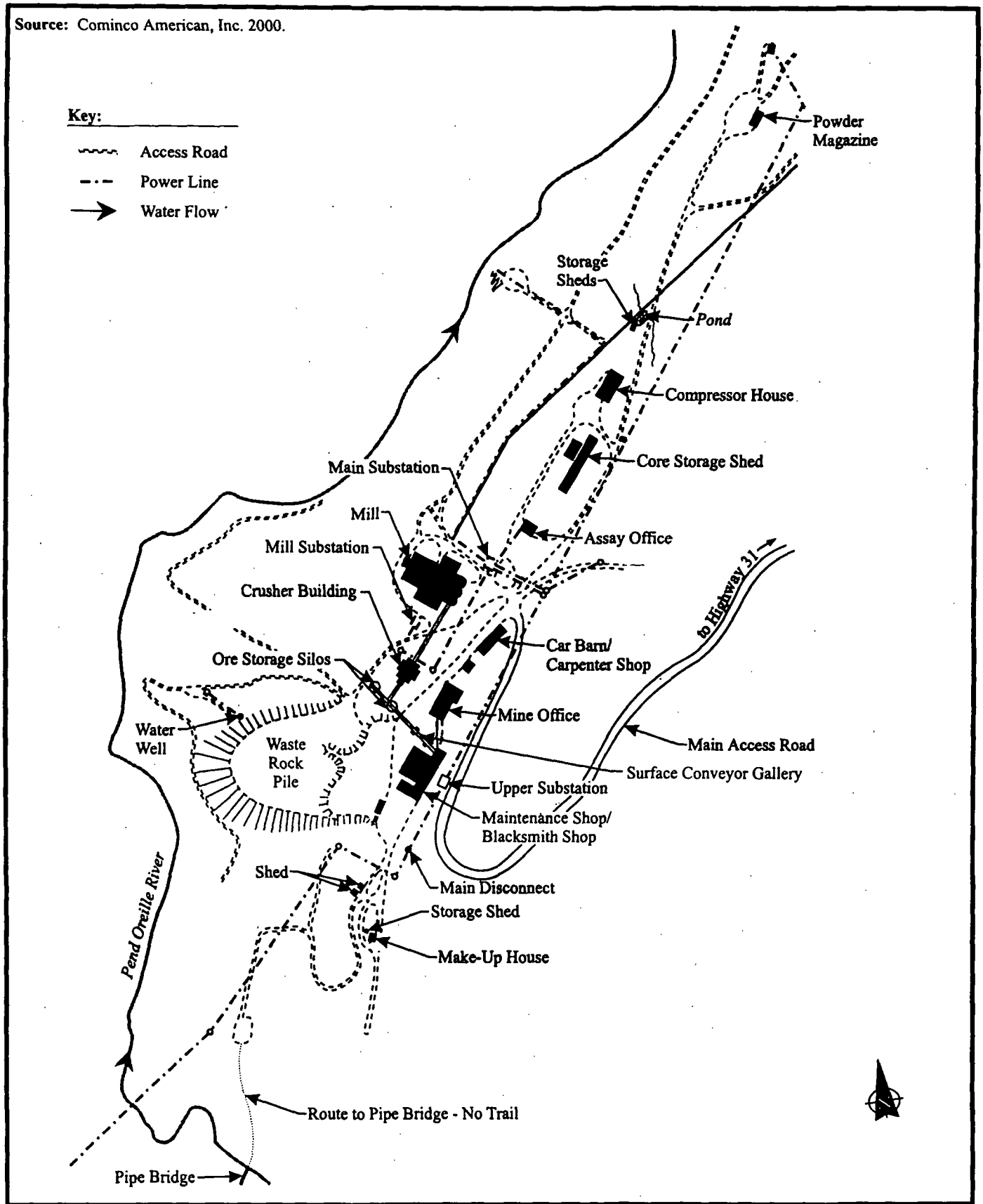
Date:	Drawn by:	
06/07/01	AES	10:START-2\01020028\S643\fig 1-1

Source: Ecology 2000.

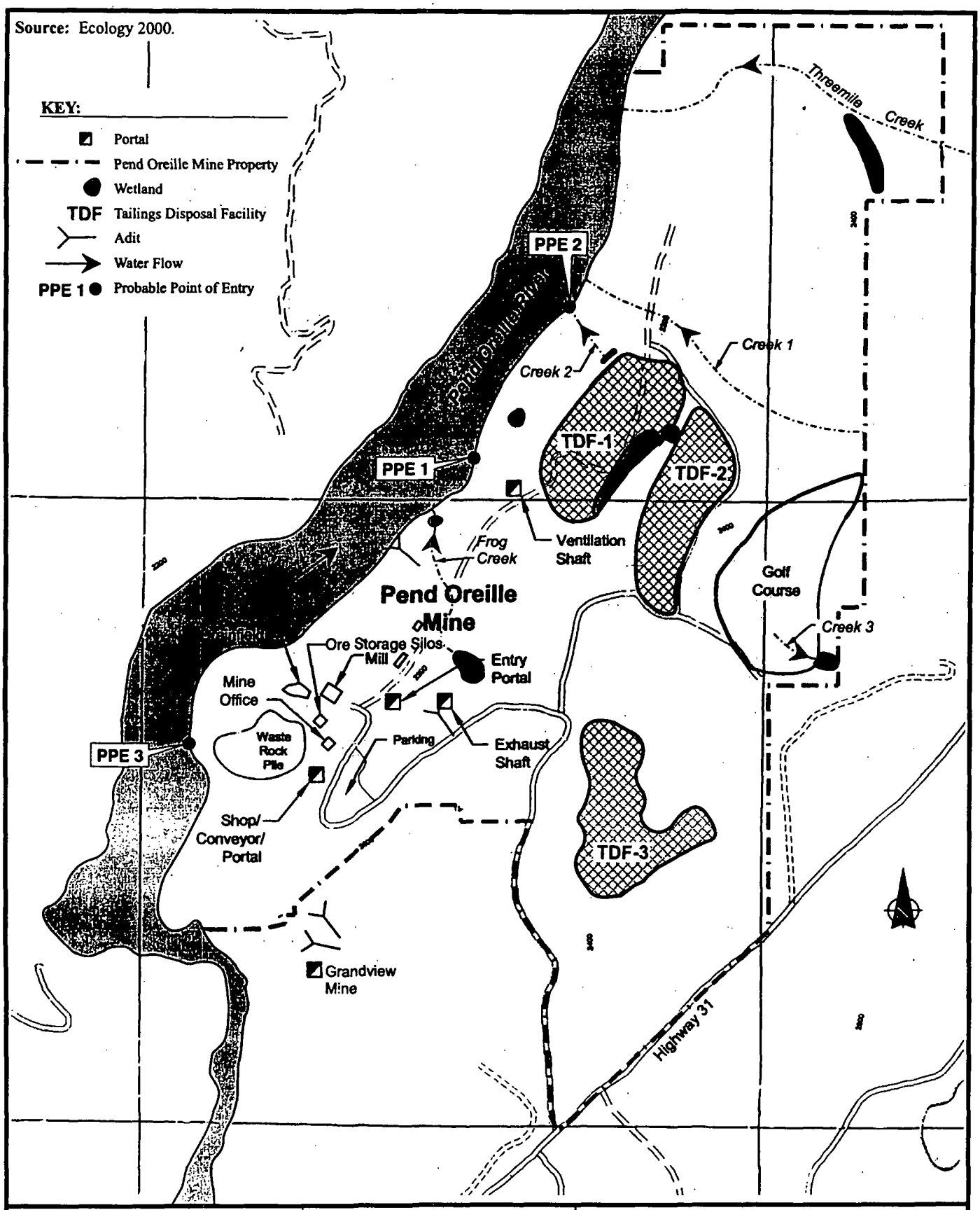


ecology and environment, inc. International Specialists in the Environment Seattle, Washington	UPPER COLUMBIA RIVER ESI MINES AND MILLS PEND OREILLE MINE Pend Oreille County, Washington	Figure 1-2 PEND OREILLE MINE VICINITY MAP	
	0 10 20 Approximate Scale in Miles	Date: 06-07-01	Drawn by: AES 10:START-2\01020028\S643\fig 1-2

Source: Cominco American, Inc. 2000.



ecology and environment, inc. International Specialists in the Environment Seattle, Washington	UPPER COLUMBIA RIVER ESI MINES AND MILLS PEND OREILLE MINE Pend Oreille County, Washington	Figure 1-3 PEND OREILLE MINE LOCATION MAP		
	0 300 600 Approximate Scale in Feet	Date: 06-07-01	Drawn by: AES	10:START-2\01020028\S643\fig 1-3



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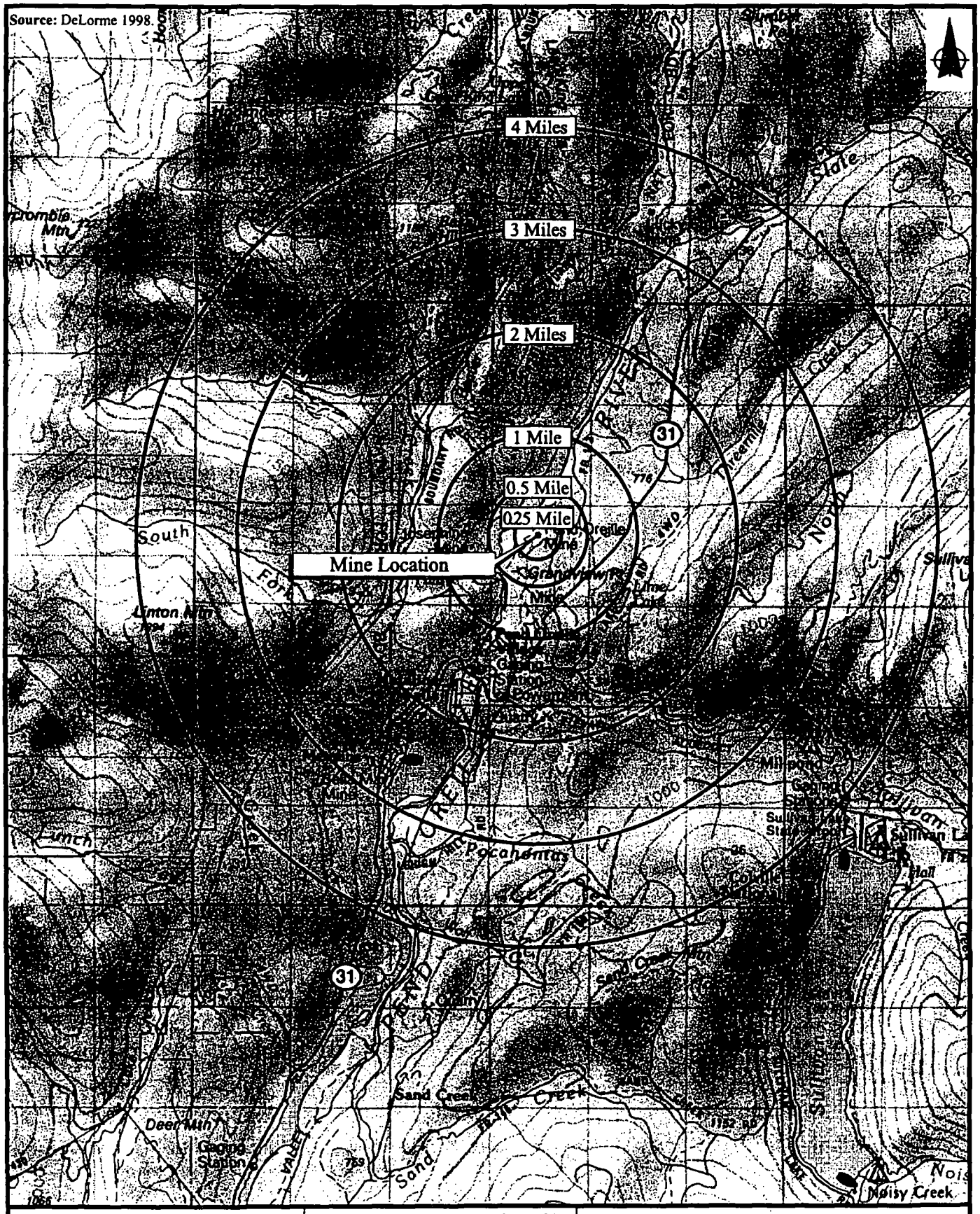
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
PEND OREILLE MINE
Pend Oreille County, Washington

0 1000 2000
Approximate Scale in Meters

Figure 1-4
PEND OREILLE MINE MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-4



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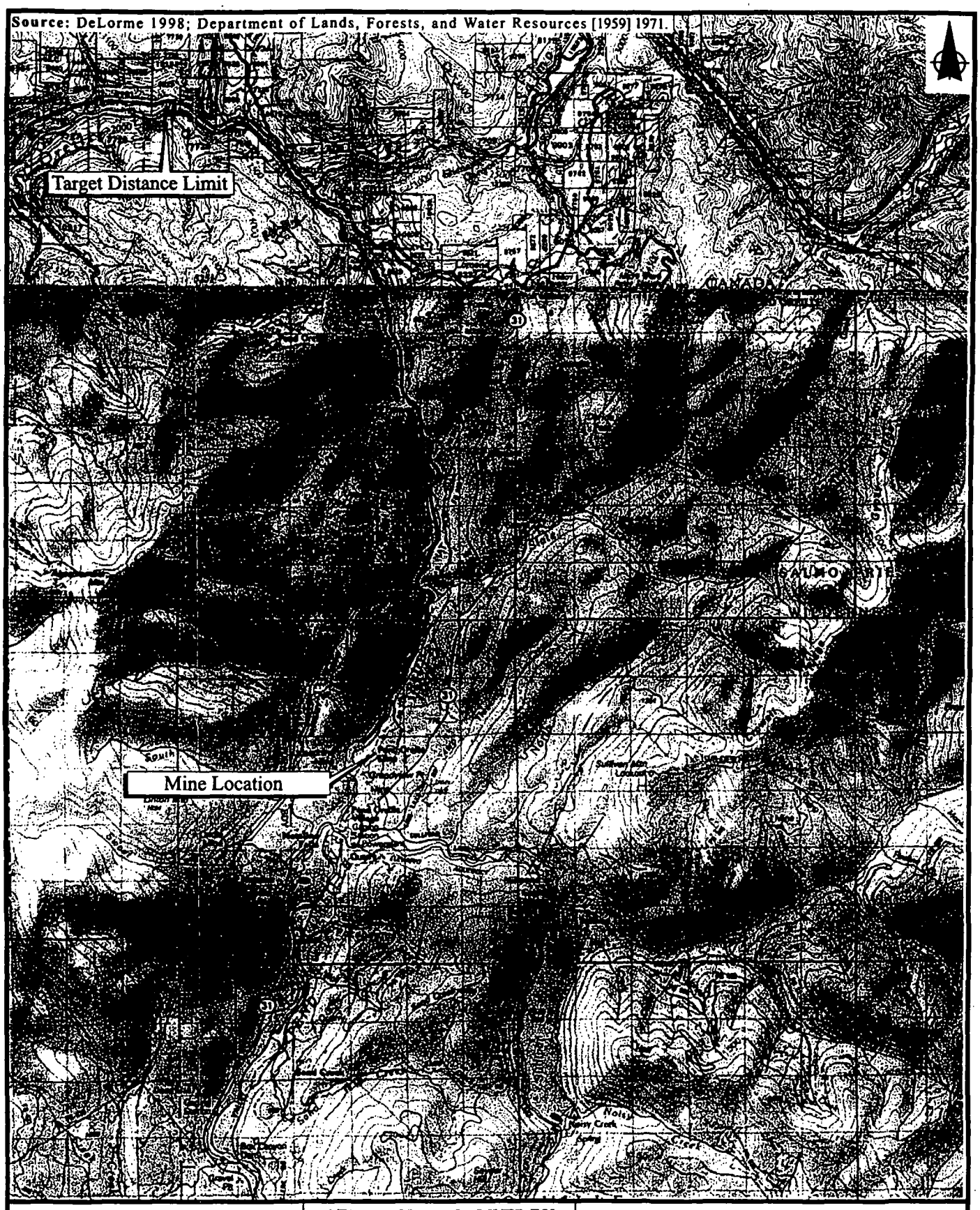
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
PEND OREILLE MINE
Pend Oreille County, Washington

0 .5 1
Approximate Scale in Miles

Figure 1-5
PEND OREILLE MINE
4-MILE MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-5



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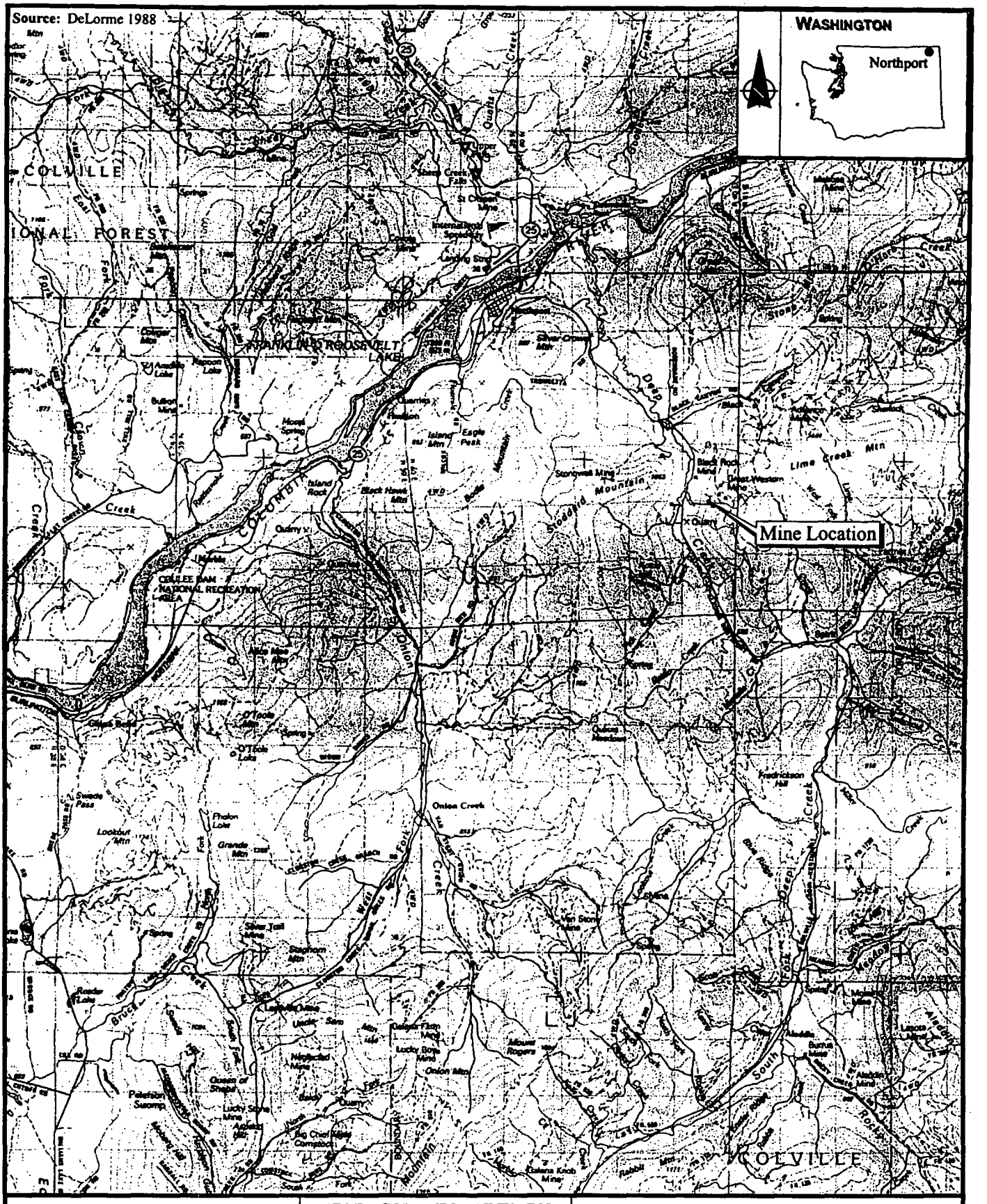
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
PEND OREILLE MINE
Pend Oreille County, Washington

0 1 2
Approximate Scale in Miles

Figure 1-6
PEND OREILLE MINE
15-MILE MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-6



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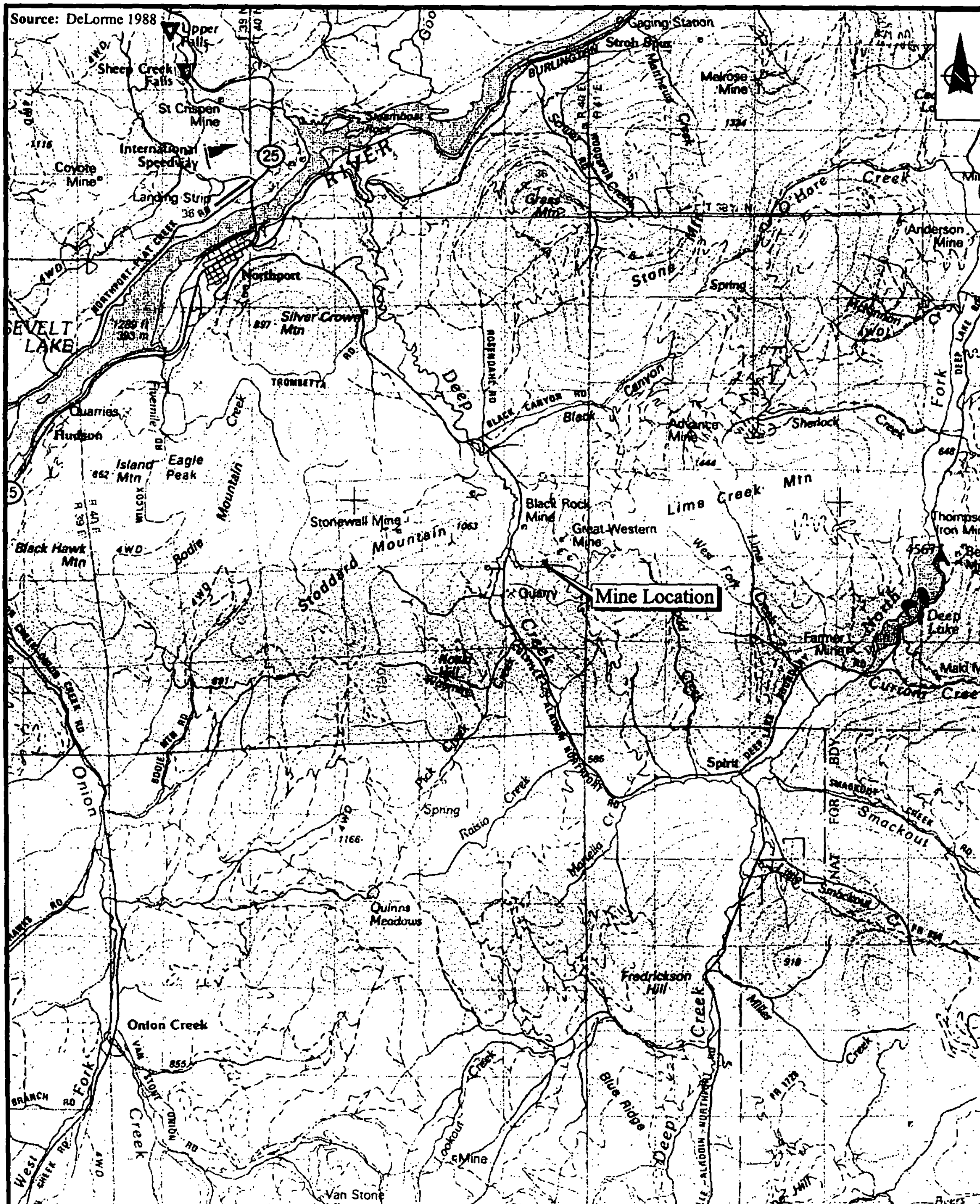
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
LAST CHANCE MINE
Stevens County, Washington

0 1.5 3
Approximate Scale in Miles

Figure 1-7
LAST CHANCE MINE
VICINITY MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-7



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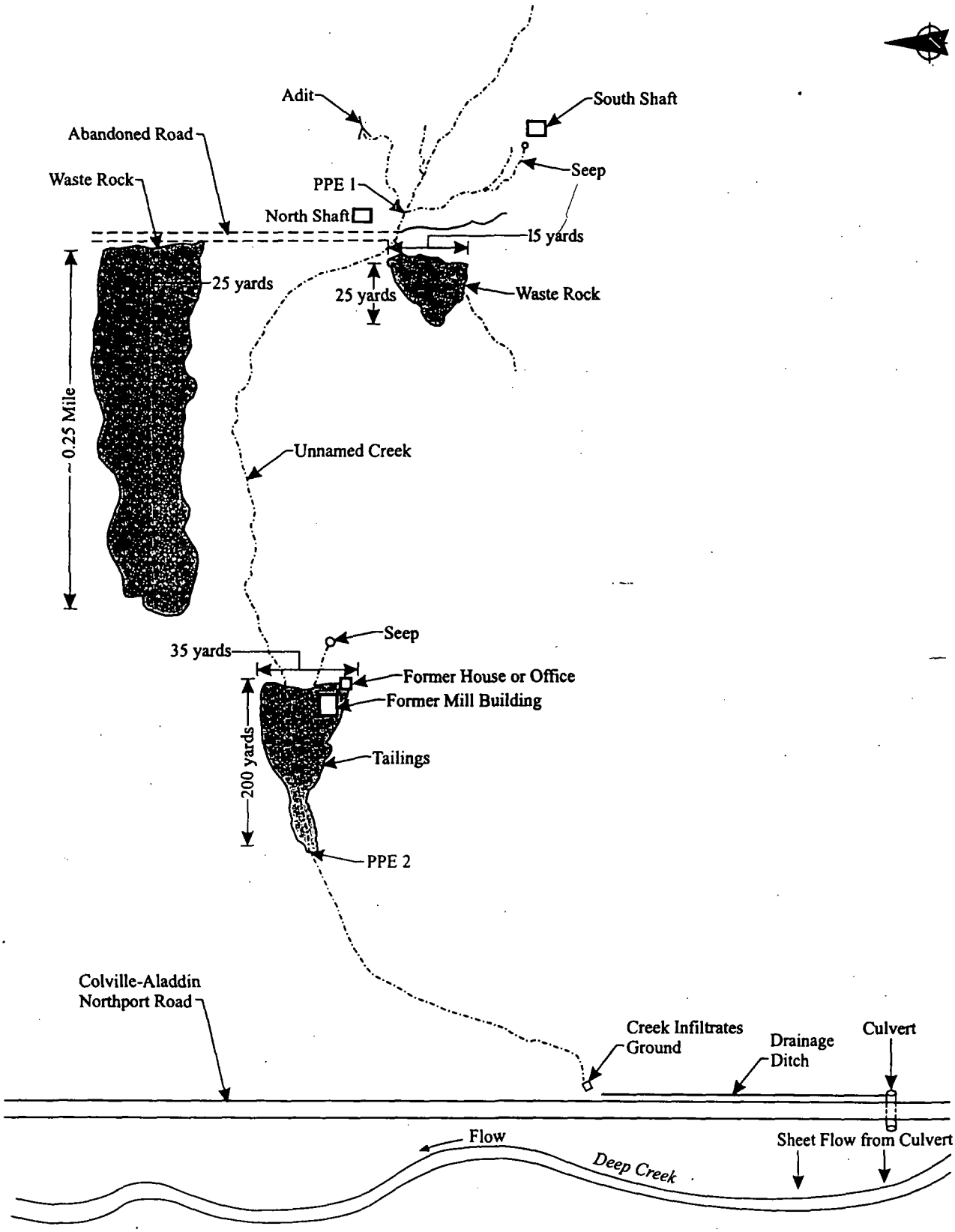
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
LAST CHANCE MINE
Stevens County, Washington

Figure 1-8

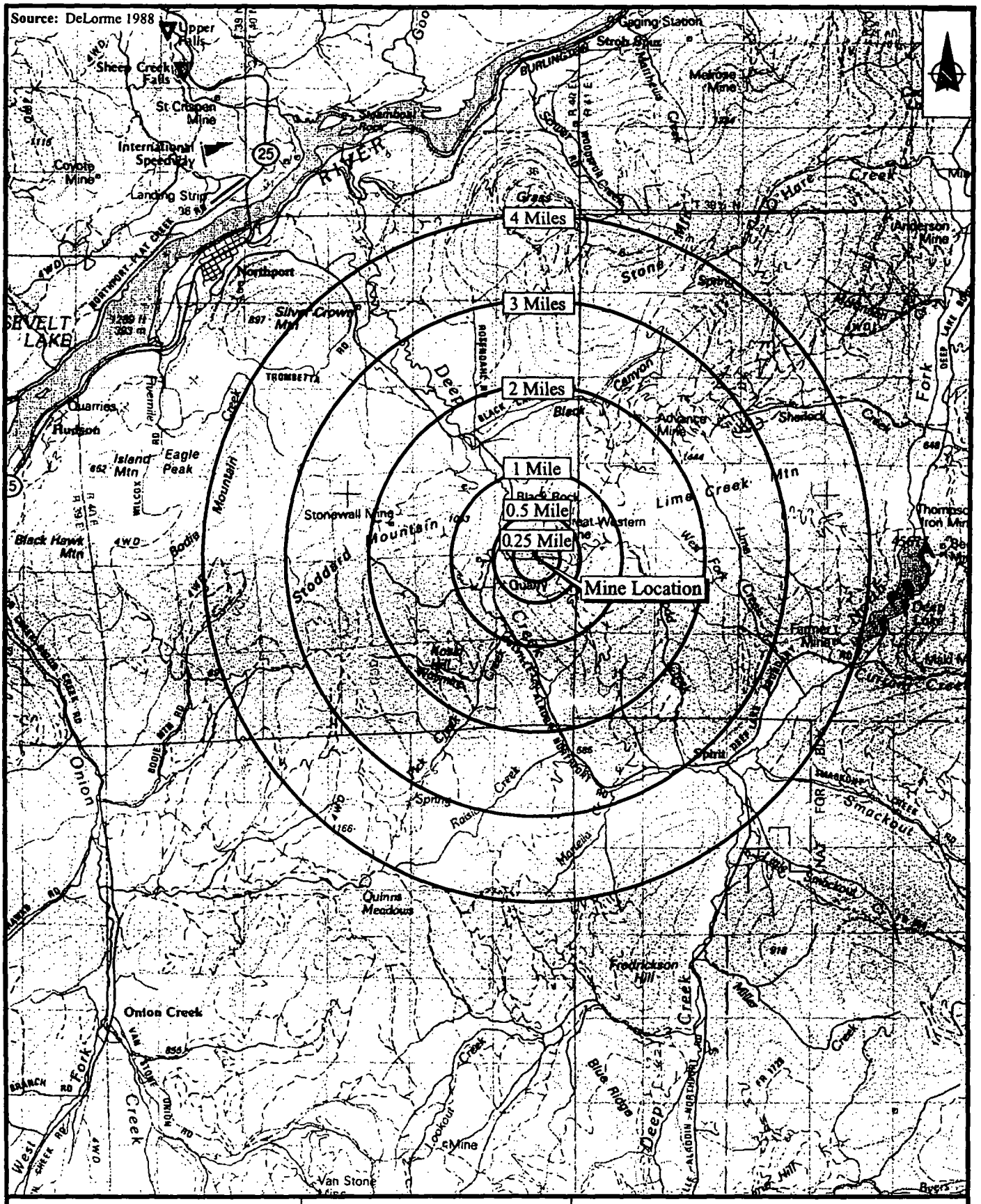
LAST CHANCE MINE
LOCATION MAP

0 1 2
Approximate Scale in Miles

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-8



ecology and environment, inc. International Specialists in the Environment Seattle, Washington	UPPER COLUMBIA RIVER ESI MINES AND MILLS LAST CHANCE MINE Stevens County, Washington	Figure 1-9 LAST CHANCE MINE MAP		
	Not to Scale	Date: 06-07-01	Drawn by: AES	10:START-2\01020028\S643\fig 1-9



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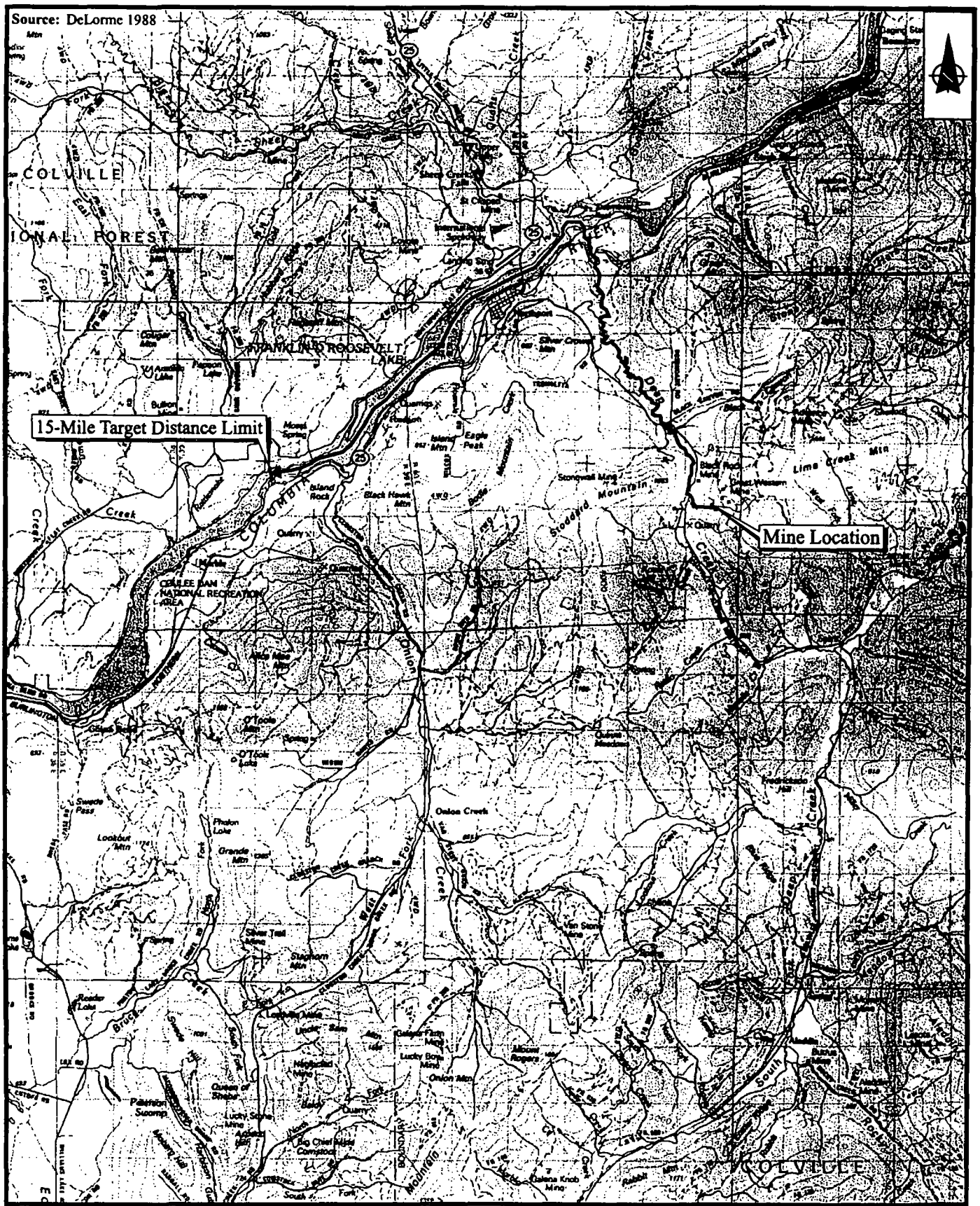
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
LAST CHANCE MINE
Stevens County, Washington

Figure 1-10
LAST CHANCE MINE
4-MILE MAP

0 1 2
Approximate Scale in Miles

Date: 06-07-01
Drawn by: AES
10:START-2\01020028\S643\fig 1-10



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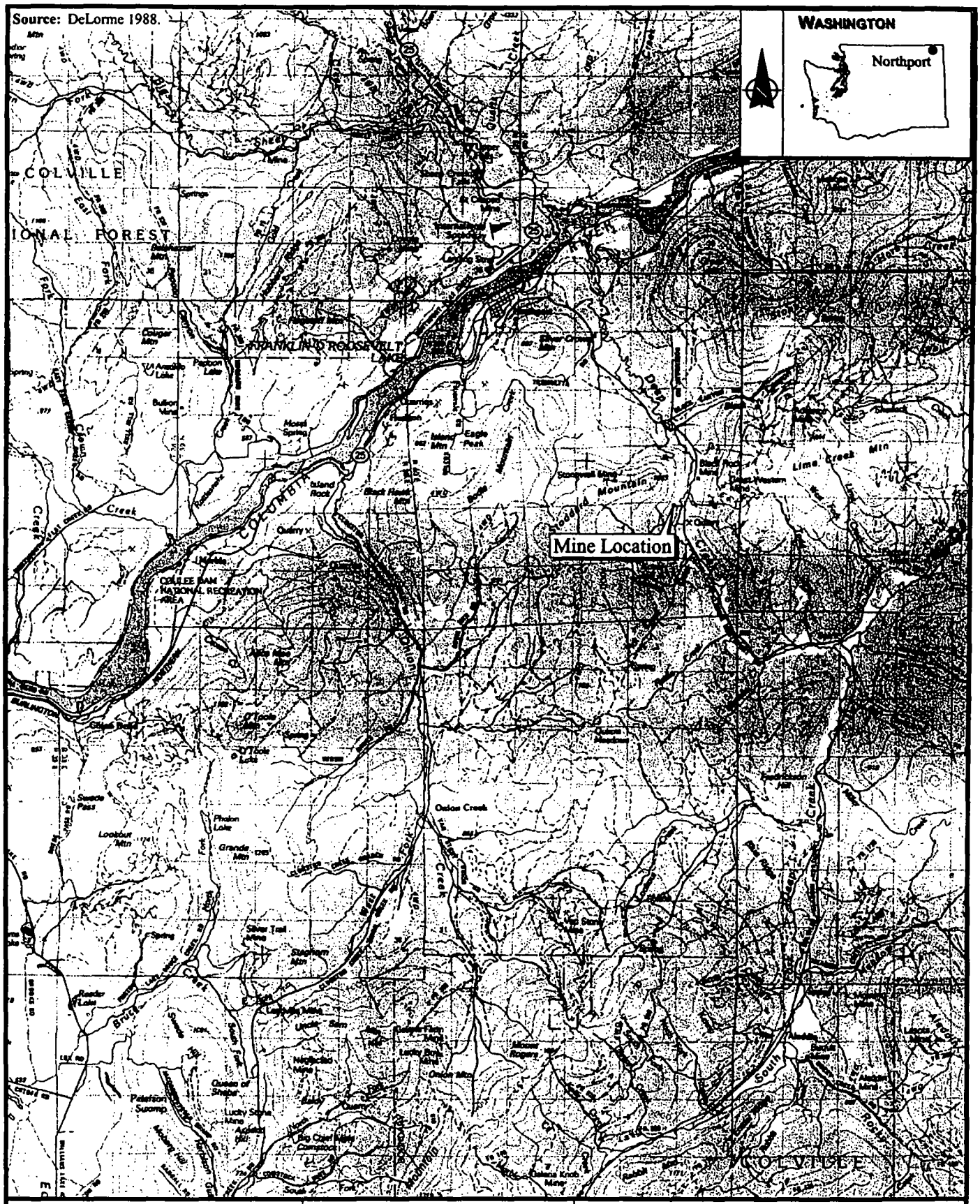
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
LAST CHANCE MINE
Stevens County, Washington

Figure 1-11
LAST CHANCE MINE
15-MILE MAP

0 1.5 3
Approximate Scale in Miles

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-11



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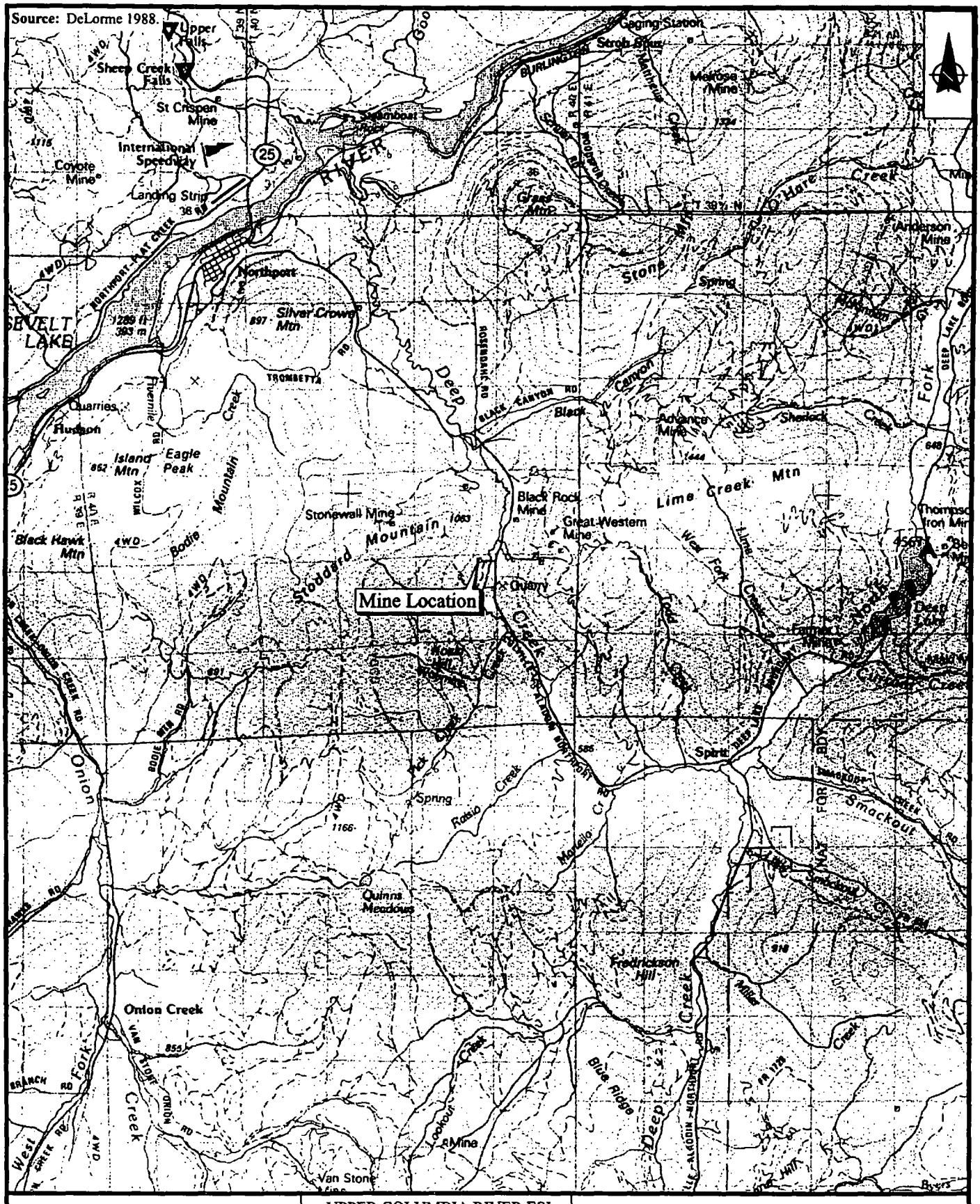
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
DEEP CREEK MINE
Stevens County, Washington

Figure 1-12
DEEP CREEK MINE
VICINITY MAP

0 1.5 3
Approximate Scale in Miles

Date: 06-07-01
Drawn by: AES
10:START-2\01020028\S643\fig 1-12



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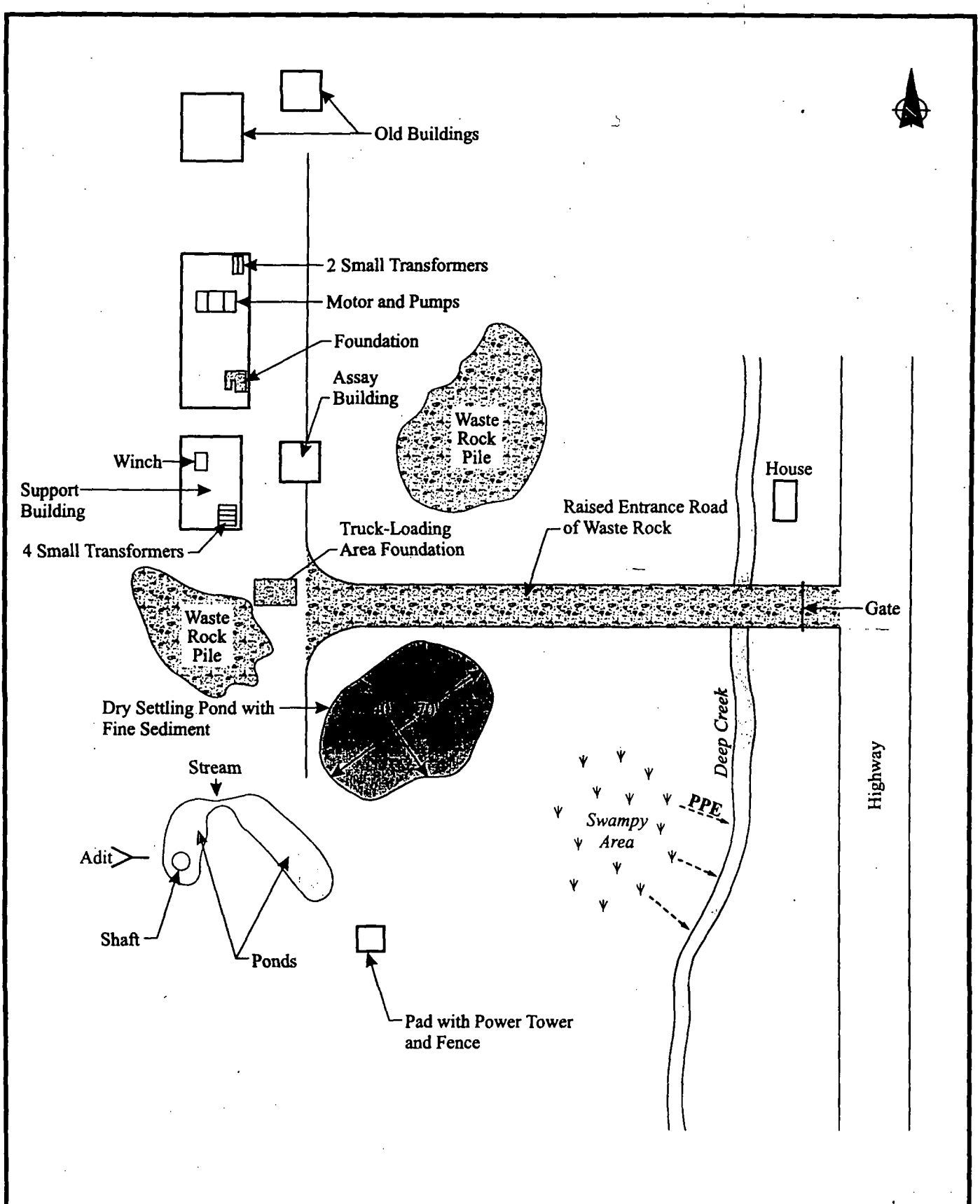
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
DEEP CREEK MINE
Stevens County, Washington

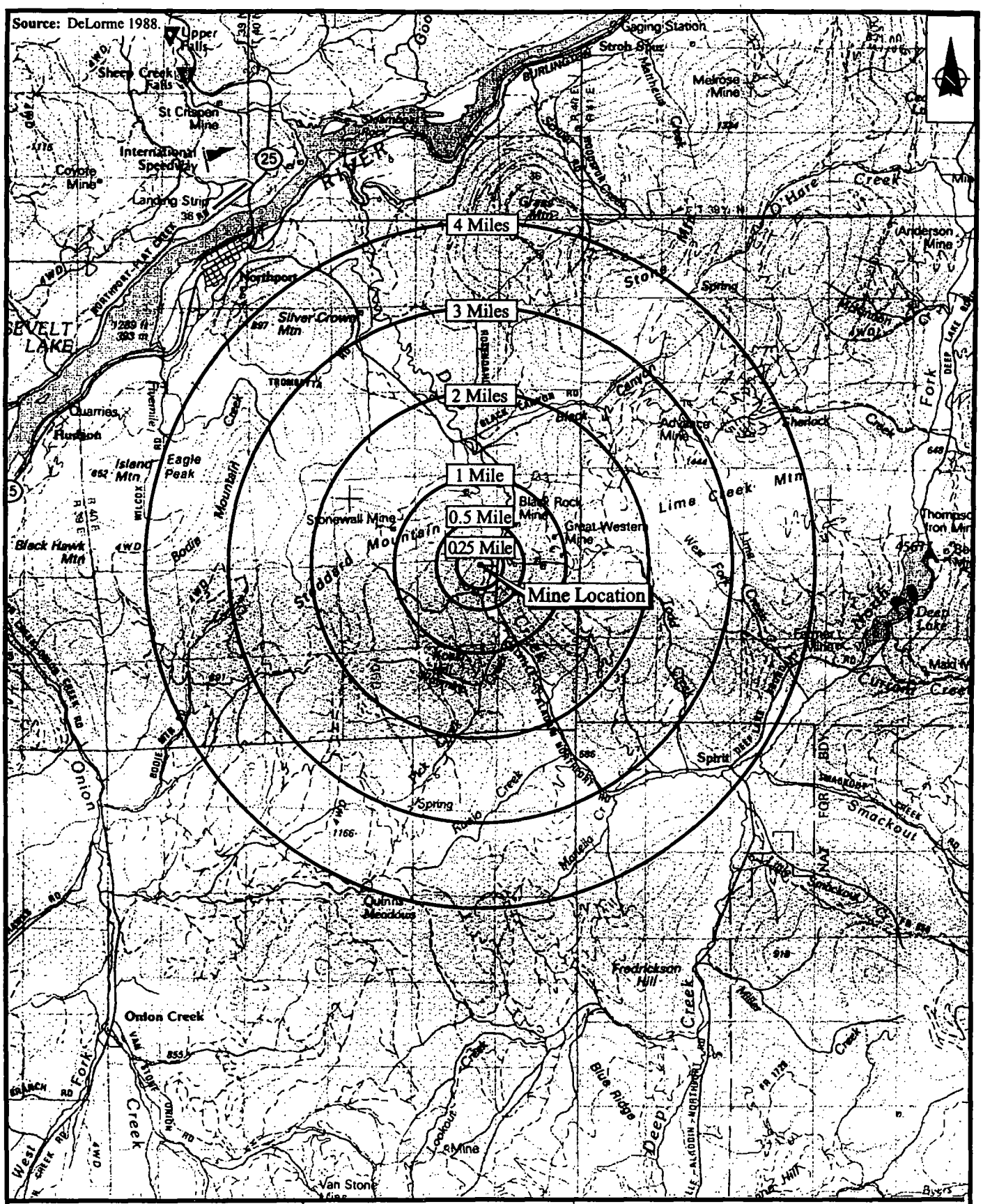
0 1 2
Approximate Scale in Miles

Figure 1-13
DEEP CREEK MINE
LOCATION MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-13



ecology and environment, inc. International Specialists in the Environment Seattle, Washington	UPPER COLUMBIA RIVER ESI MINES AND MILLS DEEP CREEK MINE Stevens County, Washington	Figure 1-14 DEEP CREEK MINE MAP		
	Not to Scale	Date: 06-07-01	Drawn by: AES	10:START-2\01020028\S643\fig 1-14



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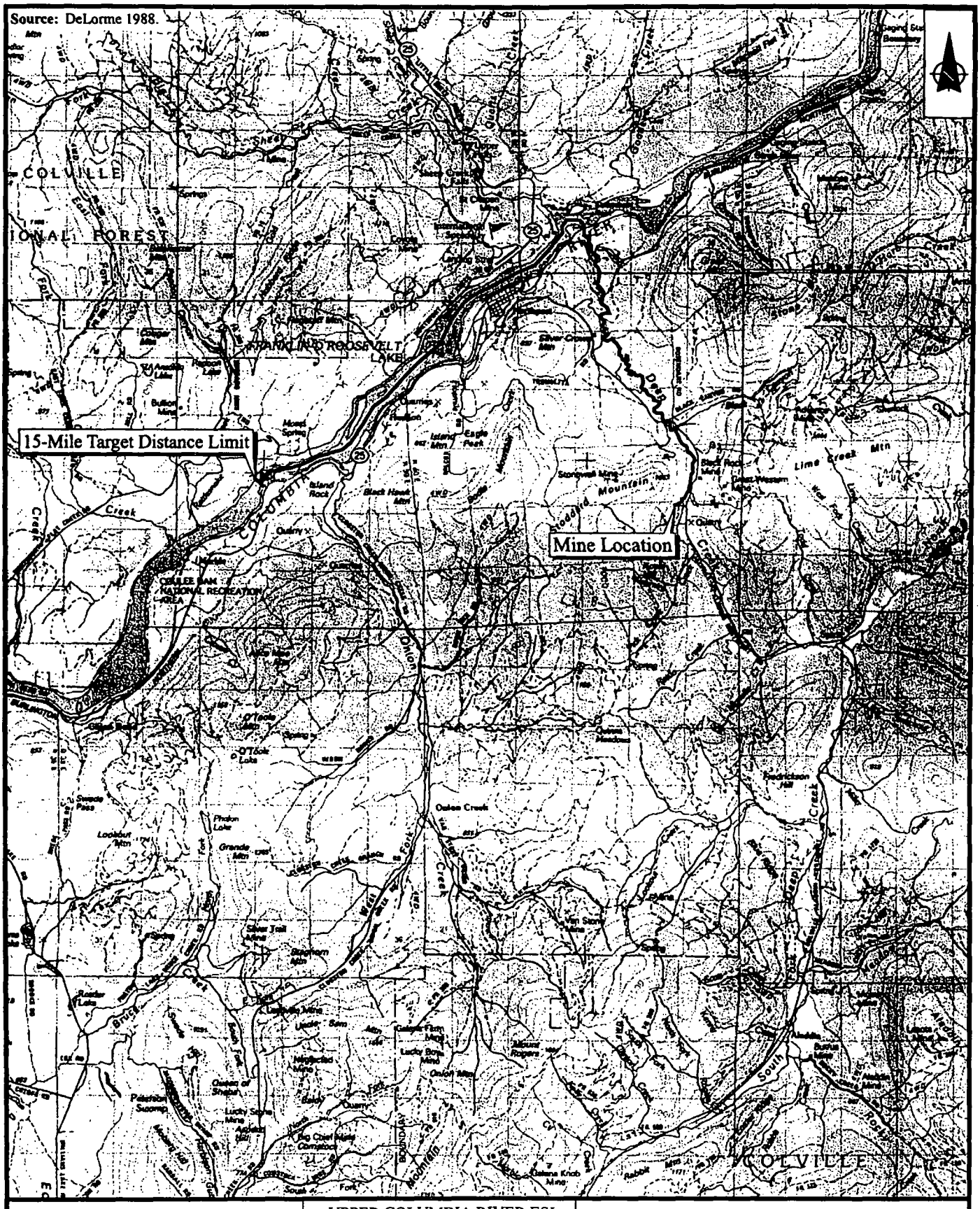
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
DEEP CREEK MINE
Stevens County, Washington

0 1 2
Approximate Scale in Miles

Figure 1-15
DEEP CREEK MINE
4-MILE MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-15



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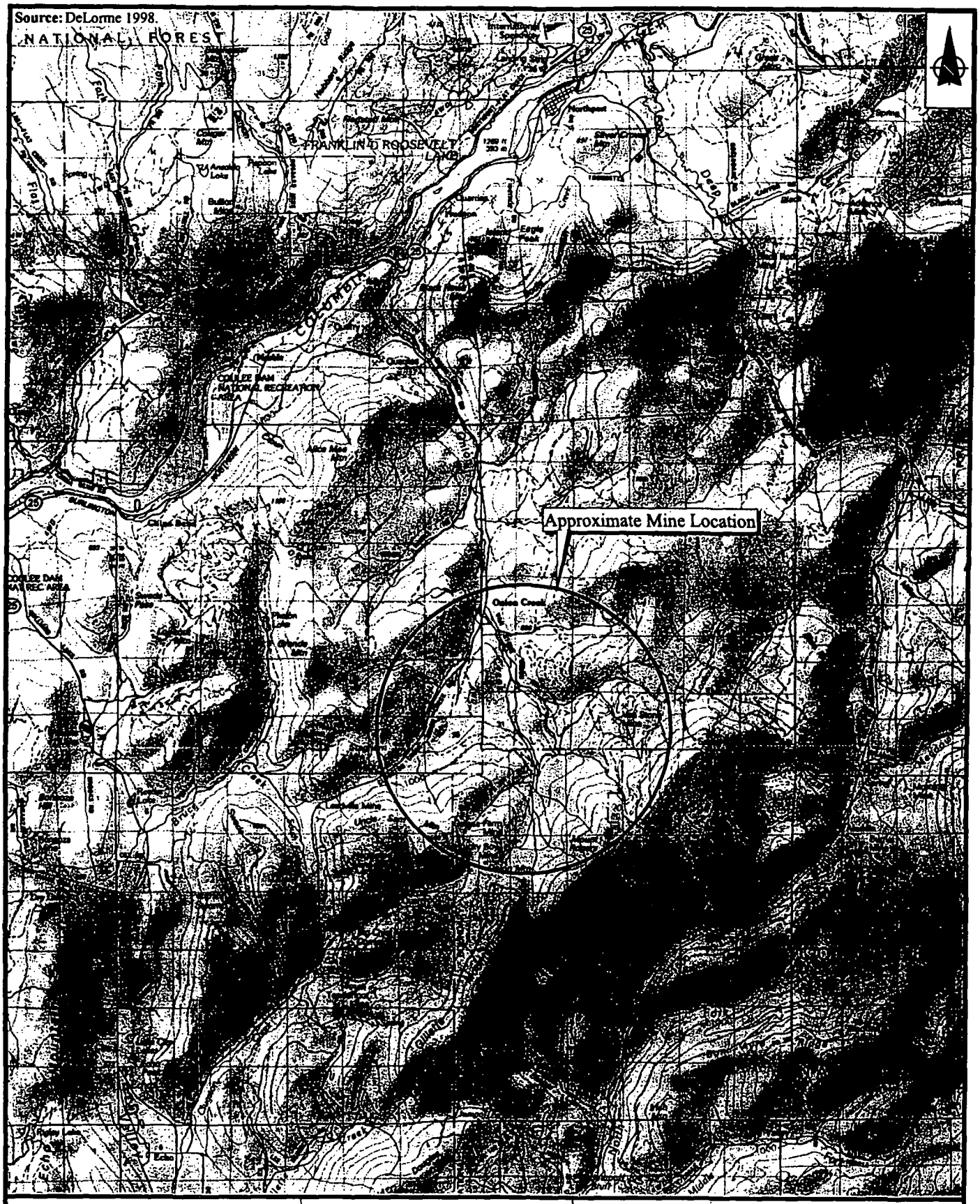
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
DEEP CREEK MINE
Stevens County, Washington

Figure 1-16
DEEP CREEK MINE
15-MILE MAP

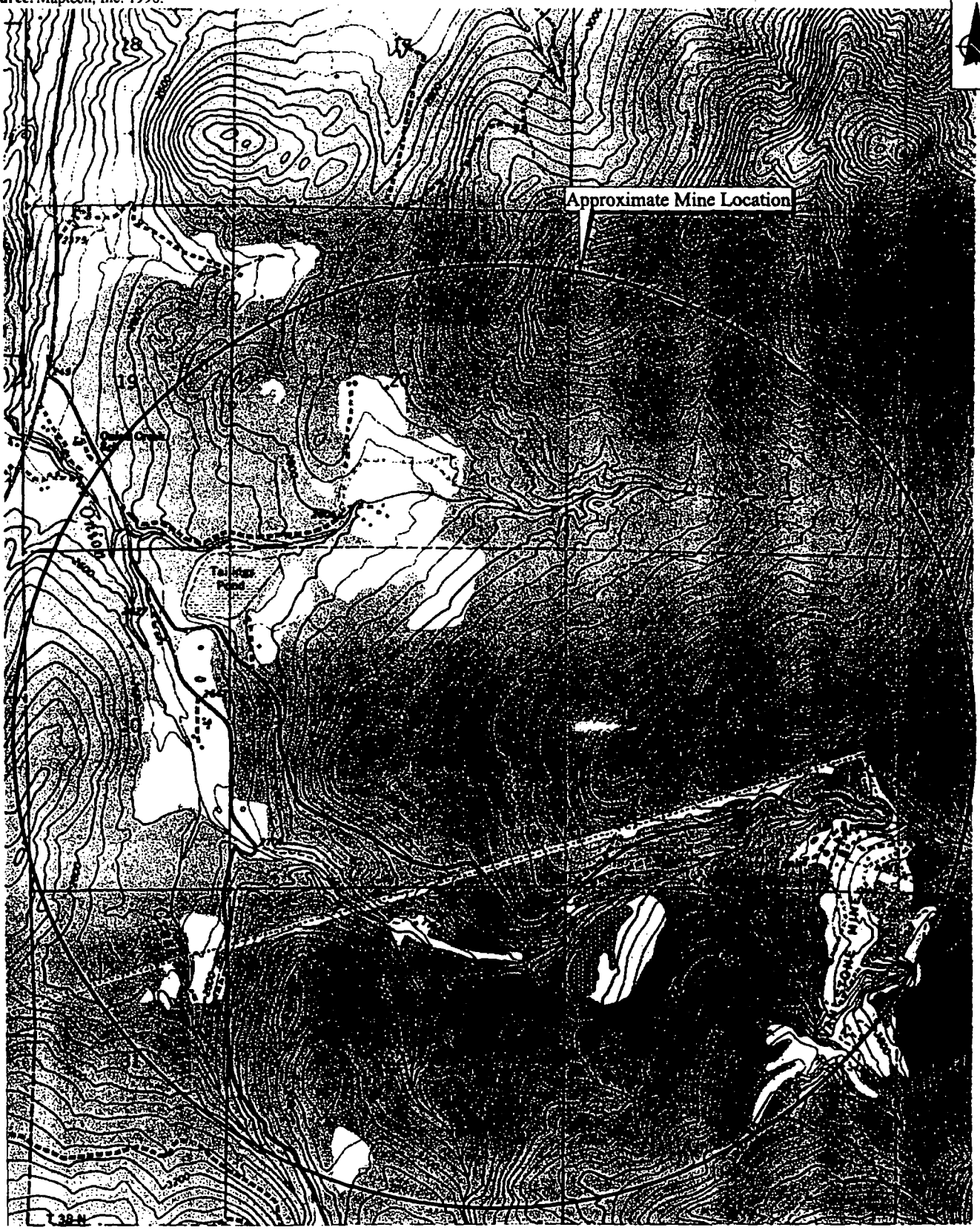
0 1.5 3
Approximate Scale in Miles

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-16



ecology and environment, inc. International Specialists in the Environment Seattle, Washington	UPPER COLUMBIA RIVER ESI MINES AND MILLS VAN STONE MINE Stevens County, Washington	Figure 1-17 VAN STONE MINE VICINITY MAP	
	0 1.5 3 Approximate Scale in Miles	Date: 06-07-01	Drawn by: AES	10:START-2\01020028\S643\fig 1-17

Source: Maptech, Inc. 1998.



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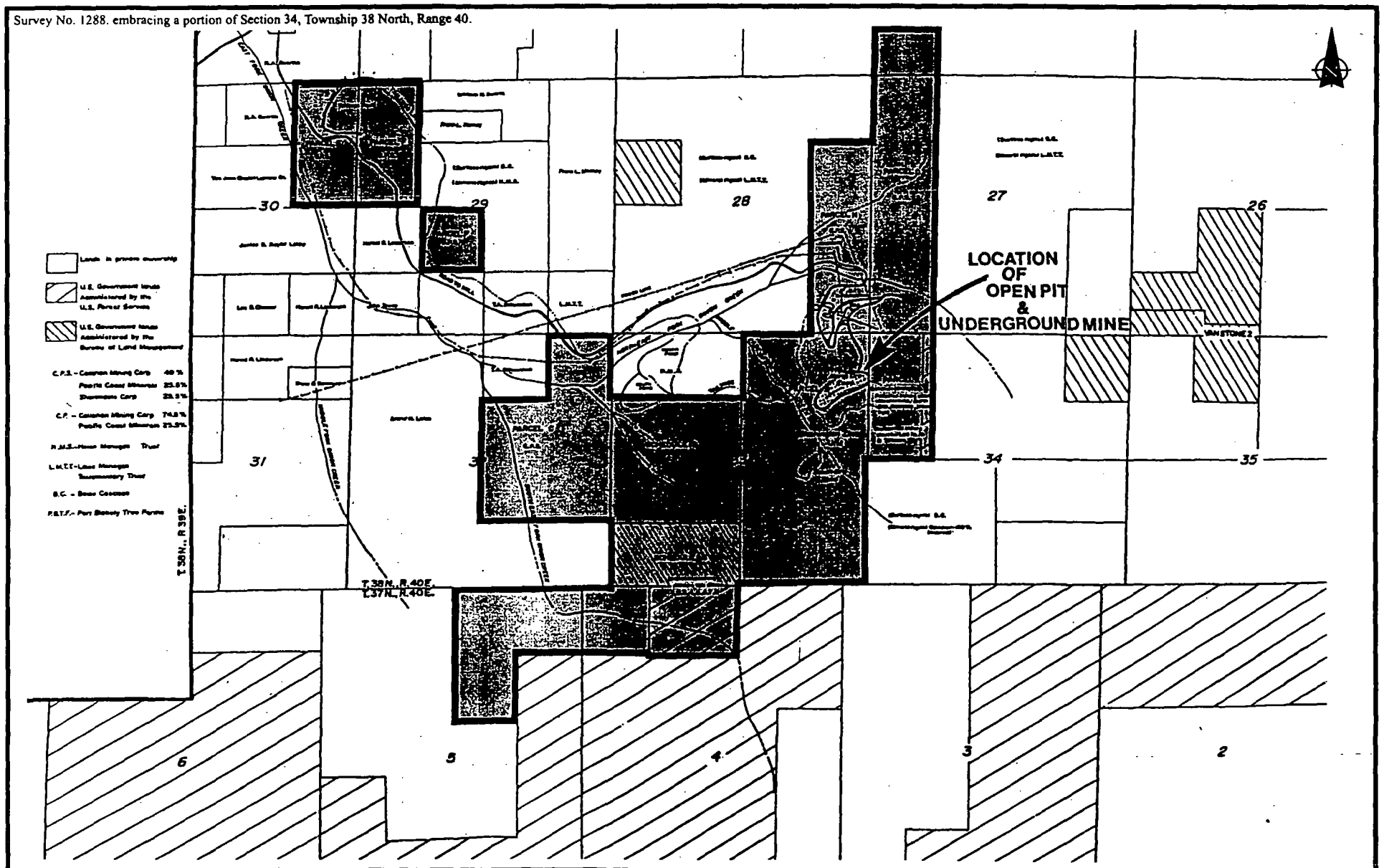
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
VAN STONE MINE
Stevens County, Washington

0 .25 .5
Approximate Scale in Miles

Figure 1-18

VAN STONE MINE LOCATION MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-18



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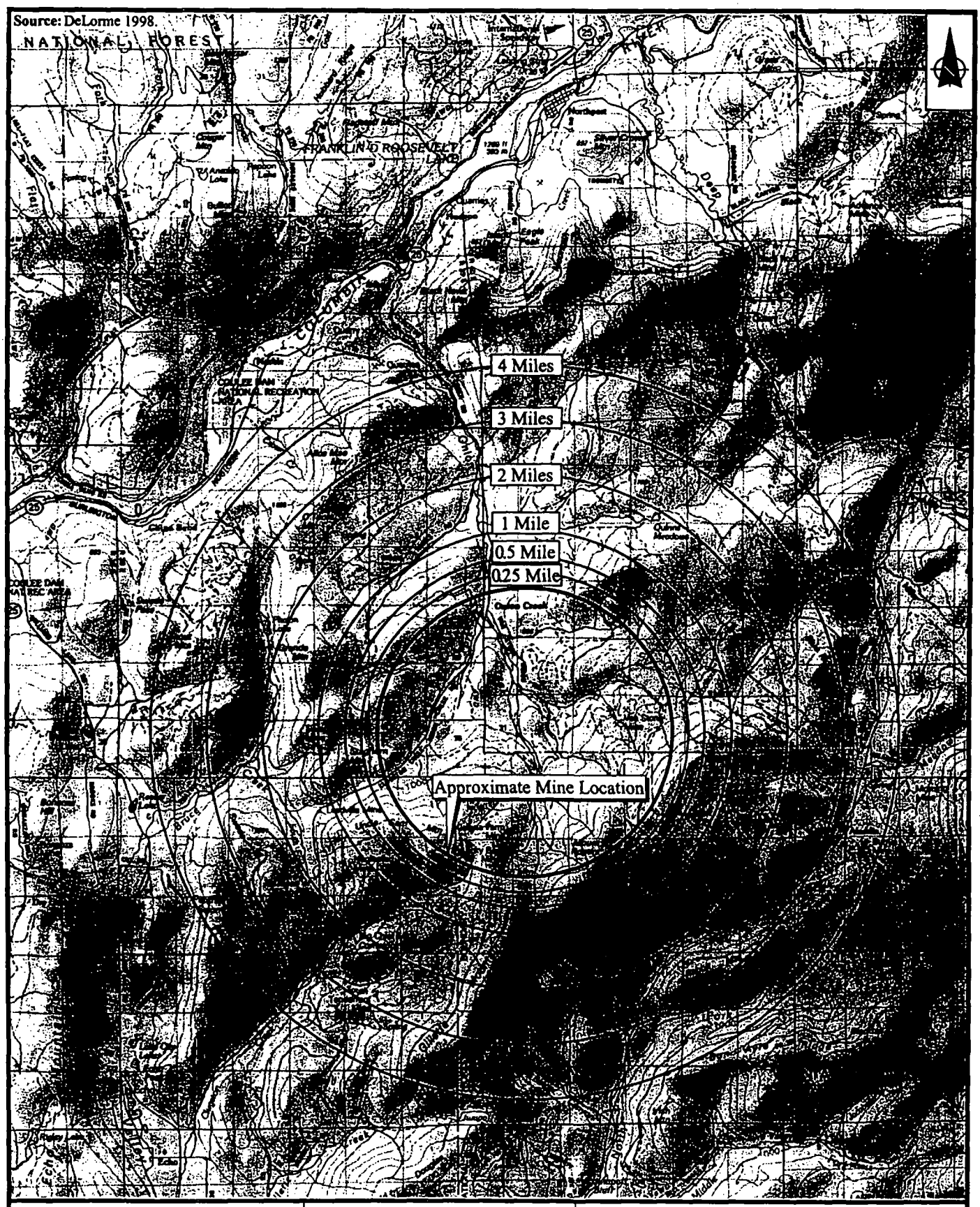
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
VAN STONE MINE
Stevens County, Washington

0 1.5 3
Approximate Scale in Miles

Figure 1-19
VAN STONE MINE MAP

Date:	Drawn by:	
06/07/01	AES	10:START-2\01020028\S643\fig 1-19



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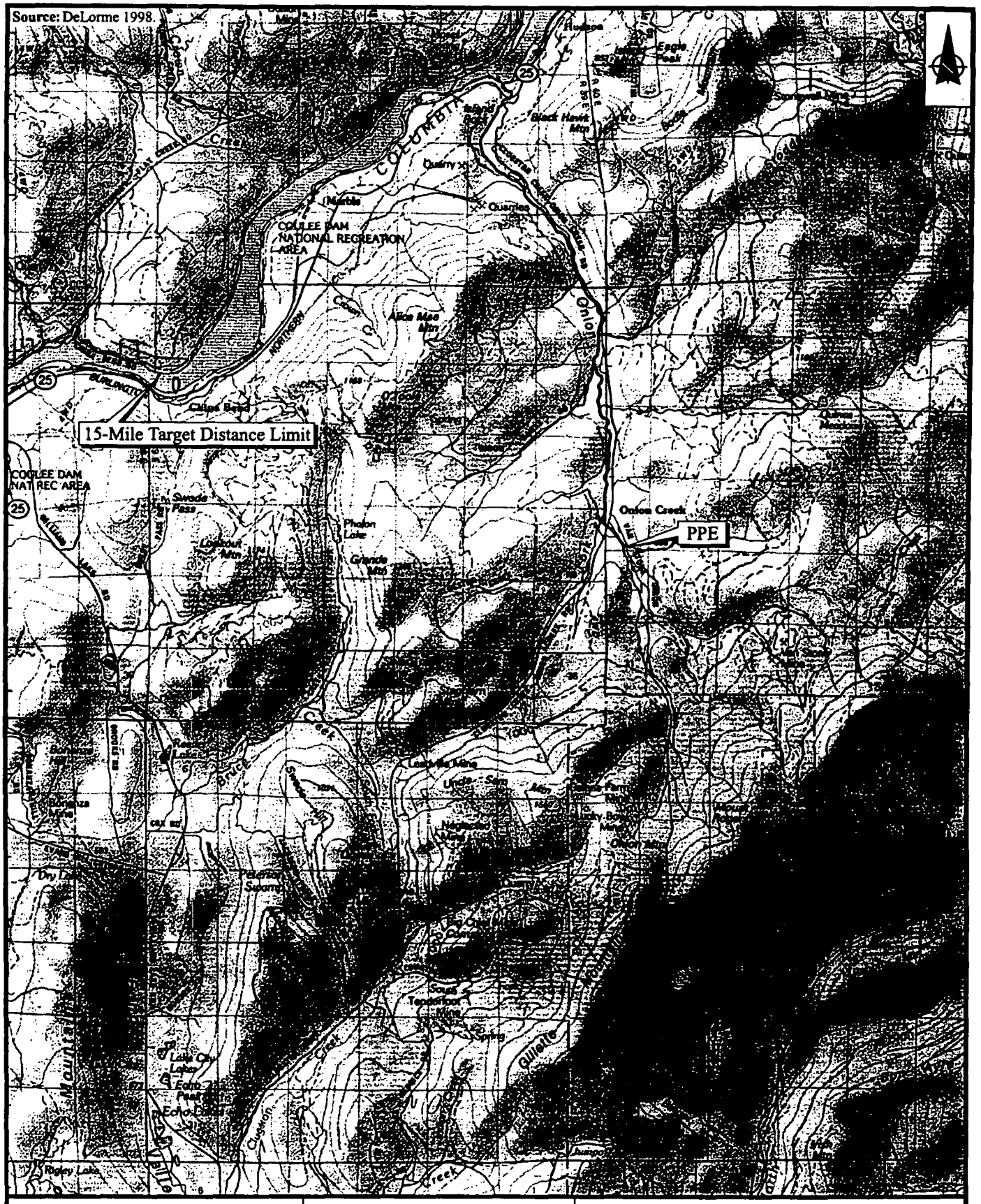
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
VAN STONE MINE
Stevens County, Washington

Figure 1-20

VAN STONE MINE 4-MILE MAP

0 1.5 3
Approximate Scale in Miles

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-20



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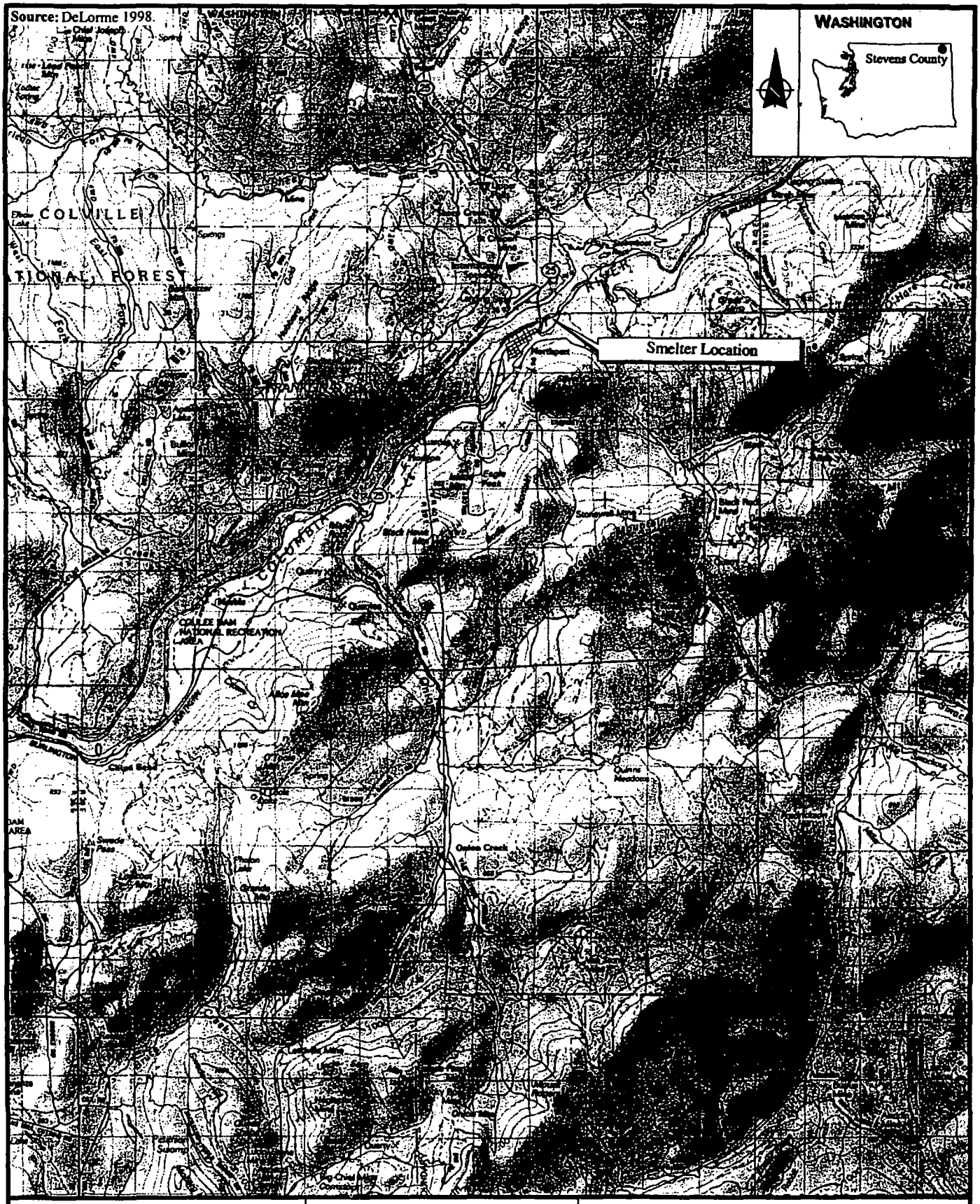
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
VAN STONE MINE
Stevens County, Washington

0 1.5 3
Approximate Scale in Miles

Figure 1-21

VAN STONE MINE 15-MILE MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-21



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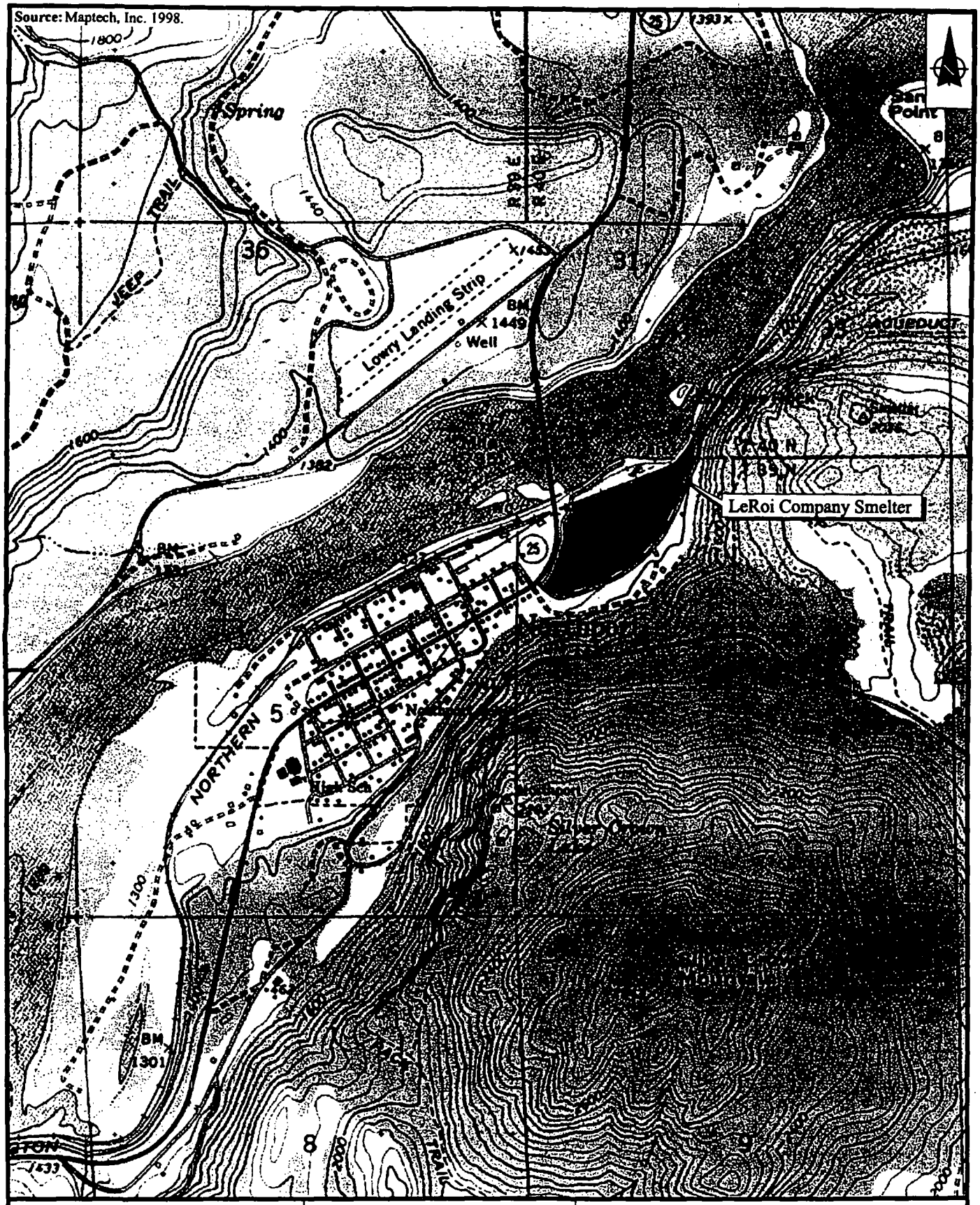
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
LEROI COMPANY SMELTER
Stevens County, Washington

Figure 1-22
LEROI COMPANY SMELTER
VICINITY MAP

0 1.5 3
Approximate Scale in Miles

Date: 06-07-01
Drawn by: AES
10:START-2\01020028\S643\fig 1-22



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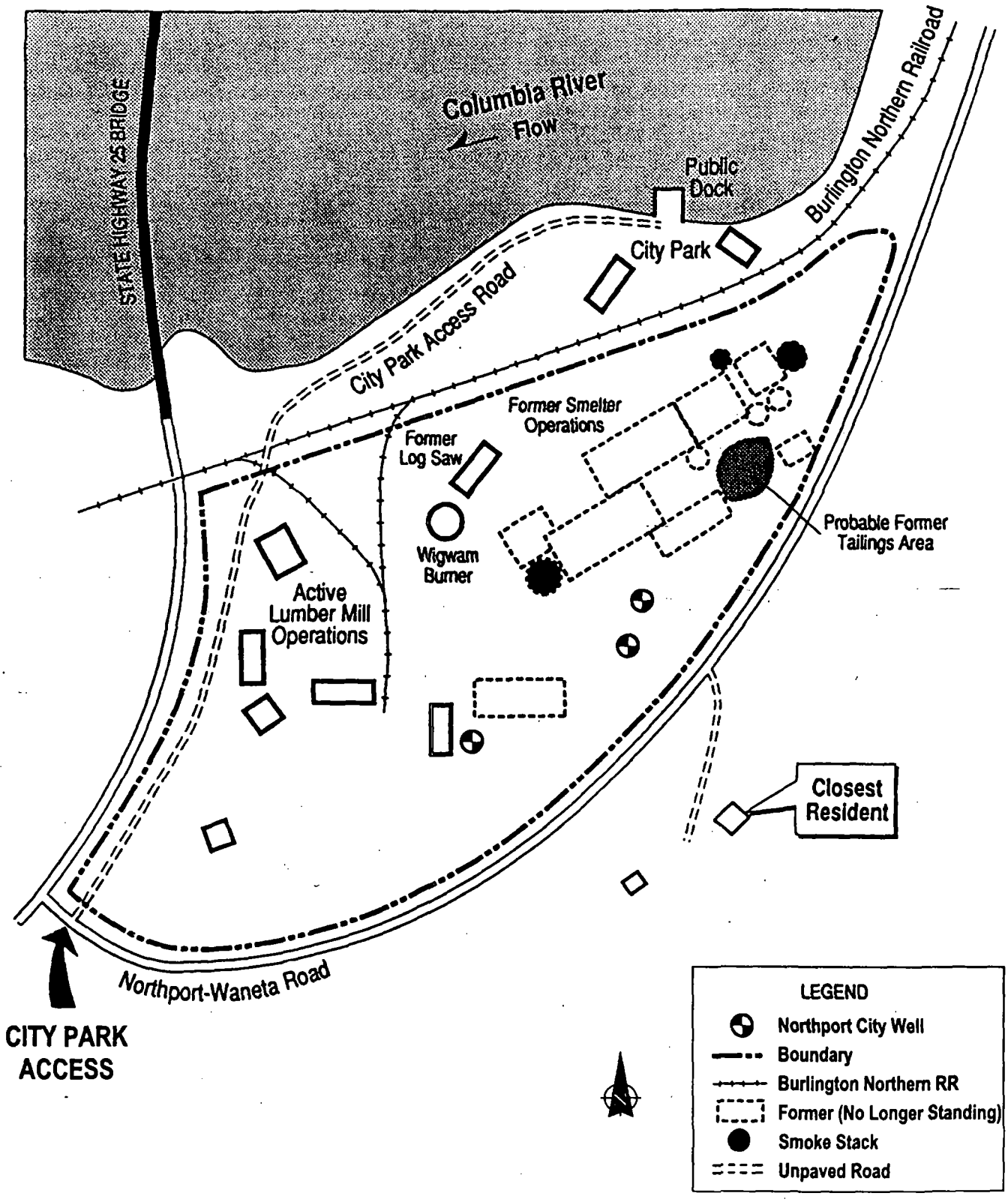
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
LEROI COMPANY SMELTER
Stevens County, Washington

0 .25 .5
Approximate Scale in Miles

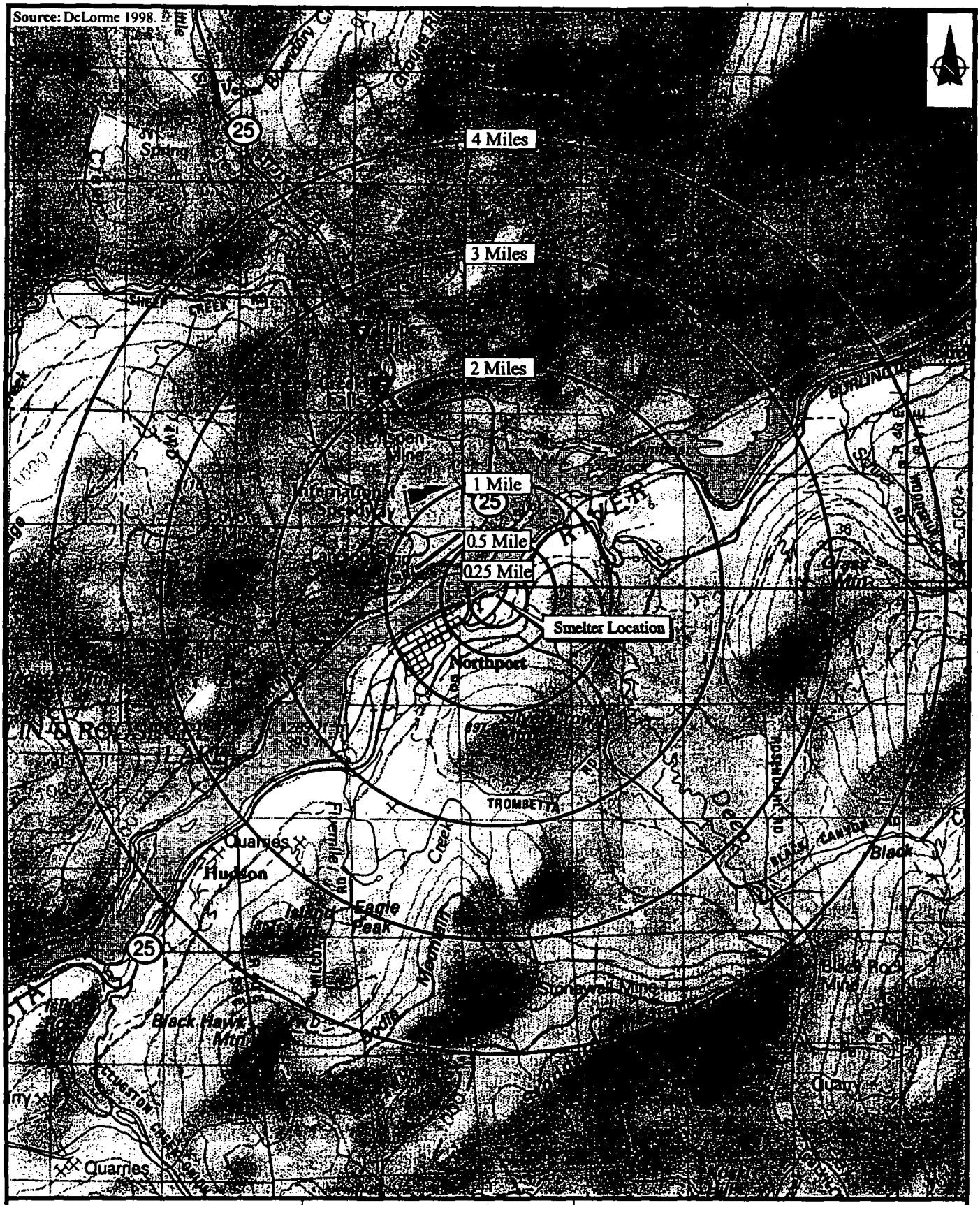
Figure 1-23
LEROI COMPANY SMELTER
LOCATION MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-23

Source: URS Consultants, Inc.



ecology and environment, inc. International Specialists in the Environment Seattle, Washington	UPPER COLUMBIA RIVER ESI MINES AND MILLS LEROI COMPANY SMELTER Stevens County, Washington	Figure 1-24 LEROI COMPANY SMELTER MAP		
	0 150 300 Approximate Scale in Feet	Date: 06-07-01	Drawn by: AES	10:START-2\01020028\S643\fig 1-24



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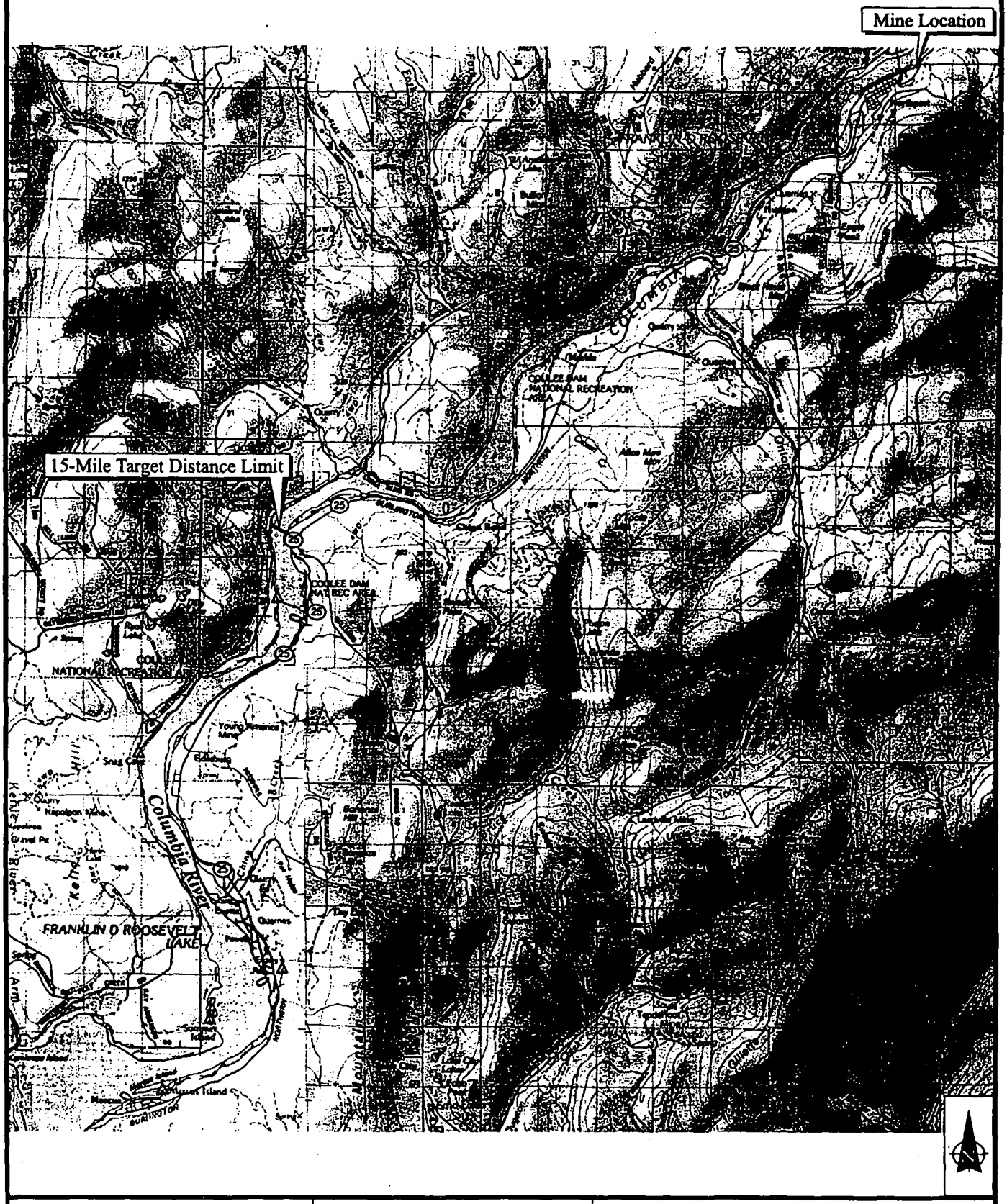
UPPER COLUMBIA RIVER ESI
MINES AND MILLS
LEROI COMPANY SMELTER
Stevens County, Washington

0 1 2
Approximate Scale in Miles

Figure 1-25
LEROI COMPANY SMELTER
4-MILE MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-25

Source: DeLorme 1998.



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UPPER COLUMBIA RIVER ESI
MINES AND MILLS
LEROI COMPANY SMELTER
Stevens County, Washington

0 1 2
Approximate Scale in Miles

Figure 1-26
LEROI COMPANY SMELTER
15-MILE MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-26

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
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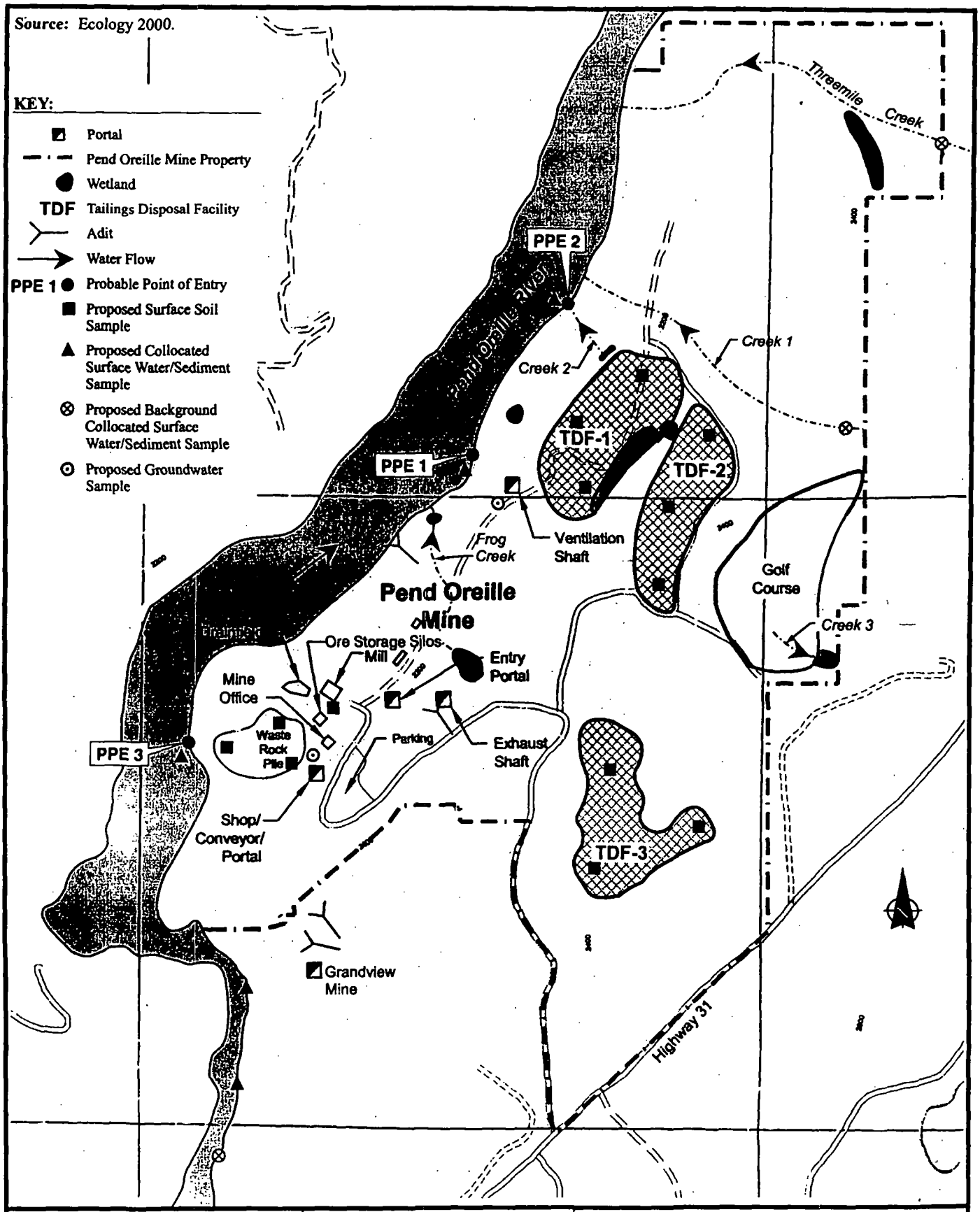

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Seattle, Washington

UPPER COLUMBIA RIVER ESI
MINES AND MILLS
PEND OREILLE MINE
Pend Oreille County, Washington

0 1000 2000
Approximate Scale in Meters

Figure 1-28
PEND OREILLE MINE
PROPOSED SAMPLE LOCATION MAP

Date:	Drawn by:	
06-07-01	AES	10:START-2\01020028\S643\fig 1-28

2. MEASUREMENT/DATA ACQUISITION

2.1 SAMPLING PROCESS DESIGN

During the Upper Columbia River ESI, samples will be collected from locations or features considered to be potential contamination sources, from selected PPE locations, and pathway targets at the Pend Oreille Mine, LeRoi Company Smelter, and Pend Oreille River. The locations or features to be sampled will be determined based on information derived from a review of background information, interviews with mine and mill representatives, visits to the mines and mills including volume of wastes observed, and XRF screening results. Table 2-1 provides information regarding the sampling design and whether the measurement is considered critical or noncritical.

At the time of sampling, site-specific conditions (i.e., topography or visual evidence of contamination) will be evaluated and incorporated, when applicable, into the placement of sampling locations. Other conditions potentially contributing to deviations from the projected sampling locations include new observations or information obtained in the field that warrant an altered sampling approach, difficulty in reaching a desired soil sampling depth caused by adverse soil conditions or obstructions, or limited access to a sampling location. Significant deviations from the planned sampling locations or number of samples to be collected will be discussed with the EPA TM before implementation and will be documented on a Sample Plan Alteration form. A sample form is included in Appendix B. Every attempt will be made to collect representative samples with the equipment being used.

This section describes sample locations (Section 2.1.1); XRF screening (Section 2.1.2); GPS (Section 2.1.3); logistics (Section 2.1.4); cooler return (Section 2.1.5); coordination with federal, state, tribes, local authorities, and other EPA contractors (Section 2.1.6); and the proposed schedule (Section 2.1.7).

2.1.1 Sample Locations

Sample locations will be selected to achieve the objectives discussed in Section 1.3.1. All samples will be submitted for off-site fixed laboratory analysis. Soil, surface water, sediment, and groundwater samples will be analyzed for TAL metals including mercury (Contract Laboratory Program Analytical Services [CLPAS] ILM04.1). Soil samples from mill areas will also be analyzed for chlorinated pesticides/PCBs (CLPAS OLM04.2).

Table 2-2 presents the anticipated number and types of samples, analytical methods, specific requirements for sample container size and type, sample preservation requirements and holding times, and special handling requirements. Table 2-3 summarizes the number of QA/quality control (QC) samples to be submitted according to the method requirements. All surface soil samples will be collected from 0 to 6 inches below ground surface (bgs). A summary of sampling locations and rationale is provided below:

- **Tailings Piles/Ponds.** Mine tailings that were deposited during mining activities may be impacting surface water and sediments in the Upper Columbia River Basin. Two discrete surface soil grab samples at the potential drainage routes and one surface soil grab sample at the native soil interface will be collected from each tailing pile/pond. It is anticipated that 32 of the 80 mines and mills may have at least one tailings pile/pond. Up to three sets of samples will be collected from each tailings pile/pond. It is anticipated that 50% of the 80 sites (excluding 16 sites which have no surface water migration pathway and the Pend Oreille Mine), or 32 sites, will have at least one tailings pile/pond. Up to three tailings piles will be sampled at each property. The surface soil samples will be collected from 0 to 6 inches bgs. If wetlands are noted in the downgradient-area of a tailings pile and are larger than 0.1 mile, a sediment sample will be collected from 0 to 6 inches bgs. Up to three wetland sediment samples will be collected during the project.
- **Waste Rock Piles.** Waste rock that was deposited during mining activities may be impacting surface water and sediments in the Upper Columbia River Basin. One surface soil sample (0 to 6 inches bgs) at the potential drainage route will be collected for every 1,000 cubic yards of waste rock. Up to three discrete grab samples will be collected from each waste rock pile. It is anticipated that 32 of the 80 mines and mills may have at least one waste rock pile. Only one waste rock pile at the adit elevation and one waste rock pile at the mill elevation, as applicable, will be sampled at each mine/mill.
- **Mill Soil Area.** Evidence of former mills, such as a concrete foundation, has been reported at several locations to be visited. If evidence of a mill is present, then three discrete surface soil grab samples will be collected from each structure or former structure. The surface soil samples will be collected from 0 to 6 inches bgs. A maximum of three surface soil samples will be collected at each mine/mill.
- **Mine Adits.** Adit discharge may be impacting surface water and sediments in the Upper Columbia River Basin. If flowing water is present, then one surface water sample will be collected from each of the adits located at each mine. A maximum of 20 adits will be sampled for all the mines.
- **Pend Oreille River Sampling.** Up to five co-located surface water and sediment sample sets along the Pend Oreille River, in addition to 6 additional sediment samples along the Pend Oreille River, are proposed for the Pend Oreille Mine. Refer to Figure 1-28 for proposed sample locations at the Pend Oreille Mine.

- **Tributary Sampling.** Nine sediment samples are proposed to be collected on tributaries flowing into the Pend Oreille River. Refer to the Sample Alteration Form for further details and sample locations.

- **Groundwater Well Sampling.** Two groundwater samples will be collected, depending on accessibility, to determine if groundwater at these well locations is being impacted as a result of a release from the Pend Oreille Mine and the LeRoi Company Smelter.

- **Other Potential Contaminant Source Areas.** Storage and disposal practices at a number of the mines and mills is unclear. If evidence of potential contamination source areas are observed, for instance, stained soil surrounding improperly disposed drums and containers, then up to two surface soil samples will be collected from 0 to 6 inches bgs. A maximum of 20 surface soil samples will be collected in the Upper Columbia River study area.

PPE Samples:

- Contaminants may be impacting surface water and sediments in the Upper Columbia River Basin. If flowing water is present on a mine or mill, then one surface water sample will be collected from each of the PPEs identified. A maximum of 20 surface water PPE samples will be collected in the Upper Columbia River study area.

Background Samples[1]:

- **Background Soil Samples.** Up to 20 representative background surface soil samples having similar characteristics to all the surface soil samples will be collected from unimpacted soil. A visual grain-size analysis using Unified Soil Classification System terms consistent with the American Society of Testing and Materials Test Method D 2488-93$^{\epsilon 1}$ will be recorded to ensure adequate comparability of background samples (Appendix C).

- **Background Surface Water/Sediment Samples.** One representative background co-located surface water and sediment sample set having similar characteristics to the surface water and sediment samples collected from the Pend Oreille River will be collected from a location upgradient from the Pend Oreille Mine. One representative contribution co-located surface water and sediment sample set will be collected from Threemile Creek. One contribution co-located surface water and sediment sample set will be collected from Creek One near the Pend Oreille Mine. Two additional background sediment samples are proposed having similar characteristics to the sediment samples collected along the Pend Oreille River. Refer to the Sample Alteration Form for further details and sample locations.

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[1]The term background is used in the context as it is defined in Hazard Ranking System scoring and not as its common usage as part of a remedial investigation/feasibility study process.

2.1.2 XRF Screening

At the direction of the EPA TM, approximately five soil locations per source will be analyzed in-situ for 120 seconds using NITON Corporation XL-700 Series XRF instruments for mercury, lead, and zinc. National Institute of Standards and Technology-traceable standards will be used for calibration prior to analysis, which will follow the EPA Region 10 START-2 SOP for X-Ray Fluorescence Field Screening, EPA SW-846 Method 6200 (Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment), and the manufacturer's instructions (Appendices D, E, and F). The sum of the concentrations of these elements will be used to determine the three highest total concentrations; soil samples for laboratory analysis will be collected from these locations. Confirmation analysis will be performed at the MEL or a CLP laboratory as designated by the EPA RSCC.

2.1.3 Global Positioning System

GPS units with data loggers will be used to identify the location coordinates of every sample collected, as well as to delineate the boundaries of the potential source areas. GPS coordinates will be provided in the final ESI report as an appendix. GPS units will also be used to identify all mines and mills not sampled, but accessed and documented. The START-2 will obtain differential correction data from a local source prior to the start of the survey in order to improve the survey resolution.

2.1.4 Logistics

Property owners or representatives of the mines and mills located on private property will be identified and consent for access will be requested prior to conducting ESI field work. Mines and mills located on federally-owned lands will be identified and the appropriate federal agency will be informed of the planned visits. Property owners or representatives will be provided advance notification of the planned visit to their mine or mill and provided the opportunity to be present during the visit. The mines and mills are accessible by vehicle and on foot. Sample aliquots collected for fixed laboratory analysis will be delivered to the EPA, Region 10, laboratory or an alternative laboratory as directed by EPA. All samples will be shipped at the end of each week in the field by a commercial airline for express delivery. Sample control and shipping are discussed in Section 2.3.

2.1.5 Cooler Return

For laboratories other than the EPA, Region 10, laboratory, E & E will provide completed airbills accompanied by plastic envelopes with adhesive backs and address labels in the COC bags taped inside of the cooler lids so the laboratory can return the coolers to E & E. The airbills will contain the following notation: "Transportation is for the U.S. Environmental Protection Agency, and the total actual transportation charges paid to the carrier(s) by the consignor or consignee shall be reimbursed by the Government, pursuant to cost reimbursement contract No. 68-S0-01-01." This notation will enable the laboratories to return the sample coolers to E & E's warehouse. The airbills will be marked for second-day economy service and will contain the appropriate TDD number for shipment.

For the EPA, Region 10, laboratory, an arrangement by E & E for cooler return in this manner is not required.

2.1.6 Coordination with Federal, State, Tribes, Local Authorities, and other EPA Contractors

The EPA has directed that the TM will be the point of contact with all other federal, state, tribal, and local officials as well as with the property owners, public, and other contractors employed by the EPA.

The START-2 will keep the TM apprised of field event progress and issues that may affect the schedule or outcome of the Upper Columbia River ESI, discuss any problems encountered, inform the EPA of unusual contacts with the public or media, and obtain guidance from the EPA regarding project activities when required. Additionally, the START-2 will notify the EPA RSCC of changes to the sampling schedule for MEL and/or CLP analyses and provide shipping information regarding every sample shipment within 24 hours of shipment or before noon on Friday for Saturday delivery.

Before initiation of the ESI field activities, the START-2 will notify the property owners of the START-2's field schedule and sampling plan. The EPA TM will maintain regular communication with Tribal representatives regarding field activities and schedule. The EPA TM will be the liaison for all contacts between the START-2 and other contractors before, during, and after this field event.

2.1.7 Schedule

The schedule for implementing the Upper Columbia River ESI is intended to be used as a guide. Adjustments to the implementation dates and the estimated project duration may be necessary to account for variable unforeseen or unavoidable conditions that the field team may encounter. Examples include inclement weather, difficulties in accessing a sampling location, or additional time needed to complete a

task. Significant schedule changes that arise in the field will be discussed with the TM at the earliest possible convenience.

ESI fieldwork is expected to commence on Sunday, June 17, 2001, and will conclude on June 30, 2001. This period comprises one day of mobilization, one day of demobilization, and 12 days to complete field activities. Outdoor work will be conducted during daylight hours only.

2.2 SAMPLING METHOD REQUIREMENTS

This section describes the sampling methods (Section 2.2.1), sampling equipment decontamination (Section 2.2.2), investigation-derived wastes (IDWs; Section 2.2.3), and SOPs (Section 2.2.4).

2.2.1 Sampling Methods

The START-2 PM and EPA TM will be responsible for ensuring that appropriate sample collection procedures are followed and will take appropriate actions to correct any deficiencies. All samples will be maintained under COC and will be stored and shipped in iced coolers. The sampling methods for each medium are as follows:

- **Tailings Piles/Ponds, Waste Rock Piles, Mill Soil Area, Other Potential Contaminant Source Areas.** Surface soil samples (0 to 6 inches bgs) will be collected using a dedicated plastic or Teflon scoop for TAL sample aliquots and stainless steel spoons for pesticide/PCB sample aliquots. Collected material will be placed in a dedicated plastic or Teflon bowl (stainless steel bowls for pesticide/PCB aliquots), thoroughly homogenized, and placed into prelabeled sample containers. For crushed waste rock samples, an attempt to separate the coarse material from the desired fine material will be made by placing the sample into a plastic bag and agitating it. Coarse materials will be discarded and the fines will be throughly homogenized and placed into a prelabeled sample container. SOP guidelines for subsurface soil sampling are provided in Appendix A.

- **Adit Water and PPE Sampling.** Surface water samples will be collected either by hand-dipping the sample container into the water at well-mixed locations within the river and spring, if possible, or by creating a funnel with a dedicated 1-liter polyethylene sample bottle with the bottom of the bottle removed. If sampling requires the sampler to enter the water, the sampler will enter downstream of the sample location. An aliquot sample will be collected separately and tested immediately for pH using pH paper. The preserved aliquot will be tested for pH following preservation to ensure the appropriate pH level has been achieved.

- **Surface Water Sampling.** Surface water samples will be collected either by hand-dipping the sample container into the water at well-mixed locations within the river, if possible, or be creating a funnel with a dedicated 1-liter polyethylene sample bottle with the bottom of the bottle removed. Surface water samples will be collected prior to the collection of sediment samples. If sampling required the sampler to enter the water, the sample will be collected from a small fiberglass hull boat. An aliquot sample will be collected separately and tested immediately for pH using pH paper. The preserved aliquot will be tested for pH following preservation to ensure the appropriate pH level has been achieved.

- **Sediment Sampling.** Sediment samples will be collected from 0 to 6 inches below the surface water/sediment interface using dedicated plastic or Teflon scoops after the corresponding co-located surface water has been collected. All sediment samples will be homogenized thoroughly in dedicated plastic or Teflon bowls, and excess water will be decanted before the samples are placed into prelabeled sample containers. Samples will be collected starting at the most downstream location and continuing upstream to reduce the potential of cross-contamination. Guidelines for sediment samples are contained in the SOP found in Appendix A.

- **Groundwater Well Sampling.** Groundwater samples collected from domestic wells will be collected using dedicated, disposable Teflon bailers. Groundwater retrieved in the bailers will be poured directly into prelabeled sample containers. Procedures for well purging and sampling are provided in the groundwater well sampling SOP in Appendix A.

2.2.2 Sampling Equipment Decontamination

To the greatest extent possible, disposable and/or dedicated personal protective and sampling equipment will be used to avoid cross-contamination. When required, decontamination will be conducted in a central location, upwind and away from suspected contaminant sources. The following procedures (as listed in Appendix A) are to be used for all sampling equipment used to collect routine samples undergoing trace organic or inorganic constituent analyses:

1. Clean with tap water and nonphosphate detergent, using a brush if necessary to remove particulate matter and surface films.
2. Rinse thoroughly with tap water.
3. Rinse with 10 percent nitric acid.
4. Rinse thoroughly with type II reagent-grade water.
5. Rinse with laboratory-grade hexane.
6. Air dry the equipment completely.
7. Rinse again with type II reagent-grade water.

8. Remove the equipment from the decontamination area and cover with plastic. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.

2.2.3 Investigation-Derived Wastes

The START-2 field team members will make every effort to minimize the generation of IDWs throughout the field event. All wastewater will be contained in 5-gallon drums, labeled, and disposed of at an approved facility based on ESI analytical results from the matrix samples. No wastewater is anticipated during the fieldwork. Disposable personal protective clothing and sampling equipment generated during field activities will be rendered unusable by tearing (when appropriate), bagged in opaque plastic garbage bags, and disposed of at an approved facility based on analytical results from matrix samples.

2.2.4 Standard Operating Procedures

The START-2 will utilize the following SOPs (Appendix A) while performing field activities:

- Field Activity Logbooks,
- Surface Water Sampling,
- Soil Sampling,
- Sediment Sampling,
- Groundwater Well Sampling,
- Sample Packaging, and
- Sampling Equipment Decontamination.

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

This section describes sample identification and COC procedures that will be used for the Upper Columbia River ESI mine and mill field activities. The purpose of these procedures is to ensure that the quality of samples is maintained during collection, transportation, storage, and analysis. All COC requirements comply with E & E's SOPs for sample handling. All sample control and COC procedures will follow the EPA (1991) *User's Guide to the Contract Laboratory Program.*

Examples of sample documents used for custody purposes are provided in Appendix B and include the following:

- Sample identification numbers,
- Sample tags or labels,
- Custody seals,
- COC and traffic report records,
- Sample collection forms,
- Analytical request forms, and
- Analytical records.

During the field effort, the project manager or delegate will be responsible for maintaining an inventory of these sample documents. This inventory will be recorded in a cross-referenced matrix of the following:

- Sample location,
- Sample identification number,
- Analyses requested and request form number(s),
- COC record numbers,
- Bottle lot numbers, and
- Airbill numbers.

Brief descriptions of the major sample identification and documentation records and forms are provided below.

2.3.1 Sample Identification

All samples will be identified using the sample numbers assigned by the EPA RSCC. Each sample label will be affixed to the jar and covered with clear tape. A sample tracking record will be kept as each sample is collected. The following will be recorded: location, matrix, sample number, observations, and depth. In addition to the EPA-assigned sample number, samples will be tracked with a sample code system designed to allow easy reference to the sample's origin and type. The sample code key will not be provided to the laboratory. Table 2-4 summarizes the sample tracking codes and locations.

2.3.1.1 Sample Tags and Labels

Sample tags attached to or fixed around the sample container will be used to identify all samples collected in the field. The sample tags will be placed on bottles so as not to obscure any QA/QC lot numbers on the bottles, and sample information will be printed legibly. Field identification will be sufficient to enable cross-reference with the project logbook. For COC purposes, all QA/QC samples will be subject to the same custodial procedures and documentation as project samples.

To minimize handling of sample containers, labels will be completed before sample collection to the extent possible. In the field, the labels will be filled out completely using waterproof ink, then attached firmly to the sample containers and protected with clear tape. The sample label will provide the following information:

- EPA Sample number,
- Sample location number,
- Date and time of collection,
- Analysis required,
- Initials of sampler, and
- pH and preservation (when applicable).

2.3.1.2 Custody Seals

Custody seals are preprinted gel-type seals, designed to break into small pieces if disturbed. Sample shipping containers (e.g., coolers, drums, and cardboard boxes, as appropriate) will be sealed in as many places as necessary to ensure security. Seals will be signed and dated before use. Clear tape will be placed over the middle of the seals to ensure that they are not broken accidentally during shipment. Upon receipt of shipment at the laboratory, the custodian will check (and certify by completing the package receipt log) that seals on shipping containers are intact.

2.3.1.3 Chain-of-Custody Records and Traffic Reports

For samples to be analyzed at a CLP laboratory, the COC record and analytical traffic report forms will be completed as described in *User's Guide to Contract Laboratory Program* (EPA 1991). The EPA's FORMS II Lite software will be used to electronically enter information for the COC and traffic report forms. The COC record and analytical traffic reports will be completed fully at least in

duplicate by the field technician designated by the project manager as responsible for sample shipment to the appropriate laboratory. Information specified on the COC record will contain the same level of detail found in the project logbook, except that the on-site measurement data will not be recorded. The custody record will include the following information:

- Name and company or organization of person collecting the samples;
- Date samples were collected;
- Type of sampling conducted (composite or grab);
- Sample number (using those assigned by the EPA RSCC);
- Location of sampling station (using the sample code system described in Table 2-4);
- Number and type of containers shipped;
- Analysis requested; and
- Signature of the person relinquishing the samples to the transporter, with the date and time of transfer noted and the signature of the designated sample custodian at the receiving facility.

If samples require rapid laboratory turnaround, the person completing the COC record will note these or similar constraints in the remarks section of the custody record.

The relinquishing individual will record all shipping data (e.g., airbill number, organization, time, and date) on the original custody record, which will be transported with the samples to the laboratory and retained in the laboratory's file. Original and duplicate custody records, together with the airbill or delivery note, constitute a complete custody record. It is the project manager's responsibility to ensure that all records are consistent and that they become part of the permanent job file.

2.3.1.4 Field Logbooks and Data Forms

Field logbooks (or daily logs) and data forms are necessary to document daily activities and observations. Documentation will be sufficient to enable participants to reconstruct events that occurred during the project accurately and objectively at a later time. All daily logs will be kept in a bound notebook containing numbered pages. All entries will be made in waterproof ink, dated, and signed. No pages will be removed for any reason.

Minimum logbook content requirements are described in the E & E SOP entitled *Preparation of Field Activity—Logbooks* (Appendix A). If corrections are necessary, they will be made by drawing a single line through the original entry (so that the original entry is legible) and writing the corrected entry

alongside. The correction will be initialed and dated. Corrected errors may require a footnote explaining the correction.

2.3.1.5 Photographs

Photographs will be taken as directed by the team leader. Documentation of a photograph is crucial to its validity as a representation of an existing situation. The following information concerning photographs will be noted in the project or task log:

- Date, time, and location where photograph was taken,
- Photographer,
- Weather conditions,
- Description of photograph taken,
- Sequential number of the photograph and the film roll number,
- Camera lens system used, and
- Direction.

2.3.2 Custody Procedures

The primary objective of COC procedures is to provide an accurate written or computerized record that can be used to trace the possession and handling of a sample from collection to completion of all required analyses. A sample is in custody when it is:

- In someone's physical possession,
- In someone's view,
- Locked up, or
- Kept in a secured area that is restricted to authorized personnel.

2.3.2.1 Field Custody Procedures

The following guidance will be used to ensure proper control of samples while in the field:

- As few people as possible will handle samples;
- Coolers or boxes containing cleaned bottles will be sealed with a custody tape seal during transport to the field or while in storage before use. Sample bottles from unsealed coolers or boxes, or bottles that appear to have been tampered with, will not be used;

- The sample collector will be responsible for the care and custody of collected samples until they are transferred to another person or dispatched properly under COC rules;
- The sample collector will record sample data in the field logbook; and
- The property team leader will determine whether proper custody procedures were followed during the fieldwork and will decide whether additional samples are required.

When transferring custody (i.e., releasing samples to a shipping agent), the following will apply:

- The coolers in which the samples are packed will be sealed and accompanied by the COC records. When transferring samples, the individuals relinquishing and receiving them must sign, date, and note the time on the COC record. This record will document sample custody transfer;
- Samples will be dispatched to the laboratory for analysis with separate COC records accompanying each shipment. Shipping containers will be sealed with custody seals for shipment to the laboratory. The COC records will be signed by the relinquishing individual, and the method of shipment, name of courier, and other pertinent information will be entered in the COC record before placement in the shipping container;
- All shipments will be accompanied by COC records identifying their contents. The original custody records kept in a sealed Ziploc bag and taped inside the lid of the cooler will accompany each cooler shipment. The other copies will be distributed appropriately to the property team leader and project manager; and
- If sent by common carrier, a bill of lading will be used. Freight bills and bills of lading will be retained as part of the permanent documentation.

2.3.2.2 Laboratory Custody Procedures

A designated sample custodian at the laboratory will accept custody of the shipped samples from the carrier and enter preliminary information about the package into a package or sample receipt log, including the initials of the person delivering the package and the status of the custody seals on the coolers (i.e., broken versus unbroken). The custodian responsible for sample log-in will follow the laboratory's SOP for opening the package, checking the contents, and verifying that the information on the COC agrees with samples received. The CLP or MEL will follow internal COC procedures as stated in the laboratory QA Manual.

The laboratory will check the temperature blank inside the cooler, and document it in the sample log-in form. Should the temperature be greater than what is required by the Statement of Work or the method, the sample custodian will inform the region and proceed to follow the course of actions stipulated in the SOW or specified by the regional QA Officer.

2.4 ANALYTICAL METHOD REQUIREMENTS

This section discusses the analytical strategy (Section 2.4.1) and analytical methods (Section 2.4.2).

2.4.1 Analytical Strategy

Analysis of samples collected during the ESI will be performed by several possible means. The EPA, Region 10, laboratory (or alternative laboratory designated by the EPA) will perform all requested analyses.

The analyses to be applied to samples sent to these laboratories are listed in Table 2-2. These analyses were selected based on the probable hazardous substances used or potentially released to the environment, given the known or suspected mine or mill usage.

2.4.2 Analytical Methods

Samples designated for off-site analytical laboratory analyses will be submitted to the EPA, Region 10, laboratory located in Manchester, Washington, or an alternative laboratory designated by the EPA. EPA and/or CLP laboratory analyses will take place under a three-week turnaround time period, with validation by the EPA QA office for these analyses taking place under the standard three-week turnaround time period. Hardcopy results from the EPA and/or CLP laboratories will be delivered to the EPA upon completion. Tables 2-1 and 2-2 provide a detailed list of sampling and analytical requirements and target DQOs for this project. Table 2-3 summarizes laboratory instrumentation and methods to be used for the ESI.

2.5 QUALITY CONTROL REQUIREMENTS

QC checks for sample collection will be accomplished by a combination of COC protocols and laboratory QA as prescribed in the sampling or analytical methods. No QC samples (i.e. double blind performance evaluation samples) are planned for this activity outside normal laboratory QC criteria outlined in the analytical methods. These QC samples include rinsate and method blanks; temperature blanks; matrix spikes; and calibration check samples. Results from these samples will be compared to the QC requirements listed in Section 4.1.2. All of the analyses that will be performed for this project will produce definitive data. DQI targets for this project are specified in Section 1.4 and are summarized in Table 2-2 of this SQAP. Bias on estimated flagged data shall be determined by the validation process.

The laboratories' DQOs for representativeness are set at 90 percent. Precision and accuracy requirements are outlined in Table 2-3.

One temperature blank consisting of a 40-milliliter glass vial of distilled water will be included in each cooler shipped to the analytical laboratories. Temperature blanks allow the laboratories to obtain a representative measurement of the temperature of samples enclosed in a cooler without disturbing the actual samples. The field team will package and label the temperature blank like a regular water sample, however the analytical laboratory will only measure the temperature of the blank. The temperature blank will not be analyzed for hazardous substances, will not be given a sample number, and will not be listed on the COC. The temperature blank will be clearly labeled: USEPA COOLER TEMPERATURE INDICATOR.

2.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

The field equipment used during this project includes NITON XRF instruments, GPS units, and Jerome 431-X Mercury Vapor Analyzers. The Mercury Vapor Analyzers will be used to continuously monitor the breathing zone during sample collection activities for health and safety purposes only. Testing, inspection, and maintenance of the all field equipment will be performed in accordance with the manufacturer's recommendations (Appendices F and G). Spare parts for the field equipment will be available from the manufacturer or regional representative generally within 24 hours.

All field instruments and equipment used for field laboratory analysis will be serviced and maintained only by qualified personnel. All instruments will be maintained by senior staff and/or electronics technicians. All repairs, adjustments, and calibrations will be documented in an appropriate logbook or data sheet that will be kept on file. The instrument maintenance logbooks will clearly document the date, the description of the problems, the corrective action taken, the result, and who performed the work.

All equipment used by E & E in the field is subject to standard preventive maintenance schedules established by corporate equipment protocols. When in use, equipment will be inspected at least twice daily, once before startup in the morning and again at the end of the work shift before overnight storage or return to the charging rack. Regular maintenance, such as cleaning of lenses, replacement of in-line filters, and removal of accumulated dust, is to be conducted according to manufacturers' recommendations and in the field as needed, whichever is appropriate. All performed preventive maintenance will be entered in the individual equipment's logbook and in the field logbook.

In addition to preventive maintenance procedures, calibration checks will be performed at least once daily before equipment use and recorded in the respective logbooks. Additional calibration checks will be performed as required. All logbooks will become part of either the permanent project file or the permanent equipment file.

2.7 INSTRUMENT CALIBRATION AND FREQUENCY

All instruments and equipment used during fixed laboratory sample analyses will be operated, calibrated, and maintained according to the manufacturers' guidelines and recommendations, as well as criteria set forth in the applicable analytical methodology references and/or in accordance with the laboratory's QA Manual and SOPs.

For field instrumentation (NITON XRF, GPS, and the Jerome 431-X Mercury Vapor Analyzers), the calibration will be performed in accordance with the manufacturers' recommendations.

2.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

This information is covered by the SOPs, the START-2 QAPP, and the START-2 QMP (E & E 2001b). Standards contained in these documents will be used to ensure the validity of data generated by E & E for this project. Sample jars are precleaned by the manufacturer; certification documenting this is enclosed with each box of jars. The START-2 will include this documentation as part of the project file. Nondedicated sampling equipment is demonstrated to be uncontaminated by the use of rinsate blanks.

2.9 DATA ACQUISITION REQUIREMENTS (NONDIRECT MEASUREMENTS)

No data from other sources will be used.

2.10 DATA MANAGEMENT

This document is meant to be combined with information presented in E & E's QAPP and QMP for the START-2, Region 10. Copies of the START-2 QAPP and QMP are available in E & E's Seattle, Washington, office. Standards contained in these documents will be used to ensure the validity of data generated by E & E for this project. The MEL and/or CLP laboratory will provide CLP-equivalent deliverables to the EPA validation chemist within three weeks of sample receipt. Data validation will be performed within three weeks of receipt of the data package and will consist of the deliverables as

described in Section 4.1.2. Data tracking, storage, and retrieval are tracked on the TDD "blue sheet," which records where the paper and electronic data are located. All paper data are stored in locked file cabinets; access to these files is restricted to key START-2 personnel. Electronic data are archived by TDD.

Table 2-1

SAMPLE INFORMATION SUMMARY
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION/MINES AND MILLS
NORTHEAST WASHINGTON

Project Sampling Location	Parameter/Limits	Design Rationale	Sampling Design Assumptions	Sample Selection Procedures[a]	Measurement Classification	Non-Standard Method Validation[b]
Five soil locations per source	Lead, mercury, zinc/ Instrument detection limit	Determine three highest source concentrations.	Residual contamination may exist from past operations. Federal listed endangered and threatened species may be impacted.	In situ analysis at potentially contaminated areas.	Non critical	EPA Method 6200 and START-2 XRF guidelines.
Tailings Piles/Ponds	TAL metals/CRDL	Determine the presence of hazardous substances.	Mine tailings contain hazardous substances which are potentially discharging into the Upper Columbia River Basin. Federal listed endangered and threatened species may be impacted.	Collected from potentially contaminated areas.	Critical	NA
Waste Rock Piles	TAL metals/CRDL	Determine the presence of hazardous substances.	Waste rock may contain hazardous substances which are potentially affecting Federal listed endangered and threatened species.	Collected from potentially contaminated areas.	Critical	NA
Mill Soil	TAL metals/CRDL; Pesticides/PCBs/CRQL	Determine the presence of hazardous substances.	Residual contamination may exist from past operations. Federal listed endangered and threatened pecies may be impacted.	Collected from potentially contaminated areas.	Critical	NA
Other Potential Contaminant Source Areas	TAL metals/CRDL	Determine the presence of hazardous substances.	Residual contamination may exist from past operations. Federal listed endangered and threatened species may be impacted.	Collected from potentially contaminated areas.	Critical	NA
Mine Adits	TAL metals/CRDL	Determine the presence of hazardous substances.	Mine adit water may contain hazardous substances which are potentially affecting Federal listed endangered and threatened species.	Collected from potentially contaminated areas.	Critical	NA
Probable Point of Entry Samples	TAL metals/CRDL	Determine the presence of hazardous substances.	Hazardous substances may have migrated into the Upper Columbia River Basin.	Collected from potentially contaminated areas.	Critical	NA
Surface Water	TAL metals/CRDL	Determine the presence of hazardous substances.	Hazardous substances may have migrated into the Pend Oreille River. On-site workers and Federal listed endangered and threatened species may be impacted.	Collected from potentially contaminated areas.	Critical	NA
Sediment	TAL metals/CRDL	Determine the presence of hazardous substances.	Hazardous substances may have migrated into the Pend Oreille River. On-site workers and Federal listed endangered and threatened species may be impacted.	Collected from potentially contaminated areas.	Critical	NA

Table 2-1

SAMPLE INFORMATION SUMMARY
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION/MINES AND MILLS
NORTHEAST WASHINGTON

Project Sampling Location	Parameter/Limits	Design Rationale	Sampling Design Assumptions	Sample Selection Procedures[a]	Measurement Classification	Non-Standard Method Validation[b]
Groundwater	TAL metals/CRDL	Determine the presence of hazardous substances.	A well is located on-site near a waste rock pile which contains hazardous substances which are potentially contaminating the groundwater. On-property workers and Federal listed endangered and threatened species may be impacted.	Collected from potentially contaminated areas.	Critical	NA
Background-All Locations	TAL metals/CRDL; Pesticides/PCBs/CRQL	Determine background concentrations.	Locations are native areas where no contaminants were deposited.	Collected from potentially contaminated areas.	Critical	NA

[a] Sample locations will be determined from on-site observations and historical information.
[b] Data will be validated based on the laboratory statement of work QC limits and laboratory and method QC limits.

Key:

CRDL	= Contract-required detection limit.	PCBs	= Polychlorinated biphenyls
Critical	= Required to achieve project objectives or limits on decision errors.	Pesticides	= Chlorinated pesticides
CRQL	= Contract-required quantitation limit.	QC	= Quality control.
NA	= Not Applicable.	TAL	= Target Analyte List.
Non Critical	= For informational purposes only.		

Table 2-2

SAMPLE ANALYSES SUMMARY
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION/MINES AND MILLS
NORTHEAST WASHINGTON

Location	Matrix[a]	Analytical Parameters and Method	Sample Preservation	Technical Holding Time[b]	Sample Container(s)
Tailings Piles/Ponds	Surface Soil (Up to 5 in situ XRF analyses per source and up to 3 samples per pile/pond)	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C	180 days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
Waste Rock Piles	Surface Soil (Up to 5 in situ XRF analyses per source and up to 3 samples per pile)	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C	180 days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
Mill Soil	Surface Soil (Up to 5 in situ XRF analyses per source and up to 3 samples per mill building for a maximum of 24 samples)	Lead, mercury, and zinc EPA Method 6200	Not applicable	Not applicable	In situ
		Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C	180 days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Pesticides/PCBs (CLPAS) OLM04.2	Cool to 4°C ± 2°C	14 days from collection to extraction; 40 days from extraction to analysis	One 8-oz wide-mouth glass jar with Teflon-lined lid
Other Potential Contaminant Source Areas	Surface Soil (Up to 20 samples)	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C	180 days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
Adit Water	Surface Water (Up to 20 samples)	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C; HNO_3 to pH ≤ 2	180 days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
Probable Point of Entry Samples	Surface Water (Up to 20 samples)	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C; HNO_3 to pH ≤ 2	180 days from collection (28 days for mercury)	One 1-L polyethylene bottle with polyethylene-lined lid
Pend Oreille River and Tributaries	Surface Water (5 samples)	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C; HNO_3 to pH ≤ 2	180 days from collection (28 days for mercury)	One 1-L polyethylene bottle with polyethylene-lined lid
	Sediment (20 samples)	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C	180 days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Pesticides/PCBs (CLPAS) OLM04.2	Cool to 4°C ± 2°C	14 days from collection to extraction; 40 days from extraction to analysis	One 8-oz wide-mouth glass jar with Teflon-lined lid

Table 2-2

SAMPLE ANALYSES SUMMARY
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION/MINES AND MILLS
NORTHEAST WASHINGTON

Location	Matrix[a]	Analytical Parameters and Method	Sample Preservation	Technical Holding Time[b]	Sample Container(s)
Groundwater	1 from on-site well at Pend Oreille Mine; 1 from well adjacent to LeRoi Company Smelter	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C; HNO_3 to pH ≤ 2	180 days from collection (28 days for mercury)	One 1-L polyethylene bottle with polyethylene-lined lid
Background	Surface Soil (Up to 20 samples)	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C	180 days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Pesticides/PCBs (CLPAS) OLM04.2	Cool to 4°C ± 2°C	14 days from collection to extraction; 40 days from extraction to analysis	One 8-oz wide-mouth glass jar with Teflon-lined lid
Background	Surface Water (3 samples)	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C; HNO_3 to pH ≤ 2	180 days from collection (28 days for mercury)	One 1-L polyethylene bottle with polyethylene-lined lid
Background	Sediment (5 samples)	Target Analyte List metals (CLPAS) ILM04.1	Cool to 4°C ± 2°C	180 days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid

[a] The number of samples presented is an estimate; the actual number of samples to be collected will be determined in the field.
[b] Technical holding times have been established only for water matrices. Water technical holding times were applied to sediment and soil samples where applicable; in some cases, recommended sediment/soil holding times are listed.

Key:

CLPAS = Contract Laboratory Program Analytical Services.
EPA = United States Environmental Protection Agency.
HNO_3 = Nitric acid.
L = Liter.
XRF = X-Ray Fluroescence.
oz = Ounce.
PCBs = Polychlorinated biphenyls.
Pesticides = Chlorinated pesticides
°C = Degrees Celsius.

Table 2-3

QA/QC ANALYTICAL SUMMARY AND FIXED LABORATORY ANALYTICAL METHODS SAMPLE INFORMATION SUMMARY UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION/MINES AND MILLS NORTHEAST WASHINGTON

Laboratory	Matrix	Parameters (Method)	Method Description/ Detection Limits	Total Field Samples[a]/ Containers	QA/QC Sample Summary Analyses/Containers				Total Field and QA/QC Analyses/ Containers[d]
					Trip Blanks	Rinsate Blanks[b]	Organic MS/MSD[c]	Inorganic MS/Dup[c]	
Field XRF	Soil	Lead, mercury, zinc EPA Method 6200	XRF/ Instrument Detection Limit	TBD / NA	NA	NA	NA	NA	TBD / NA
EPA, Region 10, or CLP Laboratory	Soil/ Sediment	TAL metals (CLPAS) ILM04.1	AA and ICP/ CRDL	572 / 572	NA	0 / 0	NA	29 / 0	601 / 572
		Pesticides/PCBs (CLPAS) OLM04.2	GCS and ECD/ CRQL	69 / 69	NA	0 / 0	4 / 0	0 / 0	73 / 69
	Water	TAL metals (CLPAS) ILM04.1	AA and ICP/ CRDL	50 / 50	NA	NA	NA	3 / 6	53 / 56

[a] The total number of field samples is estimated.

[b] The total number of rinsate blanks could vary depending on the total number of sample shipments. The sample numbers are based on one rinsate per 20 samples per nondedicated sampling device. Note that rinsate blanks consist of water aliquots for both soil and water field samples.

[c] No extra volume is required for soil/sediment samples; for water samples, triple volume is required for organic analyses and double volume is required for inorganic analyses. Sample numbers are based on one MS/MSD per 20 samples per matrix.

[d] Total analyses and containers includes field and QA/QC aliquots to be submitted for fixed laboratory analysis. Note that rinsate blanks consist of water aliquots for both soil and water field samples.

Key:

AA = Atomic absorption furnace technique.
CLP = Contract Laboratory Program.
CLPAS = Contract Laboratory Program Analytical Services.
CRDL = Contract-required detection limit.
CRQL = Contract-required quantitation limit.
ECD = Electron capture detection.
EPA = United States Environmental Protection Agency.
GCS = Gas chromatographic separation.
ICP = Inductively coupled argon plasma.
MS/DUP = Matrix spike/duplicate.
MS/MSD = Matrix spike/matrix spike duplicate.
NA = Not applicable.
QA/QC = Quality assurance/quality control.
TAL = Target Analyte List.
TBD = To be determined.
XRF = X-Ray Fluroescence.

Table 2-4

SAMPLE CODING
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION/MINES AND MILLS
NORTHEAST WASHINGTON

Digits	Descriptions	Code Example	
1,2	Area (mine or mill)	AC	A & C
		AD	Admiral Consolidated
		AN	Anaconda
		AL	Antelope
		BB	Blue Bucket
		BE	Bechtol
		BM	Bella May
		BL	Bullion
		BR	Black Rock
		BU	Burrus
		CA	Calhoun
		CF	Cliff
		CK	Copper King
		CL	Clara
		CO	Coyote
		DC	Deep Creek
		DR	Diamond R
		EP	Electric Point
		EV	Evergreen
		FA	Farmer
		FS	Frisco Standard
		GB	Gold Bar
		GL	Gladstone
		GW	Great Western
		HA	Hanley
		HM	Homestake
		HO	Hoage
		HT	Hope & Twin Cabins
		HU	Hubbard
		IR	Iroquois
		JA	Jackson
		JO	Josephine
		KE	Keystone
		KI	King Tut
		LA	Lakeview

Table 2-4

SAMPLE CODING
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION/MINES AND MILLS
NORTHEAST WASHINGTON

Digits	Descriptions	Code Example	
1,2	Project Area (mine or mill)	LC	Last Chance
		LE	Lehigh No. 1
		LF	Lucky Four
		LH	Lead Hill
		LK	Lead King
		LN	Lehigh No. 2
		LO	Lottie
		LQ	Lead Queen
		LR	Lone Star
		LS	Lucky Strike
		LT	Lead Trust
		LU	Lucile
		MA	Magma
		ME	Metaline
		MI	Minorca
		MK	Maki
		ML	Melrose
		MO	Morning
		MY	Myeerah
		MV	Mountain View
		NL	New Leadville
		NS	Northport Smelter (LeRoi Company Smelter)
		OR	Oriole
		PO	Pend Oreille
		RD	Red Top (Stevens County)
		RJ	RJ
		RO	Roosevelt
		RT	Red Top (Pend Oreille County)
		SA	Scandia
		SC	Scaman
		SE	Sterling
		SL	Silver Crown
		SR	Sterrett
		ST	Silver Trail

Table 2-4

SAMPLE CODING
UPPER COLUMBIA RIVER EXPANDED SITE INSPECTION/MINES AND MILLS
NORTHEAST WASHINGTON

Digits	Descriptions	Code Example	
1,2	Project Area (mine or mill)	SU	Sullivan
		SZ	Sierra Zinc
		TH	Thompson
		TY	Tyee
		US	Uncle Sam
		UN	United Treasure
		VS	Van Stone
		WA	Washington
		WC	West Contact
		YH	Yellowhead
		ZC	Z Canyon
3,4	Source Code	WP	(Waste Pile)
		MS	(Mill Soil)
		PP	(Probable point of entry)
		TP	(Tailings Pile/Pond)
		BK	(Background)
5,6	Sample Number	01	01
7,8	Matrix Code	GW	(Groundwater)
		SD	(Sediment)
		SS	(Surface Soil)
		SW	(Surface Water)

3. ASSESSMENT/OVERSIGHT

3.1 ASSESSMENTS AND RESPONSE ACTIONS

The EPA QA officer or designee may conduct an audit of the field activities for this project. The auditor will have the authority to issue a stop-work order upon finding a significant condition that would adversely affect the quality and usability of the data. The EPA TM will have the responsibility for initiating and implementing response actions associated with findings identified during the project audit. The actions taken also may involve the EPA project officer, contracting officer, and/or QA officer. Once the response actions are implemented, the EPA QA officer or designee may perform a follow-up audit to verify and document that the response actions were implemented effectively. In-house audits performed by the START-2 may be conducted in accordance with the E & E (2001b) START-2 QMP. No audits are planned for the Upper Columbia River mines and mills field activities.

3.2 REPORTS TO MANAGEMENT

The START-2 PM will debrief the EPA TM daily. Laboratory deliverables will be as specified in the CLP Organic and Inorganic Statements of Work (OLM04.2 and ILM04.1, respectively) for CLP and/or MEL data. No use of commercial laboratories is planned for this project. Once the resulting data are obtained, the START-2 PM will prepare owner notification tables to be submitted to the EPA TM. Mine- or mill-specific sample location maps may be requested by the EPA TM. A presentation of analytical results in tabular format and area-wide sample location maps may be requested by the EPA TM. Interpretation of the data will be included in the ESI report to be prepared by the E & E START-2 PM. The ESI report will include a summary and interpretation of analytical data including sediment and surface water sample results generated by Weston and data from the Phase 1 and Phase 2 mines/mills field events conducted by E & E. Data validation (memoranda including complete annotation of all results) will be provided by EPA to E & E both in hard copy and electronically for all analytical data generated for this project. E & E START-2 anticipates that "camera-ready" hard copy and electronic text, tables, and figures generated by Weston as part of the Upper Columbia River ESI sediment and surface water sampling are fully compatible with the E & E START-2 versions (see Section 1.6). At the direction of the EPA TM, these text, tables, and figures will be provided in a timely manner by Weston to E & E for inclusion in the ESI report. E & E will prepare a separate report for the Cleveland Mine under

TDD 01-01-0035. A separate report for the Pend Oreille Mine may be requested by the EPA TM, as determined necessary.

The START-2 corrective action program is addressed in Section 3 of the QMP. Corrective actions will be conducted in accordance with these QMP specifications.

4. DATA VALIDATION AND USABILITY

4.1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

The validation review of data packages will include an evaluation of the information provided on the analytical data sheets and required support documentation for all sample analyses; the supporting sample collection documentation, including COC; and field instrument calibration, results, and/or performance check documentation (if required by the method). The QA review also will examine adherence to the procedures as described in the cited SOPs and the specified analytical methods in the SQAP.

4.1.1 Data Reduction

Data reduction includes all processes that change the numerical value of the raw data. Field screening data will be reported without reduction. All fixed-laboratory data reduction will be performed in accordance with the appropriate methodology and will be presented as sample results.

4.1.2 Data Validation

Field screening data will be examined for adherence to applicable SOPs and will not be validated. Analytical data generated through the CLP contract will be validated by the Region 10 QA Office or its designee. Data generated by MEL will be validated by MEL or its designee. Both MEL and CLP data validations will be performed on a regular three-week turn around time which starts upon receipt of the complete analytical data package from the laboratory. All of the data validations will be performed in accordance with the QA/QC requirements specified in the SQAP, the technical specifications of the analytical methods and the following documents:

- *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (EPA 1994a); and
- *Contract Laboratory Program National Functional Guidelines for Organic Data Review* (EPA 1999).

Validation deliverables will include a QA memo discussing QA conformance and deviations issues which may have affected the quality of the data. Data usability, bases of application of qualifiers

and percentage of qualified data will also be discussed in the QA memoranda. Forms I (analysis Data Sheet) with the applied validation qualifiers and bias determination for estimated-qualified values will also be a part of the validation deliverables. The following qualifiers shall be used in data validation:

- U = The compound was analyzed for, but not detected.
- UJ = The compound was analyzed for, but was not detected; the associated quantitation limit is an estimate because quality control criteria were not met.
- J = The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.
- NJ = The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- R = Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.
- B = Detected concentration is below the method reporting limit/contract-required detection limit, but is above the instrument detection limit;
- H = High bias
- K = Unknown bias
- L = Low bias
- Q = Detected concentration is below the method reporting limit/contract-required quantitation limit, but is above the method detection limit.

In the absence of specific guidance, method-, laboratory-, and SQAP-specific criteria will be used for validation.

4.1.3 Data Assessment Procedures

Following data validation and reporting, all project-generated and -compiled data and information will be reconciled with the objectives specified in Section 1.3.1 to assess the overall success of Upper Columbia River ESI mines and mills activities. This data assessment, including points of achievement and any departure from project-specific objectives, will be discussed in the QA section of the Upper Columbia River ESI mines and mills Phase I report.

4.2 DATA VERIFICATION

Data validation deliverables will be submitted to E & E for verification. Using the QA memo, the Forms I with validation qualifiers and the electronic data deliverables (EDDs) from the laboratories, E & E will ensure that consistency exists among the three documents. E & E will ensure that the correct validation and bias qualifiers are applied to the affected values on the Forms I and EDDs as specified on the QA memo submitted by the validator.

4.3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

The data quality indicators' target for this project is discussed in Section 1.4 of this SQAP. The data validation process will be used as a tool to determine if these targets were met. Also, using the compiled data, E & E and the EPA TM will determine the variability and soundness of the data and the data gaps that will need to be filled to meet the objectives of the project. Interpretation of the data will be included in the ESI report to be prepared by E & E START-2 PM. The ESI report will include a summary and interpretation of analytical data including sediment and surface water sample results generated by Weston and data from the Phase 1 and 2 mines/mills field events conducted by E & E.

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APPENDIX A

STANDARD OPERATING PROCEDURES

TITLE:	FIELD ACTIVITY LOGBOOKS
CATEGORY:	DOC 2.1
REVISED:	April 1998

STANDARD OPERATING PROCEDURE

FIELD ACTIVITY LOGBOOKS

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Appendix

A Sample Logbook 13

1. Summary

This Standard Operating Procedure (SOP) establishes requirements for the entry of information into logbooks to ensure that E & E field activities are properly documented. The project manager (PM) and the field team leader (FTL) are responsible for ensuring that logbook entries provide sufficient information for the completion of an accurate and detailed description of field operations and meets the requirements of the contract or technical direction document (TDD).

This SOP describes logbook entry requirements for all types of projects, specifies the format that should be used, and provides examples. Some flexibility exists when implementing the SOP because different types of projects require different data collection efforts. This SOP does not address site safety logbook requirements or geotechnical logbook entries.

2. Purpose

Complete and accurate logbook entries are important for several reasons: to ensure that data collection associated with field activities is sufficient to support the successful completion of the project; to provide sufficient information so that someone not associated with the project can independently reconstruct the field activities at a later date; to maintain quality control (QC) throughout the project; to document changes to or deviations from the work plan; to fulfill administrative needs of the project; and to support potential legal proceedings associated with a specific project.

2.1 Adequate Field Information/Quality Control

QC procedures for data collection begin with the complete and systematic documentation of all persons, duties, observations, activities, and decisions that take place during field activities. It is especially important to fully document any deviations from the contract, project scope, work plans, sampling plans, site safety plans, quality assurance (QA) procedures, personnel, and responsibilities, as well as the reasons for the deviations.

Prior to entering the field, the project manager must indicate to the field team what pertinent information must be collected during field activity in order to meet the desired objectives of the data collection effort. The PM is responsible for reviewing the adequacy of the project logbooks both during and following completion of field activities, and is also responsible for meeting with the field team members to discuss any findings and to direct activities to correct any deficiencies, as appropriate. The PM also has the responsibility of ensuring that the logbooks become part of the project or TDD file.

2.2 Work Plan Changes/Deviation

The logbook is the document that describes implementation of the work plan and other appropriate contract documents and provides the basis for the project reports. It must include detailed descriptions of any and all deviation from the work plan and the circumstances that necessitate such changes. These changes will be reviewed for compliance with data quality objectives and include:

- Changes in procedures agreed to in the project planning stages;
- Any conditions that prevent the completion of the field effort, or that result in additional fieldwork must be noted (i.e., weather delays, government actions, physical obstructions, personnel/equipment problems, etc.). Persons from whom permission was obtained to make such changes must be clearly documented.
- Any modifications requested by the client or client's representative that are contradictory to the contract or outside of the existing scope of work must be documented in detail because the cost of the project could be affected by such modifications.

2.3 Evidentiary Documentation

Field activity documentation can become evidence in civil and/or criminal judicial proceedings, as well as in administrative hearings. Field logbooks serve this purpose. Accordingly, such documentation is subject to judicial or administrative review. More importantly, it is subject to the review of an opposing counsel who will attempt to discredit its evidentiary value.

The National Enforcement Investigation Center (NEIC) and the United States Environmental Protection Agency (EPA) have prepared documents outlining their documentation needs for legal proceedings. These guidelines indicate the importance of accurate and clear documentation of information obtained during the inspections, investigations, and evaluations of uncontrolled hazardous waste sites. Consequently, attention to detail must be applied by E & E personnel to all field documentation efforts for all E & E projects. Project personnel must document where, when, how, and from whom any vital project information was obtained. This information is necessary to establish a proper foundation for admissible evidence.

3. Guidelines

Logbooks should contain a summary of any meeting or discussion held with a client or with any federal, state, or other regulatory agency that was on site during the field activities. The

logbook should also describe any other personnel that appear on site, such as representatives of a potential responsible party (PRP).

The logbook can be used to support cost recovery activities. Data concerning site conditions must be recorded before the response activity or the passage of time eliminates or alters those conditions. Logbooks are also used to identify, locate, label, and track samples and their final disposition. In addition, data recorded in the logbook will assist in the interpretation of the analytical results.

Logbooks are subject to internal and external audits. Therefore, the recorded information should be consistent with and capable of substantiating other site documentation such as time cards, expense reports, chain-of-custody forms, shipping papers, and invoices from suppliers and subcontractors, etc. Logbooks also act as an important means of reconstructing events should other field documents such as data collection forms become lost or destroyed. Therefore, all mission-essential information should be duplicated in the logbook.

3.1 General Instructions

The following general guidelines must be used for all logbooks:

- At a minimum, one separate field activity logbook must be maintained for each project or TDD.
- All logbooks must be bound and contain consecutively numbered pages.
- No pages may be removed for any reason, even if they are partially mutilated or illegible.
- All field activities must be recorded in the site logbook (e.g., meetings, sampling, surveys, etc.).
- All information must be **printed legibly** in the logbook using waterproof ink, preferably black. If weather conditions do not permit this (i.e., if it is too cold or too wet to write with ink), another medium, such as pencil, may be used. The reason that waterproof ink was not used should be specifically noted in the logbook.
- The language used in the logbook should be objective, factual, and free of personal feelings or terminology that might prove inappropriate.
- Entries should be made in chronological order. Contemporaneous entries are always preferred because recollections fade or change over time. Observations that cannot be recorded during field activities should be recorded as soon after as possible. If logbook entries

are not made during field activities, the time of the activity/observation and the time that it is recorded should be noted.

- The first entry for each day will be made on a new, previously blank page.
- Each page should be dated and each entry should include the time that the activity occurred based on the 24-hour clock (e.g., 0900 for 9 a.m., 2100 for 9 p.m.).
- At the completion of the field activity, the logbook must be returned to the permanent project or TDD file.

3.2 Format

The information presented below is not meant to be all-inclusive. Each project manager is responsible for determining the specific information requirements associated with a field activity logbook. If someone other than the Project Manager is keeping the logbook, the Project Manager is responsible to convey to that individual, prior to the start of fieldwork, specific instructions on what type of information is required to be entered into the logbook. Information requirements will vary according to the nature and scope of the project. (Refer to Appendix A for an example of a completed logbook.)

Title Page

The logbook title page should contain the following items:

- Site name,
- Location,
- TDD No. or Job No.,
- PAN (an EPA site/task identification number), if applicable,
- SSID No. (Site ID number-assigned under CERCLA), if applicable,
- Start/Finish date, and
- Book ___ of ___.

First Page

The following items should appear on the first page of the logbook prior to daily field activity entries:

- TDD No. or Job No.,
- Date,
- Summary of proposed work (Reference work plan and contract documents, as appropriate),
- Weather conditions,
- Team members and duties, and
- Time work began and time of arrival (24-hour clock).

Successive Pages

In addition to specific activity entries and observations, the following items should appear on every logbook page:

- Date,
- TDD or Job No., and
- Signature (bottom of each page). If more than one person makes entries into the logbook, each person should sign next to his or her entry.

Last Page

In addition to specific activity entries and observations and the items that should appear on each successive page, the last page of the logbook should contain a brief paragraph that summarizes the work that was completed in the field. This summary can become especially important later on if more or less work was accomplished during the duration of the field activity.

3.3 Corrections

If corrections are necessary, they must be made by drawing a single line through the original entry in such a manner that it can still be read. *Do not erase or render an incorrect notation illegible.* The corrected entry should be written beside the incorrect entry, and the

correction must be initialed and dated. Most corrected errors will require a footnote explaining the correction.

4. Documentation

Although the requirements and content of the field logbook will vary according to the site and the tasks to be performed, the following information should be included in every logbook:

4.1 Prior to Fieldwork

Summary of Proposed Work

The first paragraph of **each** daily entry should summarize the work to be performed on that day. For example:

> "Collect soil and groundwater samples from previously installed wells and ship samples to Analytical Services Center (ASC). Discuss removal with site owner."

The first paragraph becomes especially important later when discussing work plan deviations or explaining why more or less work was accomplished for that day.

Personnel

Each person to be involved in activities for the day, his/her respective role (sampler, health and safety, etc.), and the agency he/she represents should be noted in the logbook.

On-Site Weather Conditions

Weather conditions may have an impact on the work to be performed or the amount of time required to perform the proposed work; therefore, all weather on-site weather conditions should be noted, including temperatures, wind speed and direction, precipitation, etc., and updated as necessary. Similarly, any events that are impacted by weather conditions should be noted in the logbook.

Site Safety Meeting

Although minutes should be recorded for all site safety meetings under separate cover, the logbook should briefly summarize the site safety meeting and any specific site conditions and resultant site safety concerns.

4.2 Site Sketch

A site sketch should be prepared on the first day of field activities to indicate prominent site and environmental features. The sketch should be made either to scale or by noting the approximate distances between site feature. Area-specific sketches should be prepared as work is undertaken in such areas, and updated sketches should be drawn as work progresses.

Site Features

Examples of features to be noted on the site sketch include the following:

- Structures such as buildings or building debris;
- Drainage ditches or pathways, swales, and intermittent streams (include direction of overland runoff flow and direction of stream flow);
- Access roads, site boundaries, and utility locations;
- Decontamination and staging areas;
- Adjacent property data: the type of property that borders the site, information pertaining to ownership, and available addressees; and
- North arrow.

Changes in Site Conditions

Any deviation from previous site sketches or drawings presented in the work plan, and any changes that have occurred since the last site visit must be noted. Differences to be noted include the following:

- Demolished buildings;
- Changes to access routes;
- Damage to wells or equipment, or changes to the amount of such equipment believed to be on site,
- Changes resulting from vandalism;
- Destruction of reference points;

- Changes resulting from environmental events or natural disasters; and

- Locations of excavations, waste piles, investigation-derived waste (IDW), drum staging areas, etc.

In short, *any* site condition that varies from the conditions described in the work plan should be noted.

4.3 Monitoring Equipment and Activities

Any monitoring equipment used during field activities should be documented in the logbook. Information to be noted includes:

- The type of equipment with model and serial numbers. (HNu, OVA, etc.);

- The frequency at which monitoring is performed;

- Calibration results and the frequency at which the equipment is calibrated or tested;

- Background readings;

- Any elevated or unusual readings; and

- Any equipment malfunctions.

It is particularly important to note elevated or unusual equipment readings because they could have an impact on personal protection levels or the activities to be performed on site. If a change in the proposed work or protection levels occurs, it should be clearly noted in the logbook.

4.4 Sample Collection Activities

Because it represents the first step in an accurate chain-of-custody procedure, field sampling documentation must be complete. The following items should be documented in the logbook:

Sample Collection Procedures

The following items pertaining to sample collection procedures should be included in the logbook:

- Any pre-sampling activities (i.e., well purging and the number of volumes purged before sample collection);

- Results of the pre-sampling activities (i.e., pH/conductivity/temperature readings for well water, results of hazard categorization testing, etc.);

- Any environmental conditions that make sample collection difficult or impossible (i.e., dry or flooded drainage paths, inclement weather conditions, etc.); and

- Any deviation from the work plan (i.e., additional samples and the reason for their collection, alternate sample locations, etc.).

Sample Information

The following information regarding sample data should be recorded in the logbook:

- Sample number and station location including relationship to permanent reference point(s);

- Name(s) of sampler(s);

- Sample description and any field screening results;

- Sample matrix and number of aliquots if a composite sample;

- Preservatives used, recipient laboratory, and requested analyses;

- QA/QC samples; and

- Shipping paper (airbill) numbers, chain-of-custody form numbers, and jar lot numbers.

Investigation-Derived Waste/Sample Shipment

Details pertaining to sampling equipment, decontamination, and IDW should be clearly delineated in the work plan. However, the following information should be included in the logbook:

- The type of IDW generated an the number of containers generated (each drum should be numbered and its contents noted);

- All information relevant to the characterization of the IDW;

- Any directions received from the client/workplan/contract relative to the management of the IDW;
- The disposition of IDW (left on site or removed from site);
- The number of sample containers shipped to the ASC or laboratory and the courier used (i.e., Federal Express, Airborne Express, etc.);
- Airbill or shipment tracking numbers; and
- The type of paperwork that accompanied the waste/sample shipment (e.g., manifests, etc.).

4.5 Photodocumentation

Photographs should be taken during all relevant field activities to confirm the presence or absence of contaminants encountered during fieldwork. Specific items to be documented include:

- Sample locations and collection activities;
- Site areas that have been disturbed or impacted, and any evidence of such impacts (i.e., stressed vegetation, seepage, discolored water, or debris);
- Hazardous materials requiring disposal, including materials that may not appear in the work plan;
- Any evidence that attests to the presence or absence of contamination; and
- Any features that do not appear in the work plan or differ from those described in the work plan.

Documentation of any photographs taken during the course of the project must be provided in the logbook with a detailed description of what is shown in the photograph and the reason for taking it. This documentation should include:

- Make, model, and serial numbers of the camera and lens,
- Film type and number of exposures,
- Roll and frame number of the photograph,

- Direction or view angle of the photograph, and
- Name of the photographer.

4.6 Data Collection Forms

Certain phases of fieldwork may require the use of project-specific data collection forms, such as task data sheets or hazard categorization data sheets. Due to the specific nature of these forms, the information that should be included in the logbook cannot be fully discussed in this SOP. However, the following data should be included in the logbook:

- Results of any field tests or hazard categorization tests (i.e., ignitability, corrosivity, reactivity, etc.);
- The source from which any field sample was collected and its condition (i.e., drum, tank, lagoon, etc.).
- Other conclusions as a result of the data collected on data collection forms.

In many cases, rubber stamps that contain routine data collection forms can be manufactured ahead of time. These forms can be stamped into the logbook on an as-needed basis.

Appendix A

Sample Logbook

RI 6130

WEDNESDAY JANUARY 26, 1994

PROPOSED WORK FOR DAY: COLLECT GROUNDWATER SAMPLES FROM WELLS AND PIEZOMETERS AT SITE 1 AND SITE 3. SHIP SAMPLES TO THE ASC. CONTAINERIZE PURGE WATER. MEET WITH FRED CANSLER AND DISCUSS REMOVAL OF CANOPIES AT SITES 1 AND 3 AND FILLING OF EXCAVATIONS.

WEATHER ON SITE: CLOUDY AND WARM WITH A HIGH TEMPERATURE OF 50° F. RAIN SHOWERS WITH WINDS FROM THE SW AT 5-15 MPH.

E&E PERSONNEL ON SITE: G. JONES, J. MAYS, S. McCONE

LOG

1330 ARRIVED ON SITE. THE GROUNDWATER SAMPLING CREW WAS PREPARING TO PURGE THE WELLS AND PIEZOMETERS IN THE FIELD ACROSS THE ROAD FROM SITE 1. PURGING OF WELLS BEING COMPLETED WITH HAND BAILERS SINCE PUMP IS INOPERATIVE.

1340 ARRIVED AT SITE 3. MW3-1 AND MW3-5 UNLOCKED AND OPEN; SECURED BOTH WELLS.

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RI 6130 1/26/94

1350 FRED CANSLER ARRIVED ON SITE. DISCUSSED REMOVAL OF CANOPIES AND CLOSURE OF EXCAVATIONS AT SITES 1 AND 3. FRED CANSLER STATED THAT HE HAS A SOURCE FOR THE ROCK AND FOR THE TOP SOIL FOR THE EXCAVATIONS.

1405 ARRIVED AT THE SITE WHERE FRED CANSLER PROPOSES TO REMOVE THE FILL FOR THE EXCAVATIONS. A HILL ON THE WEST SIDE OF THE WOODEN NICKLE IS IN THE PROCESS OF BEING REMOVED. THE ROCK CONSISTS OF WEATHERED SHALE SIMILAR TO THE ROCK REMOVED FROM THE EXCAVATIONS. FRED CANSLER PROPOSES TO USE THE ROCK TO FILL THE EXCAVATIONS TO WITHIN ONE FOOT OF GRADE.

1415 ARRIVED AT THE SITE WHERE FRED CANSLER PROPOSES TO REMOVE TOP SOIL FOR THE EXCAVATIONS. TOP SOIL REMOVED FROM THE YELLOW FREIGHT LOT IS IN PILES ON THE NORTH SIDE OF THE LOT.

1430 RETURNED TO SITE 3. FRED CANSLER WILL ARRANGE TO REMOVE THE CANOPY OVER THE EXCAVATION AT SITE 3 ON THURSDAY MORNING AND WILL ARRANGE TO BRING THE ROCK IN ON THURSDAY AFTERNOON. TWO TRUCKS WILL BE USED TO HAUL THE FILL. THE SUPPORTS HOLDING THE CANOPY

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1430 (cont) will be cut and the canopy dragged away from the excavation.

1445 Contacted Joy Inman from Environcies. Tankers will be on site on Thursday to pump out the excavation at Site 3 and on Friday to remove water at Site 1. A frac tank will be delivered to Site 1 on Thursday.

1515 Sampling Crew completed packing samples collected at Site 1. All wells and piezometers at Site 1 have been sampled.

1530 Sampling Crew completed packing samples and securing drums of purge water.

1535 Sampling Crew departed site to deliver samples to Federal Express.

1600 Contacted Tim Grady from E+E. Discussed conversation with Fred Cansler and status of well/piezometer sampling.

1615 Secured for day.

Work Completed: Collected groundwater samples from Site 1 wells and piezometers. Discussed removal of canopies and filling of excavations with Fred Cansler. Shipped samples to ASC.

1/26/94

Thursday January 27, 1994

Proposed Work for Day: Complete collection of groundwater samples at Site 3 and ship the samples to the ASC. Remove the canopies covering the excavations at Sites 1 and 3. Pump the water out of the excavations at Sites 1 and 3 and ship the water off site to OSCO. Backfill the excavation at Site 3. Remove the drums from the roll off box and transfer the drums to the warehouse.

Weather on site: Cloudy and cool with a high temperature of 45°F. Winds variable 10-20 mph.

E+E Personnel on site: G. Jones, T. Mays, S. McCune

LOG

0700 Scott McCune arrived at Site 3.

0710 Environcies personnel arrived at Site 3.

0715 Held site safety meeting. Discussed physical and chemical hazards associated with site and proposed work for the day.

0725 E+E sampling team arrived on site.

1/27/94

1/27/94 RI6130

0730 E+E Sampling Crew Commenced Collecting Samples and Purging MW3-1 and MW3-2.

0800 Fred Cansler Arrived on Site with Personnel to Remove the Canopy over the Excavation at Site 3. The Supports were Cut and the Canopy was Dragged away from the Excavation with two Tractors.

0845 The Canopy Removal at Site 3 Completed and the Crew Departed for Site 1.

0850 Commenced Pumping Water from the Excavation into Bryson Trailer # 618CS.

0915 The E+E Sampling Team Completed Collecting the Groundwater Samples from MW3-1, MW3-2, MW3-3, and MW3-4. Commenced Packing Samples.

0935 Completed Filling Bryson Trailer # 618CS with 5,000 Gallons of Water and Prepared Manifest # 00941 for Load. Commenced Loading Bryson Trailer # 429.

1000 E+E Sampling Team Departed the Site to Deliver Samples to Federal Express.

1030 Arrived at Site 1. The Cansler Crew is in the Process of Removing the Canopy over the Excavation. Canopy is not Moving as a Unit.

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1045 Returned to Site 3. All Water in the Excavation has been Removed except for the Ice. Bryson Trailer # 429 Loaded with 5,200 Gallons of Water. Prepared Manifest # 00942 for Load. Both Tankers Departed the Site.

1100 Environics Personnel Opened the Drums of Drilling Fluids, Development Water and Purge Water and Found the Drums Full of Ice. Environics will Contact Gary Shockley and Recommend that the Drums of Liquids be Transported to OSCO for Treatment since they can not be Bulked.

1200 Cansler Crew Commenced Loading Trucks with Stone from the Site West of the Wooden Nickel.

1230 Arrived at the Site where the Stone was Being Loaded. The Fill Material is all Undisturbed Weathered Bedrock.

1245 Arrived at Site 3. Two Loads of Rock Fill have been Dumped in the Excavation; an Estimated Four More Loads of Stone will be Needed to Fill the Excavation.

1300 Arrived at Site 1. Bryson Trailer # 617

43 [signature] 1/27/94

TITLE:	SURFACE WATER SAMPLING
CATEGORY:	ENV 3.12
REVISED:	March 1998

STANDARD OPERATING PROCEDURE

SURFACE WATER SAMPLING

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TITLE:	SURFACE WATER SAMPLING		
CATEGORY:	ENV 3.12	REVISED:	March 1998

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1. Introduction

This Standard Operating Procedure (SOP) outlines recommended procedures and equipment for the collection of representative liquid samples (aqueous and nonaqueous) from streams, rivers, lakes, ponds, lagoons, and surface impoundments both at the surface and at various depths in the water column. This SOP does not pertain to the collection of groundwater samples.

2. Method Summary

Sampling situations vary widely and therefore, no universal sampling procedure can be recommended. A sampling plan must be completed before any sampling operation is attempted. The sampling plan should include objectives of the study, the number and type of samples required to meet these objectives, and procedures to collect these samples based on site characteristics.

The sampling of both aqueous and nonaqueous liquids from the above-mentioned sources is generally accomplished through the use of one of the following:

- Kemmerer bottle,
- Bacon bomb,
- Dip sampler, or
- Direct method.

These sampling techniques will allow for the collection of representative samples from the majority of surface water types and impoundments encountered.

3. Potential Problems

There are two primary potential problems associated with surface water sampling: cross-contamination of samples, and improper sample collection.

Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and bottles. If this is not possible or practical, then decontamination of sampling equipment is necessary. See E & E's SOP on *Equipment Decontamination* (ENV 3.15).

Improper sample collection can involve using contaminated equipment, disturbance of stream or impoundment substrate, and sampling in a disturbed area such as that caused by a boat wake. Following proper decontamination procedures and minimizing disturbance of the sample site will minimize or eliminate these problems.

4. Equipment

Equipment needed for collecting surface water samples includes:

- Kemmerer bottle,
- Bacon bomb,
- Dip sampler,
- Line and messengers,
- Sample bottles, preservative, ziploc bags, ice, coolers,
- Chain-of-custody seals and forms, field data sheets,
- Decontamination equipment,
- Protective clothing,
- Maps/plot plan,
- Safety equipment,
- Compass,
- Tape measure,
- Survey stakes, flags, or buoys and anchors,
- Camera and film,
- Logbook, and
- Sample bottle labels.

5. Reagents

Reagents are commonly used to preserve samples and to decontaminate sampling equipment. Appropriate preservation and decontamination procedures should be selected prior to field sampling.

Preservatives commonly used include:

- Nitric acid (HNO_3) for metals analyses,
- Sodium hydroxide (NaOH) for cyanide analysis,
- Sulfuric acid (H_2SO_4) for TRPH analysis, and
- Hydrochloric acid (HCl) for VOC analysis.

Decontamination reagents include:

- Nitric acid (HNO_3),
- Acetone, and
- Deionized or distilled water.

6. Health and Safety

Personal safety is always the most important factor in any sampling operation. Sampling under unknown conditions should always be considered worst case, necessitating the selection of appropriate personal protection.

When sampling lagoons or surface impoundments containing known or suspected hazardous substances, adequate precautions must be taken to ensure the safety of sampling personnel. The sampling team member collecting the sample should not get too close to the edge of the impoundment, where bank failure may cause him/her to lose their balance. The person performing the sampling should be on a lifeline and wearing adequate protective equipment.

When conducting sampling from a boat in an impoundment or flowing waters, appropriate boating safety procedures will be followed.

7. Procedures

7.1 Sampling Considerations

7.1.1 Preparation

Prior to the initiation of any sampling operation, the immediate area should be checked for radioactivity, volatile organic compounds (VOCs), photoionization potential, airborne dust, and explosivity, as required by the Site Safety Plan. The following steps should then be taken:

- Determine the extent of the sampling effort, the sampling methods to be employed, and the equipment and supplies needed;
- Obtain necessary sampling and monitoring equipment;
- Decontaminate or preclean equipment, and ensure that it is in working order;
- Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate; and
- Use stakes, flags, or buoys and anchors to identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

7.1.2 Representative Samples

In order to collect a representative sample, the hydrology and morphology of a stream or impoundment should be determined prior to sampling. This will aid in determining the presence of phases or layers in lagoons or impoundments, flow patterns in streams, and appropriate sample locations and depths. Additional information can be found in the references listed in Section 12.

Generally, the deciding factors in the selection of a sampling device for surface water sampling are:

- The depth and flow of surface water body,
- Location from where the sample will be collected, and
- The depth at which the sample(s) is to be collected.

7.1.3 Sampler Composition

The sampling device must be constructed of the appropriate materials. Samplers constructed of glass, stainless steel, PVC, or PFTE (teflon) should be used, depending on the types of analyses to be performed (i.e., samples to be analyzed for metals should not be collected in metallic containers).

7.2 Sample Collection

7.2.1 Kemmerer Bottle

A Kemmerer bottle may be used in most situations where site access is from a boat or structure such as a bridge or pier, and where samples at depth are required. Sampling procedures are as follows:

- Using a properly decontaminated Kemmerer bottle, set the sampling device so that the sampling end pieces are pulled away from the sampling tube, allowing the substance to pass through this tube;
- Slowly lower the preset sampling device to the predetermined depth. Avoid bottom disturbance;
- When the Kemmerer bottle is at the required depth, send down the messenger, closing the sampling device; and
- Retrieve the sampler. Transfer sample to sample container.

7.2.2 Bacon Bomb

This type of sampler may be used in situations similar to those outlined for the Kemmerer bottle. Sampling procedures are as follows:

- Lower the bacon bomb sampler carefully to the desired depth, allowing the line for the trigger to remain slack at all times. When the desired depth is reached, pull the trigger line until taut; and
- Release the trigger line and retrieve the sampler. Transfer the sample to the sample container by pulling on the trigger.

7.2.3 Dip Sampler

A dip sampler is useful for situations in which a sample is to be recovered from an outfall pipe, such as through a storm sewer grating, or along a lagoon bank where direct accessibility is limited. The long handle on such a device allows access from a discrete location. The procedure is as follows:

- Assemble the device in accordance with the manufacturer's instructions,
- Extend the device to the sample location and collect the sample, and
- Retrieve the sampler.

7.2.4 Direct Method

For streams, rivers, lakes, and other surface waters, the direct method may be utilized to collect water samples from the surface. This method is not to be used for sampling lagoons or other impoundments where contact with contaminants is a concern.

Using adequate protective clothing (i.e., gloves and hip waders), access the sampling station by appropriate means (wading or boat). For shallow stream stations, collect the sample under the water surface pointing the sample container upstream. The container must also be upstream of the collector. Avoid disturbing the substrate. For lakes and other impoundments, collect the sample under the water surface avoiding surface debris and the boat wake.

8. Sample Preservation, Containers, Handling, and Storage

Sample preservation, sample containers, sample handling, and sample storage are critical concerns for many types of analyses. Once the analyses to be performed are determined, E & E's SOP on sample packaging and shipping should be consulted to determine the above parameters. This must be completed prior to field sampling.

Once the samples have been collected, the following procedure should be followed:

- Transfer the sample(s) into suitable and labeled sample containers;
- Preserve the sample, if appropriate;
- Cap and put a custody seal on the container, package appropriately, and place in an iced cooler if required;
- Record all pertinent data in the field logbook and on a field data sheet;
- Complete chain-of-custody record and sample analysis request form;
- Attach custody seals to cooler prior to shipment; and

- Decontaminate all sampling equipment prior to the collection of additional samples.

9. Calculations

This procedure does not involve specific calculations.

10. Quality Assurance

There are no specific quality assurance (QA) activities that apply to the implementation of these procedures. However, the following general QA procedures apply:

- All data must be documented on field data sheets or within field or site log-books;
- All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling or operation and must be documented; and
- All deliverables will receive a peer review prior to release.

11. Data Validation

The data generated will be reviewed according to the QA considerations listed in Section 9.

12. References

U.S. Environmental Protection Agency, 1991, *Compendium of ERT Surface Water and Sediment Sampling Procedures*, Interim Final, OSWER Directive 9360.4-03.

_____, 1984, *Characterization of Hazardous Waste Sites - A Methods Manual: Volume II, Available Sampling Methods*, (2nd ed.), EPA/600/4-84-076.

U.S. Geological Survey, 1977, *National Handbook on Recommended Methods for Water Data Acquisition, Office of Water Data Coordination, Reston, Virginia.*

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CATEGORY:	ENV 3.13	REVISED:	August 1997

STANDARD OPERATING PROCEDURE

SOIL SAMPLING

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1. Introduction

This document describes the procedures for the collection of representative soil samples. Representative sampling ensures the accurate characterization of site conditions. Analysis of soil samples may determine pollutant concentrations and the accompanying risks to public health, welfare, or the environment.

2. Scope

Included in this discussion are procedures for obtaining representative samples, quality assurance/quality control (QA/QC) measures, proper documentation of sampling activities, and recommendations for personnel safety.

3. Method Summary

Soil samples may be recovered using a variety of methods and equipment. These are dependent on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type.

Samples of near-surface soils may be easily obtained using a spade, stainless-steel spoon, trowel, or scoop. Sampling at greater depths may be performed using a hand auger, a power auger, or, if a test pit is required, a backhoe.

All sampling devices should be cleaned using pesticide-grade acetone (assuming that acetone is not a target compound) or methanol, then wrapped in clean aluminum foil, and custody sealed for identification. The sampling equipment should remain in this wrapping until it is needed. Each sampler should be used for one sample only. However, dedicated tools may be impractical if there are a large number of soil samples required. In this case, samplers should be cleaned in the field using standard decontamination procedures as outlined in E & E's SOP for Equipment Decontamination (see ENV 3.15).

4. Sample Preservation, Containers, Handling, and Storage

The chemical preservation of solids is not generally recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time.

Soil samples should be handled according to the procedures outlined in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

5. Potential Problems

Potential problems with soil sampling include cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and bottles. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection is generally the result of the use of

contaminated equipment; the disturbance of the matrix, resulting in compaction of the sample; and inadequate homogenization of the sample where required, resulting in variable, nonrepresentative results. Specific advantages and disadvantages of soil sampling equipment are presented in Table 5-1.

Table 5-1

SOIL SAMPLING EQUIPMENT

Equipment	Applicability	Advantages and Disadvantages
Trier	Soft surface soil	Inexpensive; easy to use and decontaminate; difficult to use in stony, dry, or sandy soil.
Scoop, trowel, spoon, or spatula	Soft surface soil	Inexpensive; easy to use and decontaminate; trowels with painted surfaces should be avoided.
Tulip bulb planter	Soft soil, 0 to 6 inches	Easy to use and decontaminate; uniform diameter and sample volume; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; not useful for hard soils.
Spade or shovel	Medium soil, 0 to 12 inches	Easy to use and decontaminate; inexpensive; can result in sample mixing and loss of VOCs.
Vehimeyer soil outfit	Soil, 0 to 10 feet	Difficult to drive into dense or hard material; can be difficult to pull from ground.
Soil coring device and auger	Soft soil, 0 to 24 inches	Relatively easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; can be difficult to decontaminate.
Thin-walled tube sampler	Soft soil, 0 to 10 feet	Easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); may be used to help maintain integrity of VOA samples; easy to decontaminate; can be difficult to remove cores from sampler.
Split-spoon sampler	Soil, 0 inches to bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); acetate sleeve may be used to help maintain integrity of VOA samples; useful for hard soils; often used in conjunction with drill rig for obtaining deep cores.
Shelby tube sampler	Soft soil, 0 inches to bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); tube may be used to ship sample to lab undisturbed; may be used in conjunction with drill rig for obtaining deep cores and for permeability testing; not durable in rocky soils.
Laskey sampler	Soil, 0 inches to bedrock	Excellent depth range; preserves soil cores; used in conjunction with drill rig for obtaining deep core; can be difficult to decontaminate.
Bucket auger	Soft soil, 3 inches to 10 feet	Easy to use; good depth range; uniform diameter and sample volume; acetate sleeve may be used to help maintain integrity of VOA samples; may disrupt and mix soil horizons greater than 6 inches in thickness.
Hand-operated power auger	Soil, 6 inches to 15 feet	Good depth range; generally used in conjunction with bucket auger for sample collection; destroys soil core (unsuitable for VOA and undisturbed sample collection); requires two or more equipment operators; can be difficult to decontaminate; requires gasoline-powered engine (potential for cross-contamination).
Continuous-flight auger	Soil, 0 inches to bedrock	Excellent depth range; easy to decontaminate; can be used on all soil samples; results in soil mixing and loss of VOCs.

Table 5-1

SOIL SAMPLING EQUIPMENT

Equipment	Applicability	Advantages and Disadvantages
Dutch auger	Designed specifically for wet, fibrous, or rooted soils (e.g., marshes)	
Eijkelcamp stoney soil auger	Stoney soils and asphalt	
Backhoe	Soil, 0 inches to 10 feet	Good depth range; provides visual indications as to depth of contaminants; allows for recovery of samples at specific depths; can result in loss of VOCs and soil mixing; shoring required at depth.

Note: Samplers may not be suitable for soils with coarse fragments.
Augers are suitable for soils with limited coarse fragments; only the stoney auger will work well in very gravelly soil.

6. Soil Sampling Equipment

Soil Sampling Equipment List

- Stainless-steel spoon
- Trier
- Scoop
- Trowel
- Spatula
- Stainless-steel tulip bulb planter
- Spade or shovel
- Vehimeyer soil sampler outfit
  - tubes
  - points
  - drive head
  - drop hammer
  - fuller jack and grip
- Soil-coring device
- Thin-walled tube sampler
- Split-spoon sampler
- Shelby tube sampler
- Laskey sampler
- Bucket auger
- Hand-operated power auger
- Continuous-flight auger
- Dutch auger
- Eijkelcamp stoney soil auger
- Backhoe
- Hand auger with replaceable sleeves

Sampling Support Equipment and Documentation List

- Sampling plan
- Sample location map
- Safety equipment, as specified in the Health and Safety Plan
- Decontamination supplies and equipment, as described in the Work Plan
- Compass
- Tape measure
- Survey stakes or flags
- Camera
- Stainless-steel buckets or bowls
- Sample containers, precleaned (i.e., I-Chem)
- Logbook
- Chain-of-custody forms
- Plastic sheet
- Soil gas probes
- Infiltrometer
- Pounding sleeve
- Extension rods
- T-handle

Labeling, Packaging, and Shipping Supplies

- Coolers
- Labels for sample containers and coolers (e.g., "fragile")
- Ice
- Plastic bags for sample containers and ice
- ESC paint cans and clamps for polychlorinated biphenyl (PCB) sampling
- Vermiculite
- Duct and strapping tape
- Federal Express airbills and pouches

6.1 Geophysical Equipment

Geophysical techniques can be integrated with field analytical and soil sampling equipment to help define areas of subsurface contamination. For a description of the geophysical techniques and associated applications, refer to E & E's SOP for Surface Geophysical Techniques (see GEO 4.2).

7. Reagents

This procedures does not require the use of reagents except for decontamination of equipment, as required. Refer E & E's SOP for Equipment Decontamination (see ENV 3.15) and the Site-Specific Work Plan (SSWP) for proper decontamination procedures and appropriate solvents.

8. Procedures

8.1 Office Preparation

1. The preparation of a Health and Safety Plan is required prior to any sampling. The plan must be approved and signed by the Corporate Health and Safety Officer or his/her designee (i.e., the Regional Safety Coordinator [RSC]).

2. Prepare a sampling plan to meet the data quality objectives (DQO) of the project in accordance with contract requirement. Review available background information (i.e., topographic maps, soil survey maps, geologic maps, other site reports, etc.) to determine the extent of the sampling effort, the sampling method to be employed, and the type and amounts of equipment and supplies required.

3. Obtain necessary sampling and monitoring equipment (see Section 6), decontaminate or preclean the equipment, and ensure that it is in working order.

4. Contact delivery service to confirm ability to ship all equipment and samples. Determine if shipping restrictions exist.

5. Prepare schedules and coordinate with staff, clients, and regulatory agencies, if appropriate.

8.2 Field Preparation

1. Identify local suppliers of sampling expendables (e.g., ice, plastic bags) and overnight delivery services (e.g., Federal Express).

2. Decontaminate or preclean all equipment before soil sampling, as described in E & E's SOP for Equipment Decontamination (see ENV 3.15), or as deemed necessary.

3. A general site survey should be performed prior to site entry in accordance with the Health and Safety Plan followed by a site safety meeting.

4. Identify and stake all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations will be utility-cleared by the property owner or field team prior to soil sampling.

8.3 Representative Sample Collection

The objective of representative sampling is to ensure that a sample or group of samples adequately reflect site conditions.

8.3.1 Sampling Approaches

It is important to select an appropriate sampling approach for accurate characterization of site conditions. Each approach is defined below. Table 8-1 summarizes the following sampling approaches and ranks them from most to least suitable based on the sampling objective.

Table 8-1

REPRESENTATIVE SAMPLING APPROACH COMPARISON

Sampling Objective	Judgmental	Random	Stratified Random	Systematic Grid	Systematic Random	Search	Transect
Establish Threat	1	4	3	2[a]	3	3	2
Identify Sources	1	4	2	2[a]	3	2	3
Delineate Extent of Contamination	4	3	3	1[b]	1	1	1
Evaluate Treatment and Disposal Options	3	3	1	2	2	4	2
Confirm Cleanup	4	1[c]	3	1[b]	1	1	1[c]

1 Preferred approach.
2 Acceptable approach.
3 Moderately acceptable approach.
4 Least acceptable approach.
a Should be used with field analytical screening.
b Preferred only where known trends are present.
c Allows for statistical support of cleanup verification if sampling over entire site.
d May be effective with compositing techniques if site is presumed to be clean.

8.3.1.1 Judgmental Sampling

Judgmental sampling is based on the subjective selection of sampling locations relative to historical site information, on-site investigation (site walk-over), etc. There is no randomization associated with this sampling approach because samples are collected primarily at areas of suspected highest contaminant concentrations. Therefore, any statistical calculations based on the sampling results would be unfairly biased.

8.3.1.2 Random Sampling

Random sampling involves the arbitrary collection of samples within a defined area. Refer to EPA 1984 and February 1989 for a random number table and guidelines on selecting sample coordinates. The arbitrary selection of sample locations requires each sample location to be chosen independently so that results in all locations within the area of concern have an equal chance of being selected. To facilitate statistical probabilities of contaminant concentration, the area of concern must be homogeneous with respect to the parameters being monitored. Thus, the higher the degree of heterogeneity, the less the random sampling approach will reflect site conditions (see Figure 8-1).

8.3.1.3 Stratified Random Sampling

Stratified random sampling relies primarily on historical information and prior analytical results to divide the area of concern into smaller sampling areas, or "strata." Strata can be defined by several factors, including: sampling depth, contaminant concentration levels, and contaminant source areas. Sampling locations should be selected within a strata using random selection procedures (see Figure 8-2).

8.3.1.4 Systematic Grid Sampling

Systematic grid sampling involves the division of the area of concern into smaller sampling areas using a square or triangular grid. Samples are then collected from the intersection of the grid lines, or "nodes." The origin and direction for placement of the grid should be selected by using an initial random point. The distance between nodes is dependent upon the size of the area of concern and the number of samples to be collected (see Figure 8-3).

8.3.1.5 Systematic Random Sampling

Systematic random sampling involves dividing the area of concern into smaller sampling areas as described in Section 8.3.1.4. Samples are collected within each grid cell using random selection procedures (see Figure 8-4).

8.3.1.6 Biased-Search Sampling

Search sampling utilizes a systematic grid or systematic random sampling approach to define areas where contaminants exceed cleanup standards (i.e., hot spots). The distance between the grid lines and number of samples to be collected are dependent upon the acceptable level of error (i.e., the chance of missing a hot spot). This sampling approach requires that assumptions be made regarding the size, shape, and depth of hot spots (see Figure 8-5).

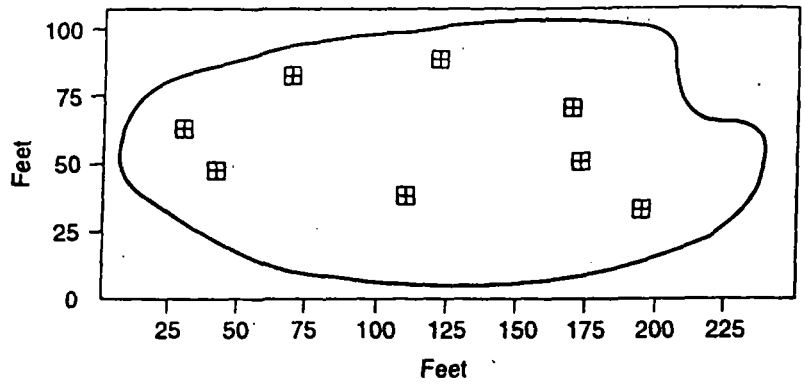


Figure 8-1 RANDOM SAMPLING**

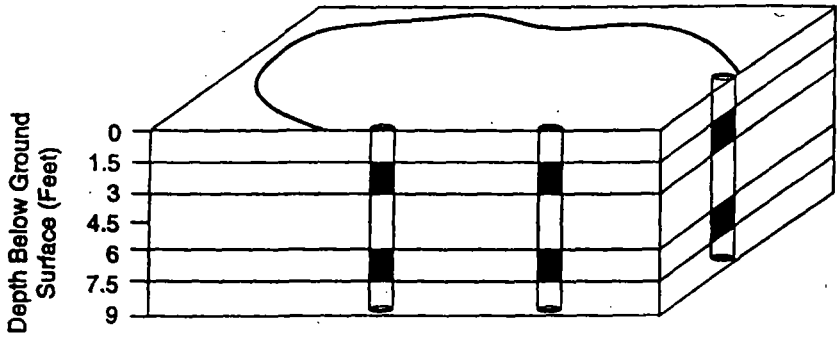


Figure 8-2 STRATIFIED RANDOM SAMPLING

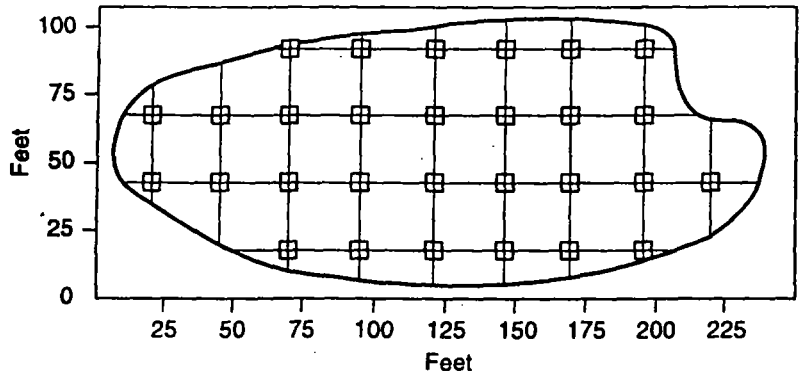


Figure 8-3 SYSTEMATIC GRID SAMPLING**

** After EPA, February 1989

Legend
- Sample Area Boundary
- Selected Sample Location
- Sample Location

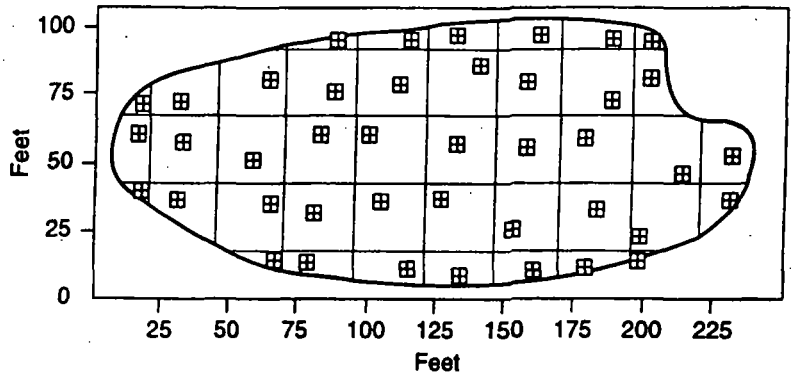


Figure 8-4 SYSTEMATIC RANDOM SAMPLING

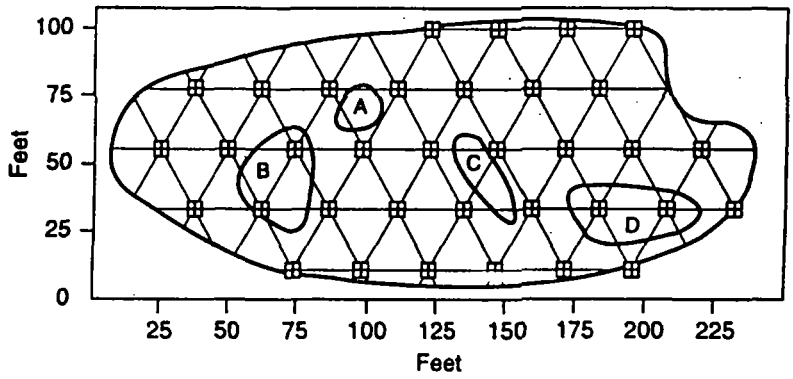


Figure 8-5 SEARCH SAMPLING

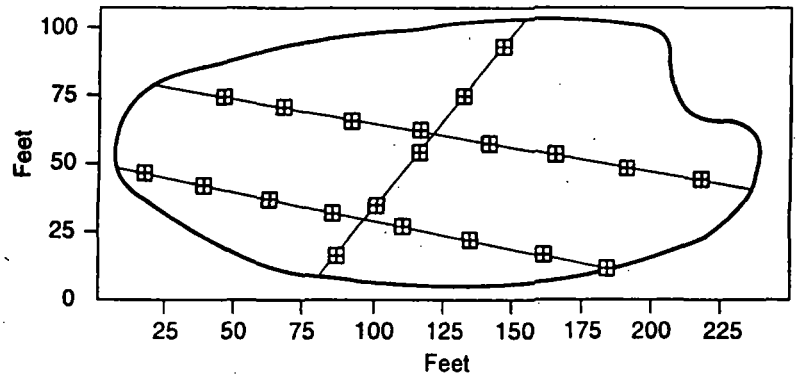


Figure 8-6 TRANSECT SAMPLING

After EPA, February 1989

Legend
- Sample Area Boundary
- Selected Sample Location
- A Sample Location

8.3.1.7 Transect Sampling

Transect sampling involves establishing one or more transect lines, parallel or nonparallel, across the area of concern. If the lines are parallel, this sampling approach is similar to systematic grid sampling. The advantage of transect sampling over systematic grid sampling is the relative ease of establishing and relocating transect lines as opposed to an entire grid. Samples are collected at regular intervals along the transect line at the surface and/or at a specified depth(s). The distance between the sample locations is determined by the length of the line and the number of samples to be collected (see Figure 8-6).

8.3.2 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, spoons, shovels, and scoops. The surface material can be removed to the required depth with this equipment; stainless-steel or plastic scoops can then be used to collect the sample.

This method can be used in most soil types, but is limited to sampling near-surface areas. Accurate, representative samples can be collected with this procedure, depending on the care and precision demonstrated by the sampling technician. The use of a flat, pointed mason trowel to cut a block of the desired soil can be helpful when undisturbed profiles are required (e.g., for volatile organic analysis [VOAs]). A stainless-steel scoop, lab spoon, or plastic spoon will suffice in most other applications. Care should be exercised to avoid the use of devices plated with chrome or other materials, as is common with garden implements such as potting trowels.

Soil samples are collected using the following procedure:

1. Carefully remove the top layer of soil to the desired sample depth with a precleaned spade;

2. Using a precleaned, stainless-steel scoop, spoon, trowel, or plastic spoon, remove and discard the thin layer of soil from the area that came into contact with the shovel;

3. Transfer the sample into an appropriate container using a stainless-steel or plastic lab spoon or equivalent. If composite samples are to be collected, place the soil sample in a stainless-steel or plastic bucket and mix thoroughly to obtain a homogeneous sample representative of the entire sampling interval. Place the soil samples into labeled containers. **(Caution: Never composite VOA samples)**;

4. VOA samples should be collected directly from the bottom of the hole before mixing the sample to minimize volatilization of contaminates;

5. Check to ensure that the VOA vial teflon liner is present in the cap, if required. Fill the VOA vial fully to the top to reduce headspace. Secure the cap tightly. The chemical preservation of solids is generally not recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time;

6. Ensure that a sufficient sample size has been collected for the desired analysis, as specified in the Sampling Plan;

7. Decontaminate equipment between samples according to E & E's SOP for Equipment Decontamination (see ENV 3.15); and

8. Fill in the hole and replace grass turf, if necessary.

QA/QC samples should be collected as specified, according to the Work Plan.

8.3.3 Sampling at Depth with Augers and Thin-Walled Tube Samplers

This system consists of an auger, a series of extensions, a T-handle, and a thin-walled tube. The auger is used to bore a hole to a desired sampling depth and is then withdrawn. The auger tip is then replaced with a tube core sampler, lowered down the borehole, and driven into the soil to the completion depth. The core is then withdrawn and the sample is collected.

Several augers are available, including: bucket type, continuous flight (screw), and posthole augers. Because they provide a large volume of sample in a short time, bucket types are better for direct sample recovery. When continuous-flight augers are used, the sample can be collected directly off the flights, usually at 5-foot intervals. The continuous-flight augers are satisfactory for use when a composite of the complete soil column is desired. Posthole augers have limited utility for sample collection, as they are designed to cut through fibrous, rooted, swampy soil.

The following procedures will be used for collecting soil samples with the hand auger:

1. Attach the auger bit to a drill rod extension, and attach the T-handle to the drill rod.

2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first 3- to 6-inches of surface soil from an area approximately 6 inches in radius around the drilling location.

3. Begin augering, periodically removing and depositing accumulated soils onto a canvas or plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole and avoids possible contamination of the surrounding area.

4. After reaching the desired depth, slowly and carefully remove the auger from the boring. When sampling directly from the auger, collect the sample after the auger is removed from boring and proceed to Step 11.

5. A precleaned stainless-steel auger sleeve can also be used to collect a sample. After reaching the desired sampling depth, remove the auger and place the sleeve inside the auger. Collect the sample with the auger. Remove the auger from the boring. The sample will be collected only from the sleeve. The soil from the auger tip should never be used for the sample.

6. Remove auger tip from dill rods and replace with a precleaned thin-walled tube sampler. Install proper cutting tip.

7. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into soil. Care should be taken to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring, as the vibrations may cause the boring walls to collapse.

8. Remove the tube sampler and unscrew the drill rods.

9. Remove the cutting tip and core from the device.

10. Discard the top of the core (approximately 1 inch), as this represents material collected before penetration of the layer in question. Place the remaining core into the sample container.

11. If required, ensure that a teflon liner is present in the cap. Secure the cap tightly onto the sample container. Place the sample bottle in a plastic bag and put on ice to keep the sample at 4°C.

12. Carefully and clearly label the container with the appropriate sample tag, addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

13. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged. Verify that the chain-of-custody form is correctly and completely filled out.

14. Record the time and date of sample collection, as well as a description of the sample in the field logbook.

15. If another sample is to be collected in the sample hole, but at a greater depth, re-attach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.

16. Abandon the hole according to applicable regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

17. Decontaminate the sampling equipment as per E & E's SOP for Equipment Decontamination (see ENV 3.15).

8.3.4 Sampling at Depth with a Trier

1. Insert the trier into the material to be sampled at a 0° to 45° angle from horizontal. This orientation minimizes the spillage of sample material. Extraction of samples may require tilting of the containers.

2. Rotate the trier once or twice to cut a core of material.

3. Slowly withdraw the trier, making sure that the slot is facing upward.

4. Transfer the sample into a suitable container with the aid of a spatula and brush.

5. If required, ensure that a teflon liner is present in the cap. Secure the cap tightly onto the sample container. Samples are handled in accordance with E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

6. Carefully and clearly label the container with the appropriate sample tag, addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

7. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged.

8. Record the time and date of sample collection as well as a description of the sample and any associated air monitoring measurements in the field logbook.

9. Abandon the hole according to applicable regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

10. Decontaminate sampling equipment as per E & E's SOP for Equipment Decontamination (see ENV 3.15).

8.3.5 Sampling at Depth with a Split-Spoon (Barrel) Sampler

The procedure for split spoon sampling describes the extraction of undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be sampled to give a complete soil column, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom the augured hole and the core extraction.

This sampling device may be used to collect information such as soil density. All work should be performed in accordance with ASTM D 1586-84, *Penetration Test and Split Barrel Sampling of Soils.*

1. Assemble the sampler by aligning both sides of the barrel and then screwing the bit on the bottom and the heavier head piece on top. Install a retaining cap in the head piece if necessary.

2. Place the sampler in a perpendicular position on the sample material.

3. Using a sledge hammer or well ring, if available, drive the tube. Do not drive past the bottom of the head piece or compression of the sample will result.

4. Record the length of the tube used to penetrate the material being sampled and the number of blows required to obtain this depth.

5. Withdraw the split spoon and open by unscrewing the bit and head. If a split sample is desired, a clean stainless-steel knife should be used to divide the tube contents in half, lengthwise. This sampler is available in 2- and 3.5-inch diameters. The required sample volume may dictate the use of the larger barrel. If needed, stainless-steel or teflon sleeves can be used inside the split spoon. If sleeves removed from the split spoon are capped immediately, volatilization of contaminants can be reduced. When split spoon sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D 1586-67 (reapproved 1974).

6. Cap the sample container, place in a double plastic bag and attach the label and custody seal. Record all pertinent data in the field logbook and complete the sample analysis request form and chain-of-custody record before taking the next sample.

7. If required, preserve or place the sample on ice.

8. Follow proper decontamination procedures and deliver samples to the laboratory for analysis.

8.3.6 Test Pit/Trench Excavation

These relatively large excavations are used to remove sections of soils when detailed examination of soil characteristics (horizontal, structure, color, etc.) are required. It is the least cost-effective sampling method due to the relatively high cost of backhoe operation.

1. Prior to any excavations with a backhoe, it is important to ensure that all sampling locations are clear of utility lines and poles (subsurface as well as above surface).

2. Using the backhoe, a trench is dug to approximately 3 feet in width and approximately 1 foot below the cleared sampling depth. Place removed or excavated soils on canvas or plastic sheets, if necessary. Trenches greater than 4 feet deep must be sloped or protected by a shoring system, as required by OSHA regulations.

3. A shovel is used to remove a 1- to 2-inch layer of soil from the vertical face of the pit where sampling is to be done.

4. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose soil for sampling. Samples are removed and placed in an appropriate container.

5. If required, ensure that a teflon liner is present in the cap. Secure the cap tightly onto the sample container. Samples are handled in accordance with E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

6. Carefully and clearly label the container with the appropriate sample tag, addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

7. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged.

8. Record the time and date of sample collection as well as a description of the sample and any associated air monitoring measurements in the field logbook.

9. Abandon the hole according to applicable State regulations. Generally, excavated holes can simply be backfilled with the removed soil material.

10. Decontaminate sampling equipment, including the backhoe bucket, as per E & E's SOP for Equipment Decontamination (see ENV 3.15).

8.4 Sample Preparation

In addition to sampling equipment, representative sample collection includes sample quantity, volume, preservation, and holding time (see Table 8-2). Sample preparation refers to all aspects of sample handling after collection. How a sample is prepared can affect its representativeness. For example, homogenizing can result in a loss of volatiles and is therefore inappropriate when volatile contaminants are the concern.

Table 8-2

STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
SW-846								
VOA[e]	14 days from date sampled	14 days from date sampled	15 g	One 40 ml vial; no air space	Two 40 ml vials; no air space	Two 40 ml vials; no air space	Cool to 4°C (ice in cooler)	Add HCl until pH <2 and cool to 4°C (ice in cooler)
Semi-VOA (BNAs)[e]	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
PCBs[d,e]	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	4 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Pesticides/PCBs[d,e]	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Metals[c]	6 months from date sampled	6 months from date sampled	10 g	300 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add HNO_3 until pH <2 and cool to 4°C (ice in cooler)
Cyanide[e]	14 days from date sampled	14 days from date sampled	10 g	100 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add NaOH until pH >12 and cool to 4°C (ice in cooler)
Hexavalent chromium[a]	24 hours from time sampled	24 hours from time sampled	10 g	50 ml	8 oz. glass jar with Teflon-lined cap	125 ml polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Total Organic Carbon (TOC)[a]	NA	28 days from date sampled	5 g	10 ml	8 oz. glass jar with Teflon-lined cap	125 ml polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add H_2SO_4 until pH <2 and cool to 4°C (ice in cooler)

Key at end of table.

Table 8-2

STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
Total Organic Halides (TOX)[a,e]	NA	7 days from date sampled	100 g	200 ml	8 oz. glass jar with Teflon-lined cap	1 L amber glass bottle	Cool to 4°C (ice in cooler)	Add H_2SO_4 until pH <2 and cool to 4°C (ice in cooler)
Total Recoverable Petroleum Hydrocarbons[c]	28 days from date sampled	28 days from date sampled	50 g	1 L	8 oz. glass jar with Teflon-lined cap	1 L amber glass bottle	Cool to 4°C (ice in cooler)	Add H_2SO_4 until pH <2 and cool to 4°C (ice in cooler)
USEPA-CLP								
VOA[e]	10 days from date received	10 days from date received	15 g	One 40 ml vial; no air space	Two 40 ml vials; no air space	Two 40 ml vials; no air space	Cool to 4°C (ice in cooler)	Add HCl until pH <2 and cool to 4°C (ice in cooler)
Semi-VOA (BNAs)[e]	10 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
PCBs[d,e]	10 days to extract from date received	5 days to extract from date received	30 g	1 L	4 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Pesticides/PCBs[d,e]	10 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Metals[c]	6 months from date sampled	6 months from date sampled	10 g	300 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add HNO_3 to pH <2 and cool to 4°C (ice in cooler)
Cyanide[e]	12 days from date received	12 days from date received	10 g	100 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add NaOH to pH >12 and cool to 4°C (ice in cooler)
NYSDEC-CLP								
VOA[b,e]	7 days from date received	10 days from date received	15 g	One 40 ml vial; no air space	Two 40 ml vials; no air space	Two 40 ml vials; no air space	Cool to 4°C (ice in cooler)	Add HCl until pH <2 and cool to 4°C (ice in cooler)
Semi-VOA (BNAs)[e]	5 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)

Key at end of table.

Table 8-2

STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
PCBs[d,e]	5 days to extract from date received	5 days to extract from date received	30 g	1 L	4 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Pesticides/PCBs[d,e]	5 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4°C (ice in cooler)	Cool to 4°C (ice in cooler)
Metals[c]	6 months from date sampled	6 months from date sampled	10 g	300 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add HNO_3 to pH <2 and cool to 4°C (ice in cooler)
Cyanide[e]	12 days from date received	12 days from date received	10 g	100 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4°C (ice in cooler)	Add NaOH to pH >12 and cool to 4°C (ice in cooler)
EPA Water and Waste								
Total dissolved solids (TDS)	NA	7 days from date sampled	NA	200 ml	NA	1 L polyethylene bottle with polyethylene-lined cap	NA	Cool to 4°C (ice in cooler)

Note: All sample bottles will be prepared in accordance with EPA bottle-washing procedures. These procedures are incorporated in E & E's Laboratory and Field Personnel Chain-of-Custody Documentation and Quality Assurance/Quality Control Procedures Manual, July 1987.

a Technical requirements for sample holding times have been established for water matrices only. However, they are also suggested for use as guidelines in evaluating soil data.
b Holding time for GC/MS analysis is 7 days if samples are not preserved.
c Maximum holding time for mercury is 28 days from time sampled.
d If one container has already been collected for PCBs analysis, then only one additional container need be collected for extractable organics, BNAs, or pesticides/PCBs analysis.
e Extra containers required for MS/MSD.

Key:

NA = Not applicable.

8.4.1 Sample Quantity and Volume

The volume and number of samples necessary for site characterization will vary according to the budget, project schedule, and sampling approach.

8.4.2 Sample Preservation and Holding Time

Sample preservation and holding times are as discussed in Section 4.

8.4.3 Removing Extraneous Material

Discard materials in a sample that are not relevant for site or sample characterization (e.g., glass, rocks, and leaves), since their presence may introduce an error in analytical procedures.

8.4.4 Homogenizing Samples

Homogenizing is the mixing of a sample to provide a uniform distribution of the contaminants. Proper homogenization ensures that the containerized samples are representative of the total soil sample collected. All samples to be composited or split should be homogenized after all aliquots have been combined. Do not homogenize samples for volatile compound analysis.

8.4.5 Compositing Samples

Compositing is the process of physically combining and homogenizing several individual soil aliquots of the same volume or weight. Compositing samples provides an average concentration of contaminants over a certain number of sampling points. Compositing dilutes high concentration aliquots; therefore, detection limits should be reduced accordingly. If the composite area is heterogeneous in concentration and its composite value is to be compared to a particular action-level, then that action-level must be divided by the total number of aliquots making up the composite for accurate determination of the detection limit.

8.4.6 Splitting Samples

Splitting samples (after preparation) is performed when multiple portions of the same samples are required to be analyzed separately. Fill the sample containers simultaneously with alternate spoonfuls of the homogenized sample (see Figure 8-7).

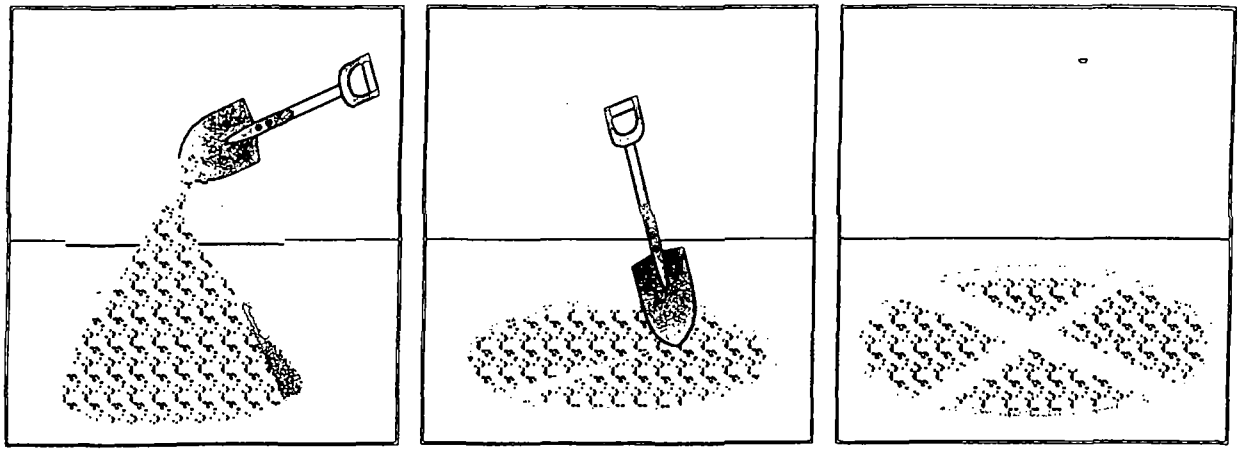
8.5 Post-Operations

8.5.1 Field

Decontaminate all equipment according to E & E's SOP for Equipment Decontamination (see ENV 3.15).

8.5.2 Office

Organize field notes into a report format and transfer logging information to appropriate forms.



Step 1:

- Cone Sample on hard, clean surface
- Mix by forming new cone

Step 2:

- Quarter after flattening cone

Step 3:

- Divide sample into quarters

Step 4:

- Remix opposite quarters
- Reform cone
- Repeat a minimum of 5 times

After: ASTM Standard C702-87

Figure 8-7 Quartering to Homogenize and Split Samples

9. Calculations

There are no specific calculations required for these procedures.

10. Quality Assurance/Quality Control

The objective of QA/QC is to identify and implement methodologies that limit the introduction of error into sampling and analytical procedures.

10.1 Sampling Documentation

10.1.1 Soil Sample Label

All soil samples shall be documented in accordance with E & E's SOP for Sample Packaging and Shipping (see ENV 3.16). The soil sample label is filled out prior to collecting the sample and should contain the following:

1. Site name or identification.
2. Sample location and identifier.
3. Date samples were collected in a day, month, year format (e.g., 03 Jan 88 for January 3, 1988).
4. Time of sample collection, using 24-hour clock in the hours: minutes format.
5. Sample depth interval. Units used for depths should be in feet and 10ths of feet.
6. Preservatives used, if any.
7. Analysis required.
8. Sampling personnel.
9. Comments and other relevant observations (e.g., color, odor, sample technique).

10.1.2 Logbook

A bound field notebook will be maintained by field personnel to record daily activities, including sample collection and tracking information. A separate entry will be made for each sample collected. These entries should include information from the sample label and a complete physical description of the soil sample, including texture, color (including notation of soil mottling), consistency, moisture content, cementation, and structure.

10.1.3 Chain-of-Custody

Use the chain-of-custody form to document the types and numbers of soil samples collected and logged. Refer to E & E's SOP for Sample Packaging and Shipping (see ENV 3.16) for directions on filling out this form.

10.2 Sampling Design

1. Sampling situations vary widely; thus, no universal sampling procedure can be recommended. However, a sampling plan should be implemented before any sampling operation is attempted, with attention paid to contaminant type and potential concentration variations.

2. Any of the sampling methods described here should allow a representative soil sample to be obtained, if the sampling plan is properly designed.

3. Consideration must also be given to the collection of a sample representative of all horizons present in the soil. Selection of the proper sampler will facilitate this procedure.

4. A stringent QA project plan should be outlined before any sampling operation is attempted. This should include, but not be limited to, properly cleaned samplers and sample containers, appropriate sample collection procedures, chain-of-custody procedures, and QA/QC samples.

11. Data Validation

The data generated will be reviewed according to the QA/QC considerations that are identified in Section 10.

11.1 Quality Assurance/Quality Control Samples

QA/QC samples are used to identify error due to sampling and/or analytical methodologies and chain-of-custody procedures.

11.1.1 Field Duplicates (Replicates)

Field duplicates are collected from one location and treated as separate samples throughout the sample handling and analytical processes. These samples are used to assess total error for critical samples with contaminant concentrations near the action level.

11.1.2 Collocated Samples

Collocated samples are generally collected 1.5 to 3.0 feet away from selected field samples to determine both local soil and contaminant variations on site. These samples are used to evaluate site variation within the immediate vicinity of sample collection.

11.1.3 Background Samples

Background or "clean" samples are collected from an area upgradient from the contamination area and representative of the typical conditions. These samples provide a standard for comparison of on-site contaminant concentration levels.

11.1.4 Rinsate (Equipment) Blanks

Rinsate blanks are collected by pouring analyte-free water (i.e., laboratory de-ionized water) on decontaminated sampling equipment to test for residual contamination. These samples are used to assess potential cross contamination due to improper decontamination procedures.

11.1.5 Performance Evaluation Samples

Performance evaluation samples are generally prepared by a third party, using a quantity of analyte(s) known to the preparer but unknown to the laboratory. The percentage of analyte(s) identified in the sample is used to evaluate laboratory procedural error.

11.1.6 Matrix Spike/Matrix Spike Duplicates (MS/MSD)

MS/MSD samples are spiked in the laboratory with a known quantity of analyte(s) to confirm percent recoveries. They are primarily used to check sample matrix interferences.

11.1.7 Field Blanks

Field blanks are prepared in the field with certified clean sand, soil, or water. These samples are used to evaluate contamination error associated with sampling methodology and laboratory procedures.

11.1.8 Trip Blanks

Trip blanks are prepared prior to going into the field using certified clean sand, soil, or water. These samples are used to assess error associated with sampling methodology and analytical procedures for volatile organics.

12. Health and Safety

12.1 Hazards Associated With On-Site Contaminants

Depending on site-specific contaminants, various protective programs must be implemented prior to soil sampling. The site Health and Safety Plan should be reviewed with specific emphasis placed on a protection program planned for direct-contact tasks. Standard safe operating practices should be followed, including minimization of contact with potential contaminants in both the vapor phase and solid matrix by using both respirators and disposable clothing.

Use appropriate safe work practices for the type of contaminant expected (or determined from previous sampling efforts):

- Particulate or Metals Contaminants
  - Avoid skin contact with, and ingestion of, soils and dusts.
  - Use protective gloves.

- Volatile Organic Contaminants
  - Pre-survey the site with an HNu 101 or OVA 128 prior to taking soil samples.
  - If monitoring results indicate organic constituents, sampling activities may be conducted in Level C protection. At a minimum, skin protection will be afforded by disposable protective clothing.

13. References

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_____, February 1989, *Methods for Evaluating the Attainment of Cleanup Standards: Volume I, Soils and Solid Media*, EPA/230/02-89/042.

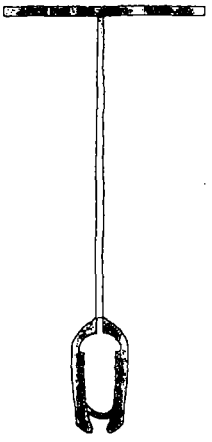
A. Sampling Augers

A. Sampling Augers

(a)
Ship Auger

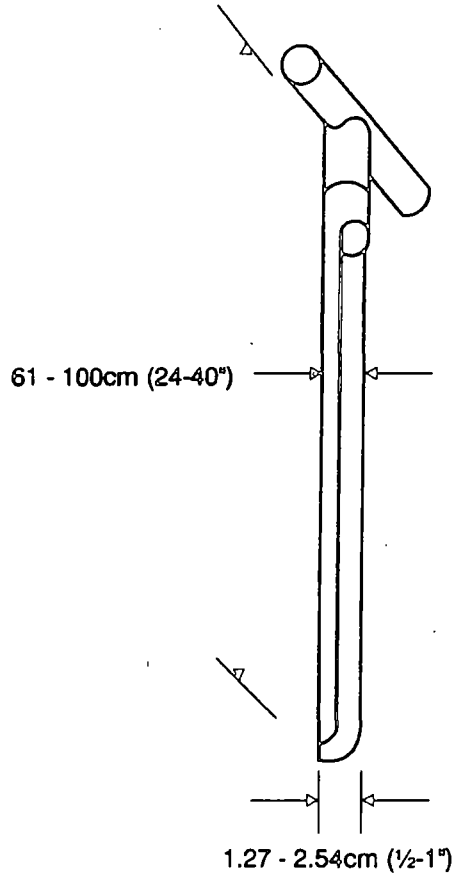
(b)
Closed-Spiral Auger

(c)
Open-Spiral Auger

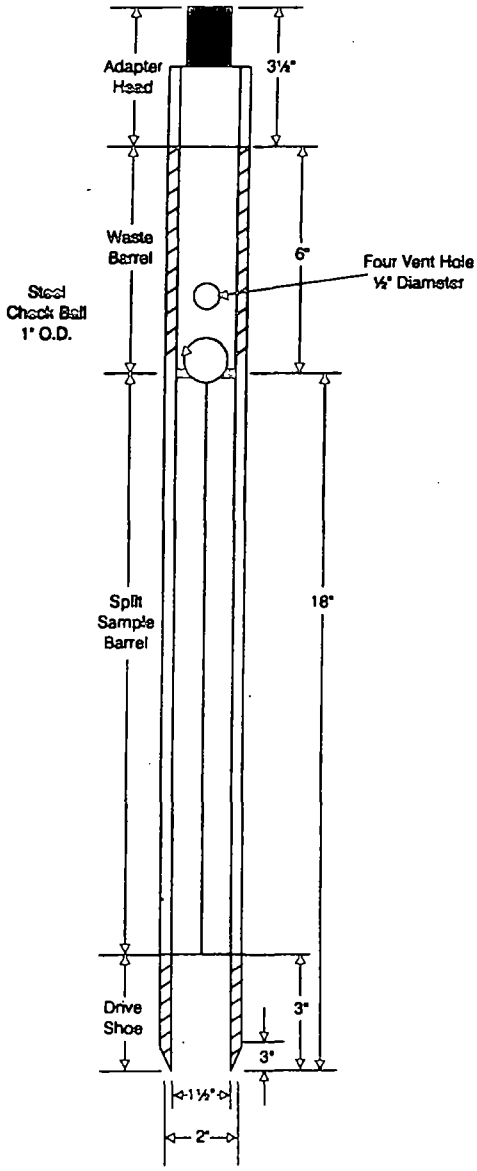


(d)
Iwan Auger

B. Sampling Trier



C. Split-Spoon Sampler



TITLE:	SEDIMENT SAMPLING
CATEGORY:	ENV 3.8
REVISED:	March 1998

STANDARD OPERATING PROCEDURE

SEDIMENT SAMPLING

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[redacted] TITLE:	SEDIMENT SAMPLING		
[redacted] CATEGORY:	ENV 3.8	[redacted] REVISED:	March 1998

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1. Introduction

This Standard Operating Procedure (SOP) describes the procedures for the collection of representative sediment samples. Analysis of sediment samples may determine whether concentrations of specific pollutants exceed established threshold action levels, and whether the concentrations of pollutants present a risk to public health, welfare, or the environment.

2. Scope

Included in this discussion are procedures for obtaining representative samples, quality assurance measures, proper documentation of sampling activities, and recommendations for personnel safety.

3. Method Summary

Sediment samples may be recovered using a variety of methods and equipment. These are dependent on 1) the depth of the water in which the samples will be collected; 2) the sediment's characteristics; 3) the volume of sediment required; and 4) the type of sample required (disturbed or undisturbed). Ultimately, the type of sampling device used should be consistent with the objective of the study.

Near-surface sediment samples may be collected using a scoop or spoon (if near shore or in shallow water), or sediment dredge or grab sampler (if in deeper water). To obtain other than surficial sediment samples, core samplers or split-spoon samplers are required.

All sampling devices should be cleaned using pesticide-grade acetone (assuming that acetone is not a target compound) or methanol, rinsed with distilled water, wrapped in aluminum foil, and custody sealed for identification. The sampling equipment should remain in this wrapping until needed. Each sampler should be used for one sample only. However, dedicated samplers may be impractical if there are a large number of sediment samples to be collected. In this case, samplers should be cleaned in the field using the decontamination procedures outlined in E & E's *Equipment Decontamination* SOP.

4. Sample Preservation, Containers, Handling, and Storage

The chemical preservation of sediments is not generally recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time. Sediment samples should be handled according to standard techniques and project-specific requirements as detailed in project work/sampling plans and quality assurance project plans.

5. Potential Problems

Potential problems with sediment sampling include cross-contamination of samples and improper sample collection. Cross-contamination problems may be eliminated or minimized through the use of dedicated sampling equipment and bottles. If this is not possible or practical, then proper decontamination of sampling equipment is necessary. Improper sample collection can involve using inadequate or inappropriate sampling devices, contaminated equipment, disturbance of the matrix resulting in compaction of the sample, and inadequate homogenization of the sample where required, resulting in variable, nonrepresentative results.

6. Equipment

The following is a list of equipment and items typically used for sediment sampling:

- Sampling plan,
- Sample location map,
- Safety equipment, as specified in the health and safety plan,
- Compass,
- Survey equipment,
- Tape measure,
- Camera,

- Four-ounce and eight-ounce glass jars with teflon liners,
- 40-ml glass vials with teflon-backed septum,
- Plastic bags for sample jars,
- Logbook,
- Labels,
- Waterproof ink pen,
- Chain-of-custody forms,
- Shipping cooler,
- Decontamination supplies and equipment, as described in the work plan,
- Canvas or plastic sheeting,
- Stainless-steel scoops,
- Stainless-steel spoons,
- Stainless-steel mixing bowls, or pans,
- Hand-driven split-spoon sampler,
- Shovel,
- Stainless-steel hand auger,
- Sediment dredge/grab sampler,
- Manual, gravity, or mechanical coring devices, and
- Teflon beaker attached to a telescoping pole.

7. Reagents

Sediment sampling does not require the use of reagents except for decontamination of equipment. Refer to E & E's *Equipment Decontamination* SOP and the site-specific work plan for proper decontamination procedures and appropriate solvents.

8. Procedures

8.1 Office Preparation

- Prepare a sampling plan in accordance with contract requirements. Conduct a literature and information search and review available background information (e.g., topographic maps, soil survey maps, geological survey maps, other site reports, etc.) to determine the extent of the sampling effort, the sampling methods to be employed, and the type and amounts of equipment and supplies required.

- E & E corporate policy requires that a health and safety plan be prepared prior to commencing any sampling activity. The plan must be approved and signed by the corporate health and safety officer or his/her designee (e.g., the regional safety coordinator [RSC]).

- Obtain necessary sampling and monitoring equipment (see Section 6), and ensure that everything is in working order.

- Contact delivery service to confirm ability to ship all equipment and samples. Determine whether shipping restrictions exist.

- Prepare schedules and coordinate with staff, clients, property owners, and regulatory agencies, if appropriate.

8.2 Field Preparation

- Identify local suppliers of sampling expendables and overnight delivery services (e.g., Federal Express).

- Decontaminate or preclean all equipment before sediment sampling, as described in E & E's *Equipment Decontamination* SOP, or as deemed necessary.

- Calibrate all health & safety monitoring equipment daily.

- A general site survey should be performed prior to site entry, in accordance with the health and safety plan. A site safety meeting identifying physical and chemical hazards should be conducted prior to sampling activities.

- Identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All locations must be cleared of utilities by the property owner or utility companies prior to sediment sampling.

8.3 Sample Collection

Numerous techniques and sampling devices may be employed to collect representative sediment samples. A number of sampling-related factors can contribute to the loss of sample integrity, including washout of fine-grained sediments during retrieval; compaction due to sample wall friction; and sampling vessel- or person-induced disturbance of surficial layers. Choosing the most appropriate sediment sampler for a study will depend on the sediment's characteristics, the volume and efficiency required, and the objectives of the study.

Most samples will be grab samples, although occasionally, sediment taken from various locations may be combined into one composite sample to reduce the amount of analytical support required.

The following procedure is used to collect surface sediment samples from small, low-flowing streams or near the shore of a pond or lake:

1. The sampler should select the sampling location furthest downstream for the first sample and work upstream. This will reduce the potential for disturbed sediments from migrating down to unsampled locations. This technique will also reduce the chances of cross-contaminating subsequent samples by sampling first in areas of suspected low contamination and working to the suspected higher concentration areas.

2. Using a precleaned, stainless-steel scoop, spoon, or other appropriate device, remove the required volume of sediment from the desired surface interval (e.g., 0-inch to 6-inch), place the sample in the appropriate precleaned glass jar, decant excess liquid as necessary, and secure the teflon-lined lid to the jar. If the sample is to be a composite sample, or if the sample is to be homogenized, the sediment is first placed in a stainless-steel mixing bowl and is homogenized prior to placement in the glass sample container. Samples for volatile organic analysis are not homogenized. Samples are handled in accordance with project-specific requirements.

3. Carefully and clearly identify the jar with the appropriate sample label, ensuring that all the categories or parameters listed in Section 10.1.1 have been addressed. Place a custody seal on the jar and lid, secure the seal in place with clear tape, and refrigerate the sample. The clear tape should also cover the jar's label.

4. Use the chain-of-custody form to document the types and number of sediment samples collected for shipment to a laboratory for analyses.

5. In the field logbook record the time and date of sample collection, as well as a description of the sample and any associated air monitoring measurements.

6. Decontaminate sampling equipment in accordance with E & E's *Equipment Decontamination* SOP.

The following procedure is used to collect subsurface sediment samples from small, low-flowing streams or near the shore of a pond or lake:

1. The sampler should select the sampling location farthest downstream for the first sample and work upstream. This will reduce the potential for disturbed sediments from migrating downstream to unsampled locations, and will also reduce the chances of cross-contaminating subsequent samples.

2. Using a precleaned split-spoon sampler or other hollow coring device, drive the sampler to the required depth with a smooth continuous motion. Remove the coring device by rotating and lifting it in a single smooth motion until the sampler is free from the sediment.

3. Before the sediment sample can be removed from the sampling device, the overlying water must be removed from the sampler by slowly pouring or siphoning it off near one side of the sampler. Care should be taken to ensure that the sediments are not disturbed, and that the fine-grained surficial sediment and organic matter are not lost while removing the overlying water.

4. Disassemble the split-spoon sampler by placing pipe wrenches on either end of the sampler. Remove both ends and open the split spoon with a precleaned stainless-steel spoon. Recover the sediment core from a core tube by pushing the sample out with a precleaned stainless-steel spoon.

5. Collect the necessary sample by cutting the core with the handle of a precleaned stainless-steel spoon, placing the sample in the appropriate precleaned glass jar, and securing the teflon-lined lid to the jar. Samples are handled in accordance with project-specific requirements.

6. Carefully and clearly label the jar with the appropriate sample tag, ensuring that all of the categories or parameters listed in Section 10.1.1 have been addressed. Place a custody seal on the jar and lid, and secure the seal in place with clear tape.

7. Use the chain-of-custody form to document the types and number of sediment samples collected and logged.

8. Record the time and date of sample collection, as well as a description of the sample and any associated air monitoring measurements, in the field logbook.

9. Decontaminate sampling equipment as per E & E's *Equipment Decontamination* SOP.

The following procedure is used to collect surface samples from rivers or from deeper lakes and ponds:

1. The sampler should select the sampling location farthest downstream for the first sample and work upstream. This will reduce the potential for disturbed sediments to migrate downstream to unsampled locations.

2. Using a precleaned sediment dredge or grab sampler, lower the sampler to the sediment layer with a polypropylene rope. Depending on the type of sampler used, the jaws of the sediment dredge will either automatically close, or will be triggered with a weighted messenger.

3. Recover the sampler and empty the sediment sample into a precleaned stainless-steel bowl. The water layer should be decanted slowly until only sediment remains in the bowl.

4. Using a precleaned stainless-steel spoon, remove the required volume of sediment. Place the sample in the appropriate precleaned glass jar, and secure the Teflon-lined lid to the jar.

5. Carefully and clearly identify the jar with the appropriate sample label, ensuring that all of the categories or parameters listed in Section 10.1.1 have been addressed. Place a custody seal on the jar and lid, and secure

the seal in place with clear tape. The clear tape should cover the sample label.

6. Use the chain-of-custody form to document the types and number of sediment samples collected for shipment to a laboratory for analyses.

7. Record the time and date of sample collection, as well as a description of the sample and any associated air monitoring measurements, in the field logbook.

8. Decontaminate sampling equipment in accordance with E & E's *Equipment Decontamination* SOP.

The following procedure is used to collect subsurface samples from rivers or from deeper lakes and ponds:

1. The sampler should select the sampling location farthest downstream for the first sample and work upstream. This will reduce the potential for disturbed sediments to migrate downstream to unsampled locations.

2. Attach a precleaned gravity or mechanical coring device to the required length of polypropylene sample line and allow the corer to freefall through the water to the bottom.

3. Determine the depth of sediment penetration, and if acceptable, retrieve the corer with a smooth, continuous lifting motion.

4. Remove the overlying water from the corer by slowly pouring or siphoning it off near one side of the sampler. Remove the nosepiece from the corer, and slide the sample out of the corer into a stainless-steel bowl or tray.

5. Collect the necessary sample by cutting the core with the handle of a stainless-steel spoon, placing the sample in the appropriate precleaned glass jar, and securing the teflon-lined lid to the jar. Samples are handled in accordance with project-specific requirements.

6. Carefully and clearly label the jar with the appropriate sample tag, ensuring that all of the categories or parameters listed in Section 10.1.1 have been addressed. Place a custody seal on the jar and lid, and secure the seal in place with clear tape.

7. Use the chain-of-custody form to document the types and number of sediment samples collected for shipment to a laboratory for analyses.

8. Record the time and date of sample collection, as well as a description of the sample and any associated air monitoring measurements, in the field logbook.

9. Decontaminate sampling equipment in accordance with E & E's *Equipment Decontamination* SOP.

8.4 Postoperations

1. Decontaminate all equipment according to E & E's *Equipment Decontamination* SOP prior to shipping the equipment back to the warehouse.

2. Organize field notes into the report format required by E & E's *Field Report Preparation* SOP. Logbooks should be maintained according to E & E's *Field Activities Log Book* SOP.

3. All samples should be shipped on the same day that they were collected to arrive at the laboratory not more than 24 hours after the samples were collected in accordance with E & E's *Sample Packaging* SOP.

9. Calculations

There are no specific calculations required for sediment sampling.

10. Quality Assurance

10.1 Sample Documentation

10.1.1 Sediment Sample Label

All sediment samples shall be documented in accordance with standard labeling techniques and project-specific requirements. The sediment sample label is completed to the fullest possible extent, prior to collecting the sample, and should contain the following minimum information:

- Site name or identification;
- Sample location and identifier;
- Date sample was collected in a day, month, year format (e.g., 03 JUN 91 for June 3, 1991);
- Time of sample collection, using 24-hour clock in the hours: minutes format; and
- Analysis required.

10.1.2 Logbook

A bound field logbook will be maintained by field personnel to record daily activities in accordance with E & E's *Field Activities Logbooks* SOP and include sample collection, tracking, and shipping information. A separate entry will be made for each sample collected. These entries should include information from the sample label and a complete description of the location from which the sediment sample was collected.

10.1.3 Chain-of-Custody

Use the chain-of-custody form to document the types and number of sediment samples collected and logged.

10.2 Sampling Plan Design

- Many of the activities critical to ensuring that the collected samples are of high quality take place in the pre-collection planning and preparation stage. Careful planning and attention to detail at this stage will result in a more successful sampling effort, and will ensure collection of the highest quality samples possible. Since site and sampling conditions vary widely, and no universal sampling procedure can be recommended, a detailed sampling plan, consistent with the objectives of the study, must be developed prior to any sampling activities.
- Any of the sampling methods described here should allow a representative sediment sample to be obtained if the sampling plan is properly designed.
- Consideration must also be given to the collection of a sample representative of all horizons present in the sediment. Selection of the proper sampling device will facilitate this procedure.

- A stringent quality assurance project plan (QAPP) should be outlined before any sampling operation is attempted. This should include, but not be limited to, the use of properly cleaned samplers and sample containers, chain-of-custody procedures, and collection of quality assurance samples such as field blanks, trip blanks, and duplicate samples.

11. Data Validation

The data generated will be reviewed according to quality assurance (QA) considerations identified in Section 10.

12. Health and Safety

Depending on site-specific contaminants, various protective programs must be implemented prior to sediment sampling. The site safety plan should be reviewed with specific emphasis placed on a protection program planned for direct contact tasks. Standard safe operating practices should be followed, including minimizing contact with potential contaminants in both vapor and solid matrix by using both respirators and disposable clothing.

Use appropriate safe work practices for the type of contaminant expected (or determined from previous sampling efforts):

Particulate or Metals Contaminants

- Avoid skin contact with and incidental ingestion of dust. Wash hands and other exposed skin areas routinely.

- Use protective gloves when collecting and handling the sediment samples.

Volatile Organic Contaminants

- Hexane acts as a carrier for a number of semivolatile organic compounds. The presence of hexane vapors in the air while decontaminating samplers indicates that the potential for exposure exists.

- If monitoring results indicate the presence of organic vapors, sampling activities must be conducted in Level C protection.

- Acetone can penetrate some types of surgical gloves; use the appropriate gloves, such as Scorpio neoprene gloves, when handling acetone.

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TITLE:	GROUNDWATER WELL SAMPLING
CATEGORY	ENV 3.7
REVISED:	March 1998

STANDARD OPERATING PROCEDURE

GROUNDWATER WELL SAMPLING

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1. Introduction

The objective of this Standard Operating Procedures (SOP) document is to provide recommended procedures for the sampling of groundwater wells, and is primarily concerned with the collection of water samples from the saturated zone of the subsurface. Every effort must be made to ensure that the sample is representative of the particular zone of water being sampled. Groundwater sampling procedures appropriate to the project objectives and site conditions will define a sampling event.

Analysis of groundwater samples may determine pollutant concentrations and its risk to public health, welfare, or the environment; extent of contaminants; and confirmation of remedial standards.

2. Scope

This document describes procedures for obtaining representative groundwater samples, quality assurance/quality control (QA/QC) measures to be followed, proper documentation of sampling activities, and recommendations for personnel safety.

3. Method Summary

Before sampling a monitoring well, the well must be purged. This may be done with a number of portable devices, including bailers, submersible pumps, bladder pumps, gas-driven pumps, gas-lift pumps, suction-lift pumps, and inertial-lift pumps. Refer to the E & E Standard Operating Procedure for *Groundwater Sampling Devices* (ENV 3.6) for information on different groundwater purging and sampling devices.

A minimum of three well volumes should be removed during well purging to ensure that a representative sample of the groundwater will be sampled. Once the purging is completed and the properly prepared sample containers have been selected, sampling may proceed. Numerous types of sampling devices may be selected for the collection of the groundwater sample, but care should be taken when selecting the sampling device, as some will affect the integrity of the sample.

Sampling should occur in a progression from the least to most contaminated well, if known. Ideally, a dedicated sampling device should be used for each well. However, dedicated sampling devices may not be practical if there are a large number of groundwater samples to be collected. In this case, sampling devices should be cleaned between sampling events using the decontamination procedures outlined in E & E Standard Operating Procedure for *Equipment Decontamination* (ENV 3.15).

4. Sample Preservation, Containers, Handling, and Storage

The type of analysis for which a sample is being collected determines the type of bottle, preservative, holding time, and filtering requirements (see Table 1). Chemical preservation and cooling of samples to 4 degrees Celcius only retards biological and chemical degradation of contaminants in the sample. Therefore, it is prudent to have the samples delivered to the laboratory as soon as possible following collection.

Sample containers should be precleaned in accordance with U.S. Environmental Protection Agency (EPA) standards and prelabeled, and preservatives should be placed in the containers prior to sample collection. When filling containers, never overfill or prerinse with the water sample, since oil or other substances may remain in the container. For analyses that may require filtered samples (e.g., metals and TOC), the samples should be filtered in the field using one 0.45-micrometer (μm) membrane filter per sample container prior to being preserved.

When all samples have been collected, a field data sheet and a chain-of-custody (C-O-C) form should be completed, and all pertinent data entered in the field logbook. Samples will be placed in a cooler to be maintained on ice at 4 degrees Celcius. Samples must be shipped to arrive at the designated laboratory well before their holding times are reached. It is preferable that these samples be shipped or delivered daily to the laboratory as outlined in the E & E Standard Operating Procedure for *Sample Packaging and Shipping* (ENV 3.16).

5. Potential Problems

5.1 General

The primary goal is to obtain a representative analysis of the groundwater body. The analysis can be compromised by field personnel in two primary ways: by collecting an unrepresentative sample, and by incorrect handling of the sample. There are numerous ways that foreign contaminants can be introduced into the sample, and these must be avoided by following strict sampling procedures and utilization of trained personnel.

Table 1

SW-846 SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS FOR WATER SAMPLES

Protocol Parameter	Holding Time	Minimum Volume	Container Type	Preservation
VOA	14 days from date sampled	One 40-ml vial; no air space	Two 40-ml vials	Add HCl until pH <2 and ice to 4° C
Semi-VOA (BNAs)	7 days to extract from date sampled	One 1-L jar	1/2-gallon amber glass bottle	Ice to 4°C
PCBs	7 days to extract from date sampled	One 1-L jar	1/2-gallon amber glass bottle	Ice to 4°C
Pesticides and PCBs	7 days to extract from date sampled	One 1-L jar	1/2-gallon amber glass bottle	Ice to 4°C
Metals	6 months from date sampled	One 300-ml bottle	1-L poly bottle	Add HNO_3 until pH <2 and ice to 4°C
Cyanide	14 days from date sampled	One 100-ml bottle	1-L poly bottle	Add NaOH until pH >12 and ice to 4°C
Hexavalent chromium	24 hours from time sampled	One 50-ml bottle	125-ml poly bottle	Ice to 4°C
TOC	28 days from date sampled	One 10-ml bottle	125-ml poly bottle	Add H_2SO_4 until pH <2 and ice to 4°C
TOX	7 days from date sampled	One 200-ml bottle	1-L amber glass bottle	Add H_2SO_4 until pH <2 and ice to 4°C
TRPHs	28 days from date sampled	One 1-L bottle	1-L amber glass bottle	Add H_2SO_4 until pH <2 and ice to 4°C

5.2 Purging

In a nonpumping well, there will be little or no vertical mixing of the water, and stratification will occur. The well water in the screened interval will mix with the groundwater due to normal flow patterns, but the water above the screened interval will remain isolated and become stagnant. Sampling team members should realize that stagnant water may contain foreign material inadvertently or deliberately introduced from the surface. To safeguard against collecting nonrepresentative stagnant water in a sample, the following guidelines and techniques should be adhered to during well purging and sampling:

- As a general rule, all monitoring wells should be pumped or bailed prior to the collection of the sample. Evacuation of a minimum of one volume of water in the well casing, and preferably three to five volumes, is recommended for a representative sample. In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, evacuation prior to sample collection is not as critical. However, in all cases where the monitoring data are to be used for enforcement actions, evacuation is recommended.

- For wells that can be pumped or bailed dry, the well should be evacuated and allowed to recover prior to sample withdrawal. If the recovery rate is fairly rapid and time allows, evacuation of more than one volume of water is preferred.

- A nonrepresentative sample can also result from excessive pumping of the monitoring well. Stratification of the leachate concentrations in the groundwater formation may occur or compounds that are heavier than water may sink to the lower portions of the aquifer. Excessive pumping can dilute or increase the contaminant concentrations from what is representative of the sampling point of interest.

Table 2

ADVANTAGES AND DISADVANTAGES OF VARIOUS GROUNDWATER SAMPLING DEVICES

Device	Advantages	Disadvantages
Bailer	◦ The only practical limitations are size and materials ◦ No power source needed ◦ Portable ◦ Inexpensive; it can be dedicated and hung in a well, reducing the chances of cross-contamination ◦ Minimal outgassing of volatile organics while sample is in bailer ◦ Readily available ◦ Removes stagnant water first ◦ Rapid, simple method for removing small volumes of purge water	◦ Time consuming, especially for large wells ◦ Transfer of sample may cause aeration
Submersible Pump	◦ Portable; can be used on an unlimited number of wells ◦ Relatively high pumping rate (dependent on depth and size of pump) ◦ Generally very reliable; does not require priming	◦ Potential for effects on analysis of trace organics ◦ Heavy and cumbersome, particularly in deeper wells ◦ Expensive ◦ Power source needed ◦ Susceptible to damage from silt or sediment ◦ Impractical in low-yielding or shallow wells
Non-Gas Contact Bladder Pump	◦ Maintains integrity of sample ◦ Easy to use	◦ Difficult to clean, although dedicated tubing and bladder may be used ◦ Only useful at depths down to approximately 100 feet • Supply of gas for operation (bottled gas and/or compressor) is difficult to obtain and is cumbersome
Suction Pump	◦ Portable, inexpensive, and readily available	◦ Only useful at depths down to approximately 25 feet ◦ Vacuum can cause loss of dissolved gases and volatile organics ◦ Pump must be primed and vacuum is often difficult to maintain ◦ May cause pH modification
Inertia Pump	◦ Portable, inexpensive, and readily available ◦ Rapid method for purging relatively shallow wells	◦ Only useful at depths down to approximately 70 feet ◦ May be time consuming to use • Labor-intensive • WaTerra pump is only effective in 2-inch diameter wells

5.3 Materials

The material used to construct groundwater purging and sampling devices can have a significant impact on the analytical results. If practical, equipment that contacts the groundwater should be constructed from stainless steel, teflon, or glass. The use of plastic should be avoided when analyzing for organics. Table 2 discusses the advantages and disadvantages of groundwater sampling devices, and Table 3 provides a ranking of sample material compatibility under various aqueous environments.

Table 3

RELATIVE COMPATIBILITY OF
RIGID GROUNDWATER SAMPLING MATERIALS

	PVC I	Galvanized Steel	Carbon Steel	Low-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/High Solids	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

Teflon
Stainless Steel 316
Stainless Steel 304
PVC I
Low-Carbon Steel
Galvanized Steel
Carbon Steel

6. Equipment Checklist

6.1 General

- Water level indicator (e.g., electric sounder, steel tape, transducer, reflection sounder, air line, etc.);
- Depth sounder;
- Appropriate keys for well cap locks;
- Steel brush;
- Organic vapor analyzer (OVA) or photo-ionization meter (HNu);
- Oil/water interface indicator (if necessary);
- Timepiece (preferably a stopwatch);
- Logbook;
- Calculator;
- Field data sheets;
- Bottle labels;
- Chain-of-custody forms;
- Custody seals;
- Sample containers;
- Engineer's rule;
- Sharp knife (locking blade);
- Tool box (screwdrivers, pliers, hacksaw, hammer, flashlight, adjustable wrench, bolt cutters, etc.);
- Leather work gloves;
- Appropriate personnel protection equipment;

- 5-gallon pails;
- Plastic sheeting;
- Sealable plastic bags;
- Shipping containers;
- Packing material;
- U.S. Department of Transportation (DOT) shipping labels;
- 55-gallon 1A2 (17-H) drums (if necessary);
- Decontamination solutions;
- Tap water;
- Non-phosphate soap;
- Aluminum foil;
- Garden sprayers;
- Brushes;
- Preservatives; and
- Distilled or deionized water, as necessary.

6.2 Groundwater Sampling Devices

Bailers

- Clean decontaminated bailers of appropriate size and construction material;
- Nylon line (enough to dedicate to each well);
- Sharp knife;
- Aluminum foil (to wrap clean bailers);

- Submersible pumps;
- Pump(s);
- Adequate power supply, generator, or battery;
- 1-inch black poly vinyl chloride (PVC) coil pipe (enough to dedicate to each well);
- Hose clamps;
- Safety cable (i.e., heavy-grade nylon line);
- Tool box supplement (pipe wrenches, wire strippers, electric tape, heat shrink, hose connectors, teflon tape);
- Winch or pulley (if desired);
- Gasoline for generator;
- Flow meter with gate valve; and
- 1-inch nipples and various pipe connectors.

Bladder Pumps

- Non-gas contact bladder pump;
- Compressor or nitrogen gas bottles;
- Batteries and charger;
- Teflon tubing (enough to dedicate to each well);
- Swagelock fitting; and
- Toolbox supplement (same as submersible pump).

Suction Pump

- Pump;
- Black coil pipe tubing (enough to dedicate to each well);

- Gasoline (if required);
- Toolbox supplement (same as submersible pump);
- Various hose connectors and nipples; and
- Flow meter with gate valve.

7. Preparation

7.1 Office Preparation

- The preparation of a Site-Specific Safety Plan (SSSP) is required prior to any sampling. The SSSP must be approved and signed by the Corporate Health and Safety Officer or designee (i.e., the Regional Safety Coordinator [RSC]);
- Prepare a Site-Specific Work Plan (SSWP) to meet the data quality objectives of the project in accordance with contract requirements. Review available background information (e.g., topographic maps, hydrogeologic maps, geologic maps, other site reports, etc.) to determine the extent of the sampling effort, the sampling method to be employed, and the type and amounts of equipment and supplies required;
- Obtain necessary sampling and monitoring equipment (see Section 6), preclean the sampling equipment, and ensure that it is in proper working order;
- Ensure that batteries are charged, including the OVA, HNu, pump control box, and large storage batteries;
- Locate local sources for preservatives and decontamination solutions. Review this matter with the RSC or site safety coordinator;
- Contact delivery service to confirm ability to ship all equipment and samples. Determine if shipping restrictions exist; and
- Prepare schedules and coordinate with staff, clients, and regulatory agencies, if appropriate.

7.2 Field Preparation

- Identify local suppliers of expendable sampling equipment such as ice and baggies, and overnight delivery services;
- Inspect all sampling equipment and reclean, if necessary, prior to groundwater sampling (see Table 4);
- A general site survey should be performed prior to site entry in accordance with the SSSP followed by a site safety meeting; and
- Identify all well locations.

Table 4

DECONTAMINATION SOLUTIONS

Type of Hazard	Name of Solution	Remarks
Amphoteric-acids and bases	Sodium bicarbonate	5-15% aqueous solution
Inorganic acids, metal processing wastes, heavy metals	Sodium carbonate	Good water softener, 10-20% aqueous solution
Solvents and organic compounds, oily, greasy unspecified wastes	Trisodium phosphate	Good rinsing solution of detergent, 10% aqueous solution
Pesticides, fungicides, cyanides, ammonia, and other non-acidic inorganic wastes	Calcium hypochlorite	Excellent disinfectant, bleaching and oxidizing agent, 10% aqueous solution
Other Types of Decontamination Solutions		
Other Detergents and Aqueous Surfactants		
Phosphate-free laboratory detergent (Alconox, Liquinox), Pennsalt 91, Oakite, Gunk, Clorox		
Solvents		
1,1,2-Trichloroethane, H2-ethyl-hexyl acetate, pesticide-grade isopropanol/acetone/methanol/hexane, heptane (nonhydrogen bonding), alcohol, diesel fuel, naphtha, beta-propiolactone, carbon tetrachloride, 8% formalinethylene, 8% hexachloromelamine, 1,2-dichloroethane (in solution), Quadcoat		
Other Solutions		
10% nitric acid, 0.1 N/10%/20% hydrochloric acid		
Water		
Potable/tap water (demonstrated to be analyte-free), distilled water, deionized water, reagent-grade distilled and deionized water		

Source: Adapted from Devinny *et al.* 1990; Mickam *et al.* 1989.

8. Reagents

Except for decontamination solutions and sample preservatives, there are no reagents required for these procedures. Refer to E & E Standard Operating Procedure for *Equipment Decontamination* (ENV 3.15), the SSSP, or the SSWP for proper decontamination procedures and appropriate solvents.

9. Field Sampling Procedures

9.1 Sampling Preparation

- Start at the least-contaminated well, if known;
- Remove locking well cap. Note the location of the well, time of day, and date in the field logbook or sample log;
- Remove the well cap covering the well riser;
- Test the well for volatile organic compounds (VOCs) and methane by conducting a headspace analysis with a combustible gas indicator, an OVA (for VOCs and methane), or an HNu (for VOCs). Record all readings in the field logbook;
- Lower water level measuring device into well until the surface of the water table is encountered;
- Measure the distance from the top of the water table to a reference point on the well riser or casing (e.g., top of inside casing [TOIC]) and record the distance in the field logbook;
- Lower the water level measuring device to the bottom of the well, and measure the total depth of the well using the same reference point on the well riser or casing. Record the distance in the field logbook.
- Measure the diameter of the well, and calculate the volume of water in the well by multiplying the number of feet of water by the number of gallons per foot (see Section 10);
- Determine the required volume of groundwater to be removed from the well (e.g., three well volumes or as indicated in the SSWP);

- Place plastic sheeting on the ground around the well to minimize the likelihood of contamination of sampling equipment from soil adjacent to the well; and

- Prepare the purging and sampling equipment.

9.2 Purging

The amount of flushing that a well receives prior to sample collection depends on the intent of the monitoring program, as well as the hydrogeologic conditions. Programs in which overall quality determinations of water resources are involved may require long pumping periods to obtain a sample that is representative of the groundwater. The pumped volume can be determined prior to sampling, or the well can be pumped until selected parameters (e.g., temperature, electrical conductance, pH, turbidity, etc.) have stabilized. Care must be taken not to exceed the recommended purging rate for monitoring well screens (see Table 5).

Monitoring for defining a contaminant plume requires a representative sample of a small volume of the aquifer. These circumstances require that the well be pumped enough to remove the stagnant water, but not enough to induce flow from other areas.

During purging, water level measurements may be taken regularly at 15- and 30-second intervals. The data may be used to compute water table or aquifer transmissivity and other hydraulic characteristics.

Information on the most commonly used groundwater purging and sampling devices can be found in E & E's SOP for *Groundwater Sampling Devices* (ENV 3.6).

9.2.1 Bailers

Equipment needed will include a clean decontaminated bailer, nylon line, a sharp knife, and plastic sheeting. Place the plastic sheeting around the well to prevent contact of the bailer or line with the ground. Attach the line to the bailer, and then lower the bailer until it is completely submerged. Pull the bailer out of the well; ensure that the line either falls onto the plastic sheeting or never touches the ground. Empty the bailer into a 5-gallon pail. Repeat the procedure until the required purge volume has been removed. When the 5-gallon pail is full, pour the water into a 55-gallon drum or handle as indicated in the SSWP.

9.2.2 Submersible Pumps

- Assemble the pump, hose, and safety cable;

- Lower the pump and assembly into the monitoring well to a point a few feet below the water level;

- Attach to a power source and commence purging operations;

Table 5

MAXIMUM RECOMMENDED PURGING RATE FOR MONITORING WELL SCREENS

Screen Type	Diameter (in)	Slot (in)	Open Area (ft^2/ft)	Open Area (%)	Recommended Pumping Rate gpm/ft at 0.1 ft/s	gpm/ft at 0.07 ft/s	gpm/ft at 0.03 ft/s
PVC (machine slot)	2	0.01	0.018	3.4	0.804	0.563	0.241
	2	0.02	0.033	6.4	1.496	1.047	0.449
	2	0.025	0.042	8.0	1.870	1.309	0.561
	2	0.04	0.060	11.5	2.693	1.885	0.808
	2	0.051	0.075	14.4	3.385	2.369	1.015
	4	0.01	0.036	3.4	1.608	1.126	0.482
	4	0.02	0.067	6.4	2.992	2.094	0.898
	4	0.025	0.083	8.0	3.740	2.618	1.122
	4	0.04	0.120	11.5	5.386	3.770	1.616
	4	0.051	0.151	14.4	6.773	4.741	2.032
PVC (wound)	2	0.01	0.047	9.0	2.119	1.484	0.636
	2	0.02	0.089	17.0	3.989	2.793	1.197
	2	0.03	0.124	23.7	5.579	3.905	1.674
	2	0.04	0.156	29.7	6.981	4.887	2.094
	2	0.05	0.183	34.9	8.197	5.738	2.459
	4	0.01	0.078	7.5	3.522	2.465	1.057
	4	0.02	0.147	14.1	6.607	4.625	1.982
	4	0.03	0.208	19.9	9.350	6.545	2.805
	4	0.04	0.262	25.0	11.750	8.225	3.525
	4	0.05	0.309	29.5	13.869	9.708	4.161
Stainless Steel (wire-wound)	2	0.01	0.090	17.1	4.021	2.814	1.206
	2	0.02	0.157	30.0	7.044	4.931	2.113
	2	0.03	0.210	40.2	9.444	6.610	2.833
	2	0.04	0.253	48.4	11.376	7.963	3.413
	2	0.05	0.287	54.8	12.872	9.010	3.862
	4	0.01	0.177	16.9	7.948	5.563	2.384
	4	0.02	0.307	29.3	13.776	9.643	4.133
	4	0.03	0.410	39.1	18.388	12.872	5.517
	4	0.04	0.492	47.0	22.097	15.468	6.629
	4	0.05	0.560	53.4	25.120	17.584	7.536

- Using a flow meter or pail and a stopwatch, determine the flow rate and calculate the time required to remove the required volume of water from the well;

- Place the purge water in 55-gallon drums or handle as indicated in the SSWP; and

- Lower the pump by stages until it is just above the screen, and continue to purge until the required volume of water has been removed from the well. In cases where the well will not yield water at a sufficient recharge rate, pump the well dry and allow it to recover.

9.2.3 Non-Gas Contact Bladder Pumps

- Assemble the teflon tubing, pump, and charged control box;

- Procedures for purging with a bladder pump are the same as for a submersible pump (Section 9.2.2); and

- Be sure to adjust the flow rate to prevent violent jolting of the hose.

9.2.4 Suction Pumps

- Assemble the pump, tubing, and power source; and

- Procedures for purging with a suction pump are the same as for a submersible pump (Section 9.2.2).

9.3 Sampling

Groundwater samples can be obtained through the use of a number of groundwater sampling devices. Each groundwater sampling device has its advantages (and disadvantages) over other devices. Ideally, groundwater sampling devices should be completely inert, economical to manufacturer, easily cleaned for reuse, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for both well purging and sample collection. There are several other factors to consider when choosing a groundwater sampling device and care should be taken when selecting the device. Refer to the E & E Standard Operating Procedure for Groundwater Sampling Devices (ENV 3.6) for additional information.

9.3.1 Bailers

- Make sure that clean plastic sheeting has been placed around the well;

- Attach a line to the bailer. If a bailer was used for purging, the same bailer and line may be used for sampling;
- Lower the bailer slowly and gently into the well, taking care not to shake the well casing or splash the bailer into the water. Lower the bailer to different points adjacent to the well screen to ensure that a representative water sample is collected;
- Slowly and gently retrieve the bailer from the well, avoiding contact with the well riser;
- Remove the cap from a sample container and place the cap on plastic sheeting or in a location where it will not be contaminated. Refer to Section 9.6 for special considerations for volatile organic analysis (VOA) samples;
- Slowly pour the water into the container;
- Filter and preserve samples as required by the SSWP. Mark the water level on the container with a pen;
- Prepare the necessary QA samples as outlined in the SSWP;
- Record sample information in the field logbook or on field data sheets, and complete the C-O-C form;
- Package samples in accordance with the SSWP; and
- Repeat this process until all groundwater samples have been collected.

9.3.2 Submersible Pumps

- Allow the monitoring well to recharge after purging, keeping the pump just above the screened interval;
- Attach a gate valve to the discharge hose, and reduce the flow rate to a manageable sampling rate;
- Prepare the sample containers;
- If no gate valve is available, discharge the sample into a clean jar and fill the sample containers from the jar;

- Complete the sampling and documentation procedures as outlined in Section 9.3.1; and

- Upon completion, remove the pump and assembly and properly decontaminate the pump prior to use in the next well. Do not reuse the discharge tubing.

9.3.3 Bladder Pump

- Allow the well to recharge after purging;

- Prepare the sample containers;

- Turn the pump on. Increase the cycle time and reduce the pressure to the minimum that will allow groundwater to come to the surface;

- Complete the sampling and documentation procedures as outlined in Section 9.3.1;

- Upon completion, remove the tubing from the well and either replace the teflon tubing and bladder with new dedicated tubing and bladder, or properly decontaminate the existing material;

- Nonfiltered groundwater samples should be collected directly from the outlet tubing into the sample containers; and

- Filtered groundwater samples should be obtained by connecting the pump outlet tubing directly to the filter unit. The pump pressure should be reduced to prevent a pressure buildup on the filter, which could damage the pump bladder.

9.3.4 Suction Pumps

- Allow the well to recharge;

- Attach a gate valve to the discharge line if the suction pump discharge rate cannot be controlled, or discharge the sample into a clean glass jar and fill the sample containers from the jar;

- Sample as outlined in Section 9.3.1; and

- Upon completion, remove the tubing and properly decontaminate the pump prior to use in the next well. Do not reuse the tubing.

9.4 Filtering

Samples being analyzed for total dissolved metals and total organic carbons (TOC) may require filtering. Two types of filters are commonly used: barrel filters and vacuum filters. A barrel filter works with a bicycle pump, which is used to build up positive pressure in the chamber containing the sample. Water is then forced through 0.45-μm filter paper into a jar. The barrel itself is filled manually.

A vacuum filter involves two chambers: the upper chamber contains the sample, and a 0.45-μm filter divides the two chambers. Using a portable vacuum pump, air is withdrawn from the lower chamber, creating a vacuum, which causes the sample to move through the filter into the lower chamber. Repeated pumping may be required to drain all of the sample into the lower chamber. If preservation of the samples is necessary, this should be done after filtering.

9.5 Post-Operation

After all samples have been collected and preserved, the sampling equipment should be properly decontaminated to prevent cross-contamination of samples.

- Decontaminate all equipment according to the SSWP;
- Replace sampling equipment in storage containers;
- Prepare groundwater samples for shipment. Check sample documentation and make sure samples are properly packed for shipment; and
- Organize field notes into a report format and transfer logging information to appropriate forms.

9.6 Special Consideration for VOA Sampling

The proper collection of a sample for dissolved VOCs requires minimal disturbance of the sample to limit volatilization and subsequent loss of volatiles from the sample.

Sample retrieval systems suitable for the valid collection of volatile organic samples include: positive-displacement bladder pumps, gear-driven submersible pumps, and syringe samplers and bailers. Field conditions and other constraints will limit the choice of appropriate systems. The principal objective is to provide a valid sample for analysis that has been subjected to the least amount of turbulence possible.

The following procedures should be followed when collecting VOA samples:

- Open the vial, set the cap in a clean place, and place the proper amount of preservatives (HCl) in the vial;
- Fill the vial to the top until a convex meniscus forms on the top of the vial. Do not overfill the vial;

- Check that the cap has not been contaminated, and carefully cap the vial. Place the cap directly over the top and screw down firmly. Do not overtighten and break the cap;

- Invert the vial and tap gently. If an air bubble appears, discard the sample and begin again. It is imperative that no entrapped air remains in the sample vial;

- Place the VOA vial in a cooler, oriented so that it is lying on its side, not straight up; and

- The holding time, under most protocol parameters, for VOAs is 14 days (see Table 1). It is recommended that samples be shipped or delivered to the laboratory daily. Ensure that the samples remain at 4°C, but do not allow them to freeze.

10. Calculations

Table 6 presents the volume of water in different size casings and holes. To determine the volume of water in a well, the calculations are as follows:

$$V = Tr2(0.163)$$

where:

V = Static volume of well in gallons

T = Depth of water in well, measured in feet (determined by subtracting the static water level from the total depth of the well)

r = Inside radius of well casing, measured in inches

0.163 = A constant conversion factor for the conversion of the casing radius from inches to feet and cubic feet to gallons

Table 6

VOLUME OF WATER IN CASING OR HOLE

Diameter of Casing or Hole (in)	Gallons per Foot of Depth	Cubic Feet per Foot of Depth	Liter per Meter of Depth	Cubic Meters per Meter of Depth
1	0.041	0.0055	0.509	0.509×10^{-3}
1.5	0.092	0.0123	1.142	1.142×10^{-3}
2	0.163	0.0218	2.024	2.024×10^{-3}
2.5	0.255	0.0341	3.167	3.167×10^{-3}
3	0.367	0.0491	4.558	4.558×10^{-3}
3.5	0.500	0.0668	6.209	6.209×10^{-3}
4	0.653	0.0873	8.110	8.110×10^{-3}
4.5	0.826	0.1104	10.260	10.260×10^{-3}
5	1.020	0.1364	12.670	12.670×10^{-3}
5.5	1.234	0.1650	15.330	15.330×10^{-3}
6	1.469	0.1963	18.240	18.240×10^{-3}
7	2.000	0.2673	24.840	24.840×10^{-3}
8	2.611	0.3491	32.430	32.430×10^{-3}
9	3.305	0.4418	41.040	41.040×10^{-3}
10	4.080	0.5454	50.670	50.670×10^{-3}
11	4.937	0.6600	61.310	61.310×10^{-3}
12	5.875	0.7854	72.960	72.960×10^{-3}
14	8.000	1.0690	99.350	99.350×10^{-3}
16	10.440	1.3960	129.650	129.650×10^{-3}
18	13.220	1.7670	164.180	164.180×10^{-3}
20	16.320	2.1820	202.680	202.680×10^{-3}
22	19.750	2.6400	245.280	245.280×10^{-3}
24	23.500	3.1420	291.850	291.850×10^{-3}
26	27.580	3.6870	342.520	342.520×10^{-3}
28	32.000	4.2760	397.410	397.410×10^{-3}
30	36.720	4.9090	456.020	456.020×10^{-3}
32	41.780	5.5850	518.870	518.870×10^{-3}
34	47.160	6.3050	585.680	585.680×10^{-3}
36	52.880	7.0690	656.720	656.720×10^{-3}

1 Gallon = 3.785 liters
1 Meter = 3.281 feet
1 Gallon water weighs 8.33 lbs. = 37.785 kilograms
1 Liter water weighs 1 kilogram = 2.205 pounds
1 Gallon per foot of depth = 12.419 liters per foot of depth
1 Gallon per meter of depth = 12.419×10^{3} cubic meters per meter of depth

11. Quality Assurance/Quality Control

The objective of QA/QC is to identify and implement methodologies that limit the introduction of error into sampling and analytical procedures. Groundwater sampling protocols appropriate to the data quality objectives and site conditions will define the specific procedures that will be followed for sampling events (see Figure 1).

There are seven primary areas of concern for QA in the collection of representative groundwater samples:

- The SSWP should be reviewed by all team personnel involved in the collection of the groundwater samples before any sampling is attempted, with attention to contaminant type and potential concentration variations;
- Log documentation should be reviewed to determine whether the required volume of purge water was removed from the well and that the temperature, electrical conductance, and pH had been stabilized to ensure that a representative water sample of the aquifer was obtained;
- The purging and sampling devices should be made of materials and utilized in a manner that will not interact with or alter the analysis;
- The results generated by these procedures are reproducible as demonstrated through the use of duplicate samples;
- The possibility of cross-contamination is reduced by collecting samples from the least contaminated well first. Rinsate blanks should be incorporated where dedicated sampling and purging equipment is not utilized and decontamination of the equipment between sampling events is required;
- Samples are properly labeled, documented (C-O-C), preserved, and shipped; and
- A record of daily field activities, such as sample collection and tracking information, is kept in a bound book.

12. Data Validation

The data generated will be reviewed according to the QA/QC considerations presented in Section 11.

Step	Procedure	Essential Elements	Recommendations
Well Inspection	Hydraulic Measurements	Water-Level Measurements	Measure the water level to ±0.3 cm (±0.01 ft).
Well Purging	Removal or Isolation of Stagnant Water	Representative Water Access	
	Determination of Well-Purging Parameters (pH, Eh, T, Ω^{-1})**	Verification of Representative Water Sample Access	Pump water until well purging parameters (e.g., pH, T, Ω^{-1}, Eh) stabilize to ±10% over at least two successive well volumes pumped.
Sample Collection Filtration* Field Determinations**	Unfiltered / Field Filtered*	Sample Collection by Appropriate Mechanism	Pumping rates should be limited to ~100mL/min for volatile organics and gas-sensitive parameters.
		Minimal Sample Handling	
	Volatile Organics, TOX	Head-Space Free Samples	Filter: Trace metals, inorganic anions/cations, alkalinity. Do not filter: TOC, TOX, volatile organic compound samples. Filter other organic compound samples only when required.
	Dissolved Gases, TOC		
	Large Volume Samples for Organic Compound Determinations	Minimal Aeration or Depressurization	
	Alkalinity/Acidity**	Minimal Air Contact Field Determination	Samples for determinations of gases, alkalinity, and pH should be analyzed in the field if at all possible.
Preservation Field Blanks Standards	Assorted Sensitive Inorganic Species NO_2, NH_4, Fe(II)	Adequate Rinsing against Contamination	
	(as needed for good QA/QC)	Minimal Air Contact Preservation	At least one blank and one standard for each sensitive parameter should be made up in the field on each day of sampling. Spiked samples are also recommended for good QA/QC
	Trace Metals for Mobile Substance Load***		
Storage Transport		Minimal Loss of Samples Integrity Prior to Analysis	Observe maximum sample holding or storage periods recommended by the Agency. Documentation of actual holding periods should be carefully performed.

* Denotes samples that should be filtered to determine dissolved constituents. Filtration should be accomplished preferably with in-line filters and pump pressure or by N_2 pressure methods. Samples for dissolved gases or volatile organics should not be filtered. In instances where well development procedures do not allow for turbidity-free samples and may bias analytical results, split samples should be spiked with standards before filtration. Both spiked samples and regular samples should be analyzed to determine recoveries from both types of handling.

** Denotes analytical determinations that should be made in the field.

See Puls and Barcelona (1989).

Figure 1 Generalized Flow Diagram of Groundwater Sampling Protocol

13. Health and Safety

Depending on the site-specific contaminants, the type of personnel protective equipment (PPE) used during the purging and sampling of the wells is outlined in the SSSP. The SSSP should be reviewed with specific emphasis placed on the safety procedures to be followed for the well sampling tasks. Standard safe operating practices should be followed, such as minimizing contact with potential contaminants in both the vapor phase and liquid matrix through the use of respirators and protective clothing.

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CATEGORY:	ENV 3.16
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STANDARD OPERATING PROCEDURE

SAMPLE PACKAGING

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STANDARD OPERATING PROCEDURE

SAMPLING EQUIPMENT DECONTAMINATION

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1 Decontamination Solvents 7

1. Scope and Application

The purpose of this procedure is to provide a description of methods for preventing or reducing cross-contamination and general guidelines for designing and selecting decontamination procedures for use at potential hazardous waste sites. The decontamination procedures chosen will prevent introduction and cross-contamination of suspected contaminants in environmental samples, and will protect the health and safety of site personnel.

2. Method Summary

Removing or neutralizing contaminants that have accumulated on personnel and equipment ensures protection of personnel from permeating substances, reduces/eliminates transfer of contaminants to clean areas, prevents the mixing of incompatible substances, and minimizes the likelihood of sample contamination.

Cross-contamination can be removed by physical decontamination procedures. The abrasive and non-abrasive methods include the use of brushes, high pressure water, air and wet blasting, and high pressure Freon cleaning. These methods should be followed by a wash/rinse process using appropriate cleaning solutions. A general protocol for cleaning with solutions is as follows:

1. Physical removal.
2. Non-phosphate detergent plus tap water.
3. Tap water.
4. 10% nitric acid.
5. Distilled/deionized water rinse.
6. Solvent rinse.
7. Total air dry.
8. Triple rinse with distilled/deionized water.

This procedure can be expanded to include additional or alternate solvent rinses that will remove specified target compounds if required by site-specific work plans (WP) or as directed by a particular client.

3. Interferences

The use of distilled/deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment provided that it has been verified by laboratory analysis to be analyte-free distilled/deionized water. Distilled water available from local grocery stores and pharmacies is generally not acceptable for final decontamination rinses. Contaminant-free deionized water is available from commercial vendors and may be shipped directly to the site or your hotel.

The use of an untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal water treatment system.

4. Equipment/Apparatus

The following are standard materials and equipment used as a part of the decontamination process:

- Appropriate protective clothing;
- Air purifying respirator (APR);
- Field log book;
- Non-phosphate detergent;
- Selected high purity, contaminant-free solvents;
- Long-handled brushes;
- Drop cloths (plastic sheeting);
- Trash containers;
- Paper towels;
- Galvanized tubs or equivalent (e.g., baby pools);
- Tap water;
- Contaminant-free distilled/deionized water;
- Metal/plastic container for storage and disposal of contaminated wash solutions;
- Pressurized sprayers, H_2O;
- Pressurized sprayers, solvents;
- Trash bags;
- Aluminum foil;
- Sample containers;
- Safety glasses or splash shield; and
- Emergency eyewash bottle.

5. Reagents

There are no reagents used in this procedure aside from decontamination solutions used for the equipment. The type of decontamination solution to be used shall depend upon the type and degree of contamination present and as specified in the project/site-specific Quality Assurance Project Plan (QAPP).

In general, the following solvents are utilized for decontamination purposes:

- 10% nitric acid wash (reagent grade nitric acid diluted with deionized/distilled water – 1 part acid to 10 parts water)[a];
- Acetone (pesticide grade)[b] ;
- Hexane (pesticide grade)[b];
- Methanol; and
- Methylene chloride[b].

[a]Only if sample is to be analyzed for trace metals.
[b]Only if sample is to be analyzed for organics requiring specific or specialized decontamination procedures. These solvents must be kept away from samples in order to avoid contamination by decon solvents.

6. Procedures

Decontamination is the process of removing or neutralizing contaminants that have accumulated on both personnel and equipment. Specific procedures in each case are designed accordingly and may be identified in either the Health and Safety Plan (HSP), WP, QAPP, or all three.

As part of the HSP, a personnel decontamination plan should be developed and set up before any personnel or equipment enters the areas of potential contamination. Decontamination procedures for equipment will be specified in the WP and the associated QAPP. These plans should include:

- Number and layout of decontamination stations;
- Decontamination equipment needed (see Section 4);
- Appropriate decontamination methods;
- Procedures to prevent contamination of clean areas;
- Methods and procedures to minimize worker contact with contaminants during removal of protective clothing;

- Methods and procedures to prevent cross-contamination of samples and maintain sample integrity and sample custody; and
- Methods for disposal of contaminated clothing, equipment, and solutions.

Revisions to these plans may be necessary for health and safety when the types of protective clothing, site conditions, or on-site hazards are reassessed based on new information.

Prevention of Contamination

Several procedures can be established to minimize contact with waste and the potential for contamination. For example:

- Employing work practices that minimize contact with hazardous substances (e.g., avoid areas of obvious contamination, avoid touching potentially hazardous substances);
- Use of remote sampling, handling, and container-opening techniques;
- Covering monitoring and sampling equipment with plastic or other protective material;
- Use of disposable outer garments and disposable sampling equipment with proper containment of these disposable items;
- Use of disposable towels to clean the outer surfaces of sample bottles before and after sample collection; and
- Encasing the source of contaminants with plastic sheeting or overpacks.

Proper procedures for dressing prior to entrance into contaminated areas will minimize the potential for contaminants to bypass the protective clothing. Generally, all fasteners (zippers, buttons, snaps, etc.) should be used, gloves and boots tucked under or over sleeves and pant legs, and all junctures taped (see the Health and Safety Plan for these procedures).

Decontamination Methods

All personnel, samples, and equipment leaving the contaminated area of a site must be decontaminated to remove any chemicals or infectious organisms that may have adhered to them. Various decontamination methods will either physically remove, inactivate by chemical detoxification/disinfection/sterilization, or remove contaminants by both physical and chemical means.

In many cases, gross contamination can be removed by physical means. The physical decontamination techniques can be grouped into two categories: abrasive methods and non-abrasive methods.

6.1 Abrasive Cleaning Methods

Abrasive cleaning methods work by rubbing and wearing away the top layer of the surface containing the contaminant. The following reviews the available abrasive methods.

Mechanical

Mechanical methods include using brushes with metal, nylon, or natural bristles. The amount and type of contaminants removed will vary with the hardness of bristles, length of time brushing, and degree of brush contact. Material may also be removed by using appropriate tools to scrape, pry, or otherwise remove adhered materials.

Air Blasting

Air blasting equipment uses compressed air to force abrasive material through a nozzle at high velocities. The distance between nozzle and surface cleaned, air pressure, and time of air blasting dictate cleaning efficiency. The method's disadvantages are its inability to control the exact amount of material removed and its large amount of waste generated.

Wet Blasting

Wet blast cleaning involves the use of a suspended fine abrasive. The abrasive/water mixture is delivered by compressed air to the contaminated area. By using very fine abrasives, the amount of materials removed can be carefully controlled.

6.2 Non-abrasive Cleaning Methods

Non-abrasive cleaning methods work by either dissolution or by forcing the contaminant off a surface with pressure. In general, less of the equipment surface is removed using non-abrasive methods.

High-Pressure Water

This method consists of a high-pressure pump, an operator controlled directional nozzle, and high-pressure hose. Operating pressure usually ranges from 340 to 680 psi, which relates to flow rates of 20 to 140 lpm.

Steam Cleaning

This method uses water delivered at high pressure and high temperature in order to remove accumulated solids and/or oils.

Ultra-High-Pressure Water

This system produces a water jet from 1,000 to 4,000 atm. This ultra-high-pressure spray can remove tightly-adhered surface films. The water velocity ranges from 500 m/sec. (1,000 atm) to 900 m/sec. (4,000 atm). Additives can be used to enhance the cleaning action, if approved by the QAPP for the project.

High-Pressure Freon Cleaning

Freon cleaning is a very effective method for cleaning cloth, rubber, plastic, and external/internal metal surfaces. Freon 113 (trichlorotriflorethane) is dense, chemically stable, relatively non-toxic, and leaves no residue. The vapor is easily removed from the air by activated charcoal. A high pressure (1,000 atm) jet of liquid Freon 113 is directed onto the surface to be cleaned. The Freon can be collected in a sump, filtered, and reused.

Physical removal of gross contamination should be followed by a wash/rinse process using cleaning solutions. One or more of the following methods utilize cleaning solutions.

Dissolving

Removal of surface contaminants can be accomplished by chemically dissolving them. although the solvent must be compatible with the equipment and protective clothing. Organic solvents include alcohols, ethers, ketones, aromatics, straight-chain alkanes, and common petroleum products. Halogenated solvents are generally incompatible with protective clothing and are toxic. Table 1 provides a general guide to the solubility of contaminant categories in four types of solvents.

Surfactants

Surfactants reduce adhesion forces between contaminants and the surface being cleaned and prevents reposition of the contaminants. Non-phosphate detergents dissolved in tap water is an acceptable surfactant solution.

Rinsing

Contaminants are removed and rinsing through dilution, physical attraction, and solubilization.

Disinfection/Sterilization

Disinfectants are a practical means of inactivating infectious agents. Unfortunately, standard sterilization methods are impractical for large equipment and personal protective clothing.

6.3 Field Sampling Equipment Cleaning Procedures

The following steps for equipment cleaning should be followed for general field sampling activities.

1. Physical removal (abrasive or non-abrasive methods).
2. Scrub with non-phosphate detergent plus tap water.
3. Tap water rinse.
4. 10% nitric acid (required during sampling for inorganics only).
5. Distilled/deionized water rinse.
6. Solvent rinse (required during sampling for organics only).
7. Total air dry (required during sampling for organics only).
8. Triple rinse with distilled/deionized water.

Table 1 lists solvent rinses which may be required for elimination of particular chemicals. After each solvent rinse, the equipment should be air-dried and triple-rinsed with distilled/deionized water.

Table 1

DECONTAMINATION SOLVENTS

Solvent	Soluble Contaminants
Water	Low-chain compounds Salts Some organic acids and other polar compounds
Dilute Bases For example: - detergent - soap	Acidic compounds Phenol Thiols Some nitro and sulfonic compounds
Organic Solvents: For example: - alcohols (methanol) - ethers - ketones - aromatics - straight-chain alkanes (e.g., hexane) - common petroleum products (e.g., fuel oil, kerosene)	Nonpolar compounds (e.g., some organic compounds)
WARNING: Some organic solvents can permeate and/or degrade the protective clothing.	

Solvent rinses are not necessarily required when organics are not a contaminant of concern. Similarly, an acid rinse is not necessarily required if analysis does not include inorganics.

NOTE: Reference the appropriate analytical procedure for specific decontamination solutions required for adequate removal of the contaminants of concern.

Sampling equipment that requires the use of plastic or teflon tubing should be disassembled, cleaned, and the tubing replaced with clean tubing, if necessary, before commencement of sampling or between sampling locations.

7. Quality Assurance/Quality Control

QA/QC samples are intended to provide information concerning possible cross-contamination during collection, handling, preparation, and packing of samples from field locations for subsequent review and interpretation. A field blank (rinsate blank) provides an additional check on possible sources of contamination from ambient air and from sampling instruments used to collect and transfer samples into sample containers.

A field blank (rinsate blank) consists of a sample of analyte-free water passed through/over a precleaned/decontaminated sampling device and placed in a clean area to attempt to simulate a worst-case condition regarding ambient air contributions to sample contamination.

Field blanks should be collected at a rate of one per day per sample matrix even if samples are not shipped that day. The field blanks should return to the lab with the trip blanks originally sent to the field and be packed with their associated matrix.

The field blank places a mechanism of control on equipment decontamination, sample handling, storage, and shipment procedures. It is also indicative of ambient conditions and/or equipment conditions that may affect the quality of the samples.

Holding times for field blanks analyzed by CLP methods begin when the blank is received in the laboratory (as documented on the chain of parameters and associated analytical methods).

Holding times for samples and blanks analyzed by SW-846 or the 600 and 500 series begins at the time of sample collection.

8. Health and Safety

Decontamination can pose hazards under certain circumstances even though performed to protect health and safety. Hazardous substances may be incompatible with decontamination methods (i.e., the method may react with contaminants to produce heat, explosion, or toxic products). Decontamination methods may be incompatible with clothing or equipment (e.g., some solvents can permeate and/or degrade protective clothing). Also, a direct health hazard to workers can be posed from chemical decontamination solutions that may be hazardous if inhaled or may be flammable.

The decontamination solutions must be determined to be compatible before use. Any method that permeates, degrades, or damages personal protective equipment should not be used. If decontamination methods do pose a direct health hazard, measures should be taken to protect personnel or modified to eliminate the hazard.

All site-specific safety procedures should be followed for the cleaning operation. At a minimum, the following precautions should be taken:

1. Safety glasses with splash shields or goggles, neoprene gloves, and laboratory apron should be worn.

2. All solvent rinsing operations should be conducted under a fume hood or in open air.

3. No eating, smoking, drinking, chewing, or any hand-to-mouth contact is permitted.

9. References

Field Sampling Procedures Manual, New Jersey Department of Environmental Protection, 1988.

A Compendium of Superfund Field Operations Methods, EPA 540/p-87/001.

Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual, USEPA Region IV, April 1, 1986.

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/OSHA/USCG/EPA, October 1985.

APPENDIX B

SUPPLEMENTAL FORMS, SAMPLE DOCUMENTATION, AND CHAIN-OF-CUSTODY FORMS

SAMPLE PLAN ALTERATION FORM

Project Name and Number: _____

_____

_____

Material to be Sampled: _____

_____

_____

Measurement Parameters: _____

_____

_____

Standard Procedure for Field Collection & Laboratory Analysis (cite references): _____

_____

_____

_____

Reason for Change in Field Procedure of Analytical Variation: _____

_____

_____

Variation from Field or Analytical Procedure: _____

_____

_____

Special Equipment, Materials, or Personnel Required: _____

_____

_____

_____

CONTACT	APPROVED SIGNATURE	DATE
Initiator:		
START PL:		
EPA TM:		
EPA QA Officer:		

MJY1Ø6 - TOTAL METALS MJY1Ø7 - TOTAL METALS MJY1Ø8 - TOTAL METALS

MJY1Ø6 - TOTAL METALS MJY1Ø7 - TOTAL METALS MJY1Ø8 - TOTAL METALS

MJY1Ø6 - CYANIDE MJY1Ø7 - CYANIDE MJY1Ø8 - CYANIDE

MJY1Ø6 - CYANIDE MJY1Ø7 - CYANIDE MJY1Ø8 - CYANIDE

MJY1Ø6 MJY1Ø7 MJY1Ø8

MJY1Ø6 MJY1Ø7 MJY1Ø8

MJY1Ø6 MJY1Ø7 MJY1Ø8

JN996 - EXTRACTABLE JN997 - EXTRACTABLE JN998 - EXTRACTABLE

JN996 - EXTRACTABLE JN997 - EXTRACTABLE JN998 - EXTRACTABLE

JN996 - EXTRACTABLE JN997 - EXTRACTABLE JN998 - EXTRACTABLE

JN996 - EXTRACTABLE JN997 - EXTRACTABLE JN998 - EXTRACTABLE

JN996 - VOA JN997 - VOA JN998 - VOA

JN996 - VOA JN997 - VOA JN998 - VOA

JN996 JN997 JN998

JN996 JN997 JN998

JN996 JN997 JN998

JN996 JN997 JN998

Tag No. 153684	Sampler Name(s) (Please Print)	**Preservative:** Ice ☐ No ☐ **Chemical:** Acid☐ Base☐ Other☐
Month/Day/Year/Time		**Analyses**
		General Chemistry
		BOD Anions Cations Solids (TSS, TDS, SS)
		COD, TOC, Nutrients
		Oil and Grease
		Inorganics
		Metals
		Cyanide
		Organics
Project Code	Station Location / No.	Volatiles
		Semi-volatiles
		Pesticides/PCBs
		PAHs
		Herbicides
		Phenols
		Other (Write In)
CLP No.		Regional Tracking No.
Remarks:		

☆ U.S. GPO:1997—591-913

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10

CUSTODY SEAL

Date:
Signature:

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10

EPA United States Environmental Protection Agency
Contract Laboratory Program

Inorganic Traffic Report & Chain of Custody Record
(For Inorganic CLP Analysis)

Case No.

1. Project Code	Account Code	2. Region No.	Sampling Co	4. Date Shipped	Carrier
Regional Information		Sampler (Name)		Airbill Number	
Non-Superfund Program		Sampler Signature		5. Ship To	
Site Name		3. Purpose* Lead: ☐ SF ☐ PRP ☐ ST ☐ FED; Early Action: ☐ CLEM ☐ PA ☐ REM ☐ RI ☐ SI ☐ ESI; Long-Term Action: ☐ FS ☐ RD ☐ RA ☐ O&M ☐ NPLD			
City, State	Site Spill ID			ATTN:	

6. Matrix (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Field QC
5. Soil/Sediment
6. Oil (High only)
7. Waste (High only)
8. Other (specify in Column A)

7. Preservative (Enter in Column D)
1. HCl
2. HNO_3
3. NaOH
4. H_2SO_4
5. $K_2CR_2O_7$
6. Ice only
7. Other (specify in Column D)
N. Not preserved

CLP Sample Numbers (from labels)	A Matrix (from Box 6) Other:	B Conc. Low Med High	C Sample Type: Comp./ Grab	D Preservative (from Box 7) Other:	E - RAS Analysis: Diss. Metals	Total Metals	Cyanide	Low only: NO_2/NO_3	Fluoride	High only: pH	Conduct.	F Regional Specific Tracking Number or Tag Numbers	G Station Location Identifier	H Mo/Day/ Year/Time Sample Collection	I Corresponding CLP Organic Sample No.	J Sampler Initials	K Field QC Qualifier (B = Blank, S = Spike, D = Duplicate, R = Rinsate, PE = Perform. Eval., N = Not a QC Sample)

Shipment for Case Complete? (Y/N)	Page ___ of ___	Sample(s) to be Used for Laboratory QC	Additional Sampler Signatures	Chain of Custody Seal Number(s)

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks: Is custody seal intact? Y/N/none	

DISTRIBUTION: Green - Region Copy; White - Lab Copy for Return to Region; Pink - CLASS Copy; Yellow - Lab Copy for Return to CLASS

EPA Form 9110-1 (2/98)

SEE REVERSE FOR ADDITIONAL STANDARD INSTRUCTIONS
*SEE REVERSE FOR PURPOSE CODE DEFINITIONS

A21-012-13 REV

380159

☆ U.S. GOVERNMENT PRINTING OFFICE: 1993-430-800

EPA United States Environmental Protection Agency
Contract Laboratory Program

Organic Traffic Report & Chain of Custody Record
(For Organic CLP Analysis)

Case No.

1. Project Code	Account Code	2. Region No.	Sampling Co.	4. Date Shipped	Carrier
Regional Information		Sampler (Name)		Airbill Number	
Non-Superfund Program		Sampler Signature		5. Ship To	
Site Name		3. Purpose*			
City, State	Site Spill ID			ATTN:	

3. Purpose*
- Lead: SF, PRP, ST, FED
- Early Action: CLEM, PA, REM, RI, SI, ESI
- Long-Term Action: FS, RD, RA, O&M, NPLD

6. Matrix (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Field QC
5. Soil/Sediment
6. Oil (High only)
7. Waste (High only)
8. Other (Specify in Column A)

7. Preservative (Enter in Column D)
1. HCl
2. HNO3
3. NaHSO4
4. H2SO4
5. Ice only
6. Other (Specify in Column D)
N. Not preserved

CLP Sample Numbers (from labels)	A Matrix (from Box 6) Other:	B Conc.: Low Med High	C Sample Type: Comp Grab	D Preservative (from Box 7) Other:	E RAS Analysis VOA	BNA	Pest/PCB	High only ARO/TOX	F Regional Specific Tracking Number or Tag Numbers	G Station Location Identifier	H Mo/Day/Year/Time Sample Collection	I Corresponding CLP Inorganic Sample No.	J Sampler Initials	K Field QC Qualifier B = Blank, S = Spike, D = Duplicate, R = Rinsate, PE = Performance Eval., — = Not a QC Sample

Shipment for Case Complete? (Y/N)	Page ____ of ____	Sample(s) to be Used for Laboratory QC	Additional Sampler Signatures	Chain of Custody Seal Number(s)

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks: Is custody seal intact? Y/N/none	

A21-012-15-REV.

DISTRIBUTION: Blue - Region Copy; White - Lab Copy for Return to Region; Pink - CLASS Copy; Yellow - Lab Copy for Return to CLASS

EPA Form 0110-2 (2/98)

SEE REVERSE FOR ADDITIONAL STANDARD INSTRUCTIONS
*SEE REVERSE FOR PURPOSE CODE DEFINITIONS

DATE: SAMPLE COLLECTION SHEET TDD:

LOCATION	SAMPLE NUMBER	SAMPLE MEDIA	SAMPLE TYPE	DEPTH	TIME	SAMPLER	CONTAINER	NOTES

APPENDIX C

D 2488-93$^{\epsilon 1}$ ASTM STANDARD PRACTICE FOR DESCRIPTION AND IDENTIFICATION OF SOILS (Visual-Manual Procedure)

Designation: D 2488 – 93[ε1]

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

ε[1] Note—Paragraph 1.6 was added editorially in October 1998.

1. Scope

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (disturbed and undisturbed).

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (See Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 The values stated in inch-pound units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 8.

1.6 *This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2113 Practice for Diamond Core Drilling for Site Investigation[2]

D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)[2]

D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)[2]

3. Terminology

3.1 *Definitions:*

3.1.1 Except as listed below, all definitions are in accordance with Terminology D 653.

NOTE 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:
Cobbles—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and
Boulders—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1.1 *clay*—soil passing a No. 200 (75-μm) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index equal

[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.1.2 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

coarse—passes a 3-in. (75-mm) sieve and is retained on a ¾-in. (19-mm) sieve.

fine—passes a ¾-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.1.3 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.1.4 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.1.5 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.1.6 *sand*—particles of rock that will pass a No. 4 (4.75-mm) sieve and be retained on a No. 200 (75-µm) sieve with the following subdivisions:

coarse—passes a No. 4 (4.75-mm) sieve and is retained on No. 10 (2.00-mm) sieve.

medium—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425-µm) sieve.

fine—passes a No. 40 (425-µm) sieve and is retained on a No. 200 (75-µm) sieve.

3.1.1.7 *silt*—soil passing a No. 200 (75-µm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit is below the "A" line (see Fig. 3 of Test Method D 2487).

Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Fig. 1a and Fig. 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

NOTE 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols*.

Dual Symbol—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

Borderline Symbol—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together; one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D 4083 when working with frozen soils.

6. Apparatus

6.1 *Required Apparatus*:

6.1.1 *Pocket Knife or Small Spatula.*

6.2 *Useful Auxiliary Apparatus*:

6.2.1 *Small Test Tube and Stopper* (or jar with a lid).

6.2.2 *Small Hand Lens.*

7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

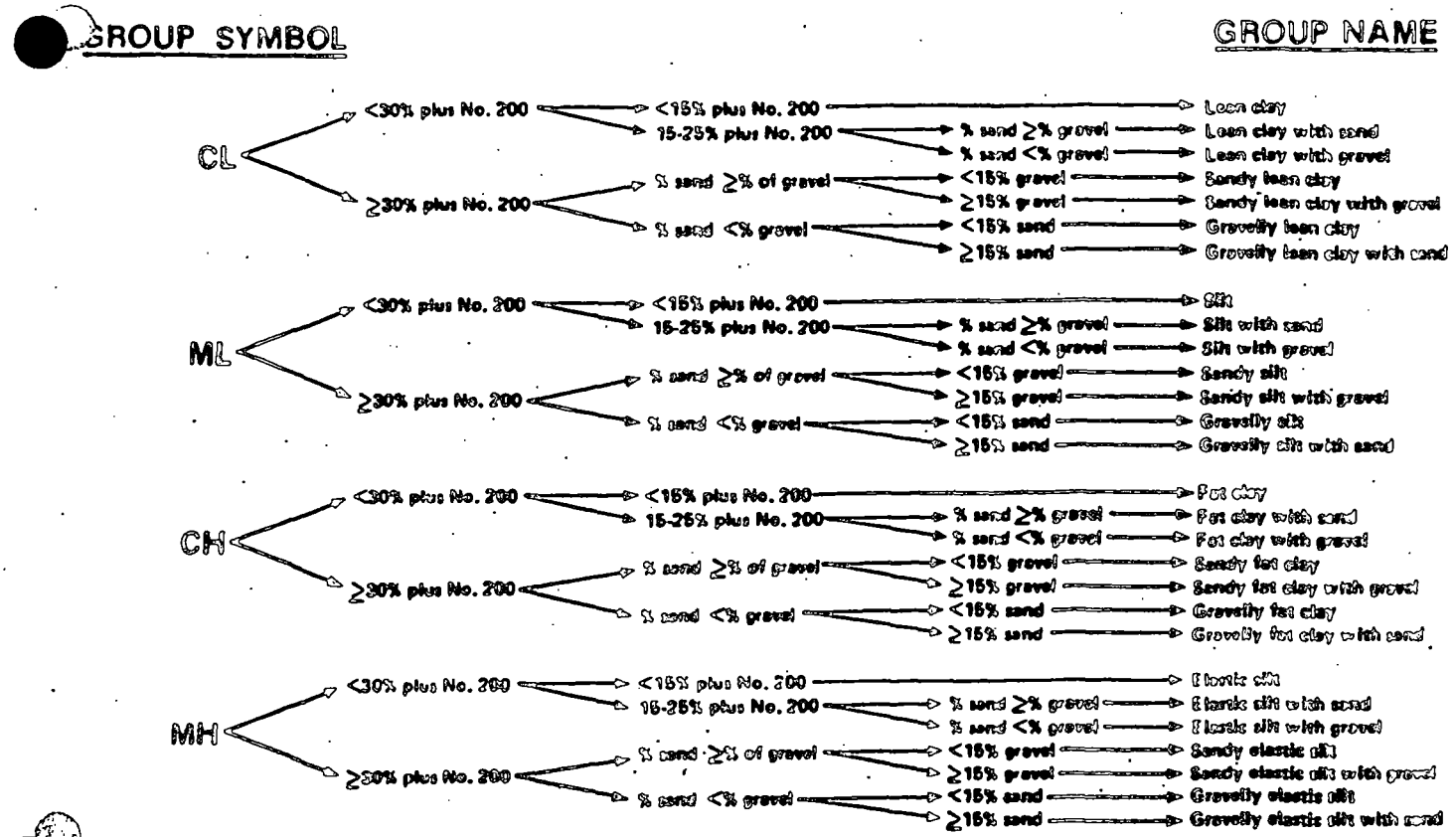
7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 *N*) to three parts water (This reagent is optional for use with this practice). See Section 8.

8. Safety Precautions

8.1 When preparing the dilute HCl solution of one part concentrated hydrochloric acid (10 *N*) to three parts of distilled

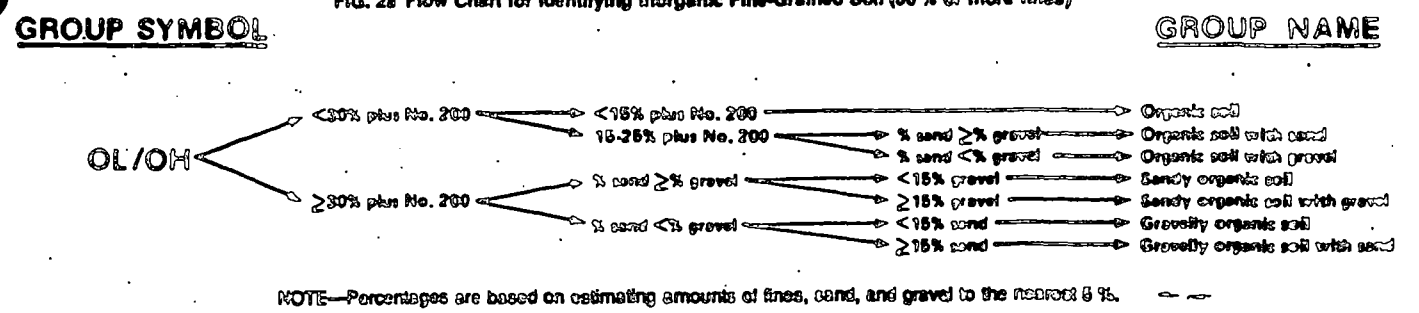
recycled paper

geology and environment



NOTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)



NOTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1 b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)

water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 **Caution**—Do not add water to acid.

9. Sampling

9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

NOTE 5—Preferably, the sampling procedure should be identified as having been conducted in accordance with Practices D 1452, D 1587, or D 2113, or Method D 1586.

9.2 The sample shall be carefully identified as to origin.

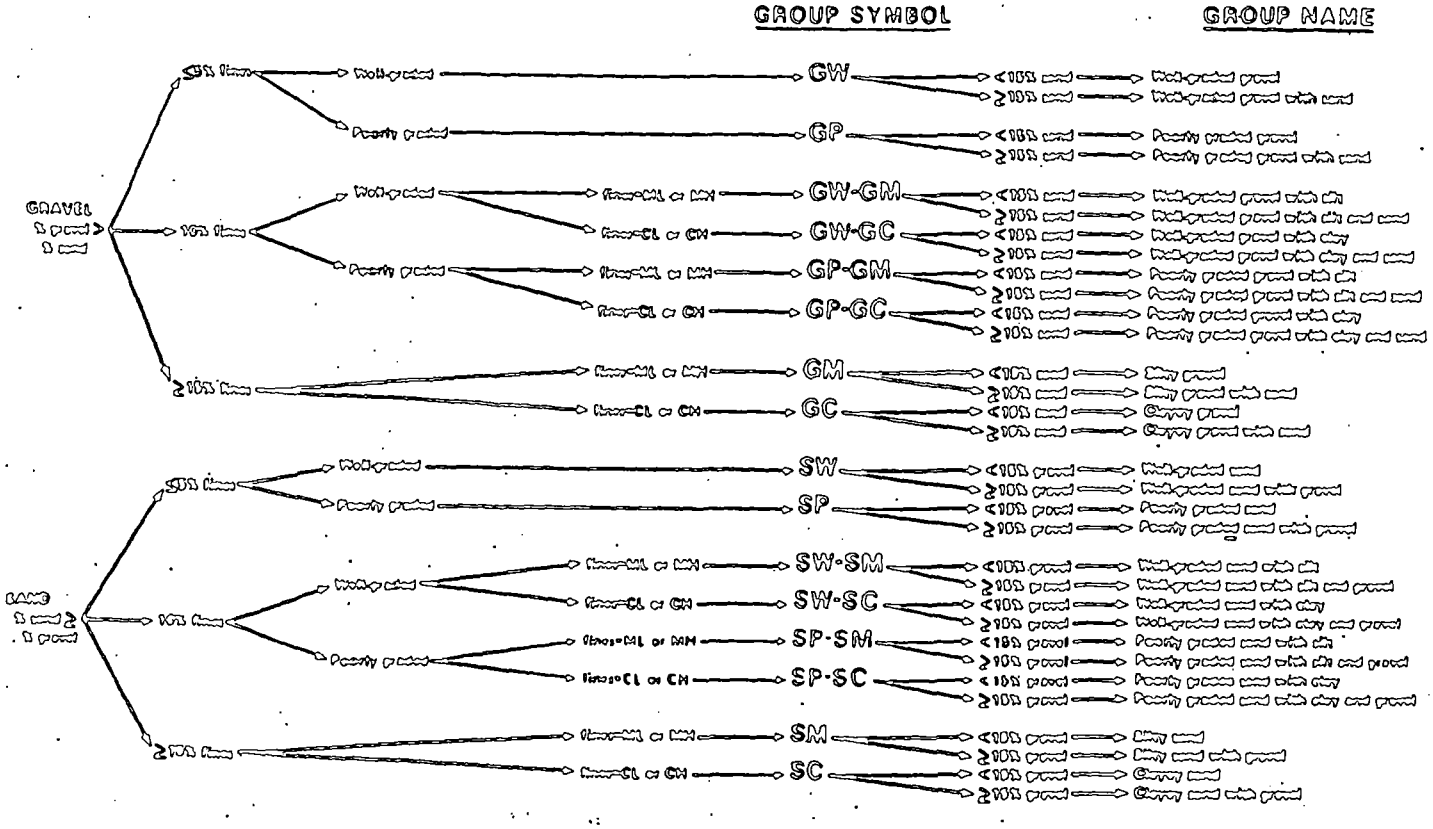
NOTE 6—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:

Maximum Particle Size, Sieve Opening	Minimum Specimen Size, Dry Weight
4.75 mm (No. 4)	100 g (0.25 lb)
9.5 mm (⅜ in.)	200 g (0.5 lb)
19.0 mm (¾ in.)	1.0 kg (2.2 lb)
38.1 mm (1½ in.)	6.0 kg (13 lb)
75.0 mm (3 in.)	60.0 kg (132 lb)

NOTE 7—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.
FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be noted in the report.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

Description	Criteria
angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
subangular	Particles are similar to angular description but have rounded edges
subrounded	Particles have nearly plane sides but have well-rounded corners and edges
rounded	Particles have smoothly curved sides and no edges

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the criteria in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

10.7 *Consistency*—For intact fine-grained soil, describe the consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

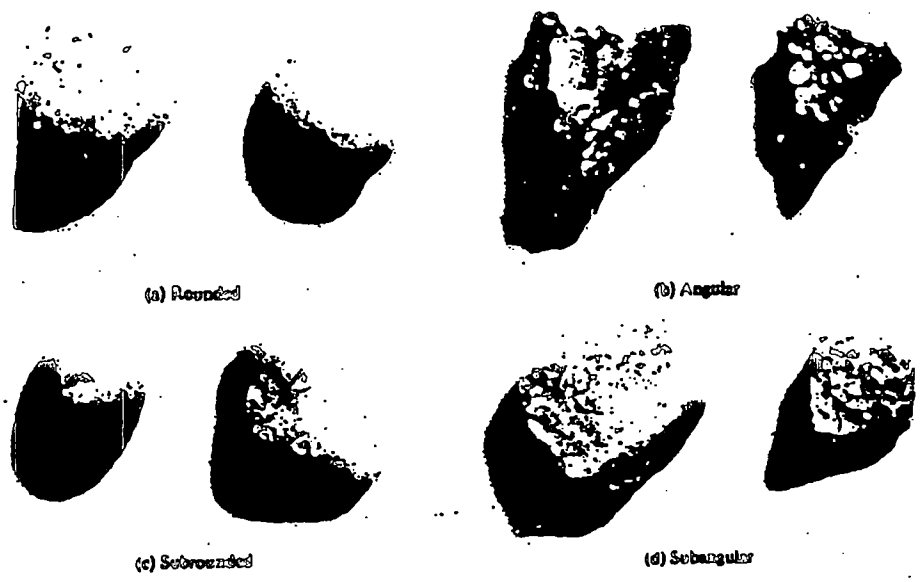


FIG. 3 Typical Angularity of Bulky Grains

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.

Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and elongated	Particles meet criteria for both flat and elongated

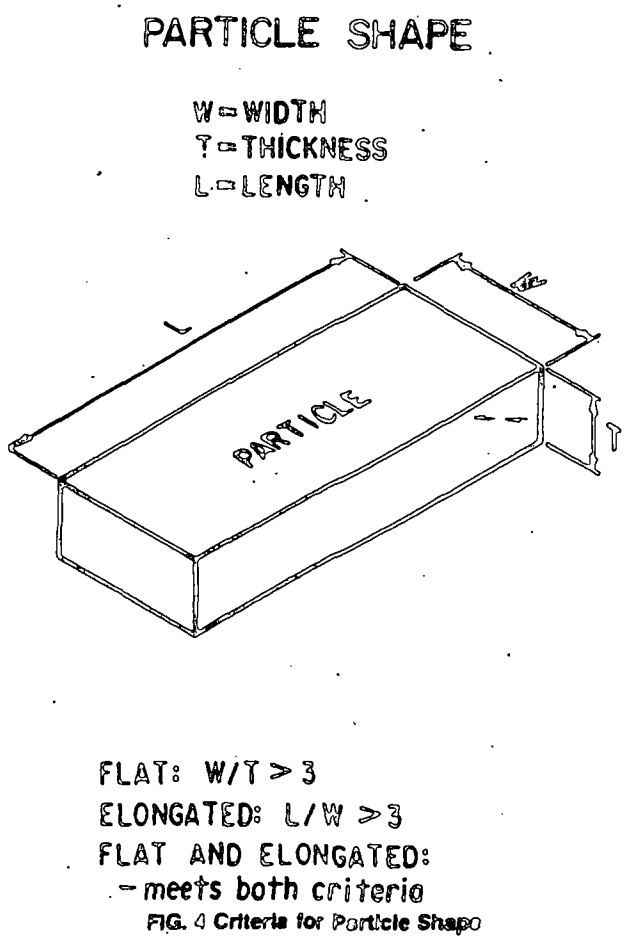


FIG. 4 Criteria for Particle Shape

10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.6. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maximum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the particles are hit by a hammer, for example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. "Hard" means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpretation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

TABLE 3 Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

TABLE 4 Criteria for Describing the Reaction With HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

TABLE 5 Criteria for Describing Dilatancy

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about ¼ in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very hard	Thumbnail will not indent soil

TABLE 6 Criteria for Describing Toughness

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Medium	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

TABLE 7 Criteria for Describing Dilatancy

Description	Criteria
rolled	Alternating layers of varying material or color with layers of least 6 mm (¼ in.) thick; note thickness
nated	Alternating layers of varying material or color with the layers less than 6 mm (¼ in.) thick; note thickness
ured	Breaks along definite planes of fracture with little resistance to fracturing
ickensided	Fracture planes appear polished or glassy, sometimes striated
ky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
ed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
ogeneous	Same color and appearance throughout

Identification of Peat

1.1 A sample composed primarily of vegetable tissue in ous stages of decomposition that has a fibrous to amor- s texture, usually a dark brown to black color, and an nic odor, shall be designated as a highly organic soil and be identified as peat, PT, and not subjected to the tification procedures described hereafter.

Preparation for Identification

.1 The soil identification portion of this practice is based e portion of the soil sample that will pass a 3-in. (75-mm) . The larger than 3-in. (75-mm) particles must be re- d, manually, for a loose sample, or mentally, for an intact le before classifying the soil.

2 Estimate and note the percentage of cobbles and the tage of boulders. Performed visually, these estimates e on the basis of volume percentage.

8—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 9—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be considered in the total of 100 % for the components.

13. Preliminary Identification.

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 *Dry Strength*:

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

NOTE 10—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accordance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

TABLE 8 Criteria for Describing Toughness

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very high	The dry specimen cannot be broken between the thumb and a hard surface

14.3 *Dilatancy*:

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 *Toughness*:

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

14.7 *Identification of Inorganic Fine-Grained Soils*:

TABLE 9 Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

TABLE 10 Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness

TABLE 11 Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A ⅛-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

NOTE 11—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

14.8 *Identification of Organic Fine-Grained Soils*:

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

NOTE 12—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

Soil Symbol	Dry Strength	Dilatancy	Toughness
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Medium to high	None to slow	Medium
MH	Low to medium	None to slow	Low to medium
CH	High to very high	None	High

clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words "with sand" or "with gravel" (whichever is more predominant) shall be added to the group name. For example: "lean clay with sand, CL" or "silt with gravel, ML" (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percentage of gravel, use "with sand."

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words "sandy" or "gravelly" shall be added to the group name. Add the word "sandy" if there appears to be more sand than gravel. Add the word "gravelly" if there appears to be more gravel than sand. For example: "sandy lean clay, CL", "gravelly fat clay, CH", or "sandy silt, ML" (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percent of gravel, use "sandy."

15. Procedure for Identifying Coarse-Grained Soils (Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is stimated to be more than the percentage of sand.

15.2 The soil is a *sand* if the percentage of gravel is stimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the ercentage of fines is estimated to be 5 % or less:

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *ell-graded sand*, SW, if it has a wide range of particle sizes d substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as *oorly graded sand*, SP, if it consists predominantly of one e (uniformly graded), or it has a wide range of sizes with ne intermediate sizes obviously missing (gap or skip ded).

5.4 The soil is either a *gravel with fines* or a *sand with fines* e percentage of fines is estimated to be 15 % or more.

5.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey l*, SC, if the fines are clayey as determined by the edures in Section 14.

.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, if the fines are silty as determined by the procedures in on 14.

5 If the soil is estimated to contain 10 % fines, give the dual identification using two group symbols.

5.1 The first group symbol shall correspond to a clean l or sand (GW, GP, SW, SP) and the second symbol shall pond to a gravel or sand with fines (GC, GM, SC, SM).

5.2 The group name shall correspond to the first group ol plus the words "with clay" or "with silt" to indicate the ity characteristics of the fines. For example: "well- l gravel with clay, GW-GC" or "poorly graded sand with P-SM" (see Fig. 2).

If the specimen is predominantly sand or gravel but s an estimated 15 % or more of the other coarse-grained ent, the words "with gravel" or "with sand" shall be o the group name. For example: "poorly graded gravel nd, GP" or "clayey sand with gravel, SC" (see Fig. 2).

If the field sample contains any cobbles or boulders, or words "with cobbles" or "with cobbles and boulders" shall be added to the group name. For example: "silty gravel with cobbles, GM."

16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

NOTE 13—*Example: Clayey Gravel with Sand and Cobbles, GC*—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

NOTE 14—Other examples of soil descriptions and identification are given in Appendix X1 and Appendix X2.

NOTE 15—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

Trace—Particles are present but estimated to be less than 5 %
Few—5 to 10 %
Little—15 to 25 %
Some—30 to 45 %
Mostly—50 to 100 %

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D 2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

17. Precision and Bias

17.1 This practice provides qualitative information only, therefore, a precision and bias statement is not applicable.

18. Keywords

18.1 classification; clay; gravel; organic soils; sand; silt; soil classification; soil description; visual classification

TABLE 13 Checklist for Description of Soils

1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
   Gravel—fine, coarse
   Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

For fined samples:

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, blocky, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

In-Place Conditions—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft^3; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size, coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incororated into a descriptive system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as "Sandy Lean Clay (CL)"; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; "Poorly Graded Sand with Silt (SP-SM)"; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—About 60 % gravel-size broken shells; about 30 % sand and sand-size shell pieces; about 10 % fines; "Poorly Graded Gravel with Sand (GP)."

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; "Poorly Graded Gravel (GP)"; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard, angular sand-size particles; dry, tan; no reaction with HCl.

X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL/SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP, SC/GC, GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP, SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML, CH/MH, SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH, MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay
ML/CL clayey silt
CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

X4.1 *Jar Method*—The relative percentage of coarse- and ne-grained material may be estimated by thoroughly shaking mixture of soil and water in a test tube or jar, and then lowing the mixture to settle. The coarse particles will fall to e bottom and successively finer particles will be deposited th increasing time; the sand sizes will fall out of suspension 20 to 30 s. The relative proportions can be estimated from : relative volume of each size separate. This method should correlated to particle-size laboratory determinations.

X4.2 *Visual Method*—Mentally visualize the gravel size ticles placed in a sack (or other container) or sacks. Then, the same with the sand size particles and the fines. Then, ntally compare the number of sacks to estimate the percent- of plus No. 4 sieve size and minus No. 4 sieve size present. The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

X4.3 *Wash Test (for relative percentages of sand and fines)*—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

X4.3.1 While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

X5. ABBREVIATED SOIL CLASSIFICATION SYMBOLS

5.1 In some cases, because of lack of space, an abbrevi- system may be useful to indicate the soil classification bol and name. Examples of such cases would be graphical , databases, tables, etc.

5.2 This abbreviated system is not a substitute for the full : and descriptive information but can be used in supple- ary presentations when the complete description is refer- l.

X5.3 The abbreviated system should consist of the soil classification symbol based on this standard with appropriate lower case letter prefixes and suffixes as:

Prefix	Suffix
s = sandy	s = with sand
g = gravelly	g = with gravel
	c = with cobbles
	b = with boulders

X5.4 The soil classification symbol is to be enclosed in

hesis. Some examples would be:

Group Symbol and Full Name	Abbreviated
CL, Sandy lean clay	s(CL)
SP-SM, Poorly graded sand with silt and gravel	(SP-SM)g
GP, poorly graded gravel with sand, cobbles, and boulders	(GP)ccb
ML, gravelly silt with sand and cobbles	g(ML)cc

X6. RATIONALE

Changes in this version from the previous version, D 2488 – 90, include the addition of X5 on Abbreviated Soil Classification Symbols.

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APPENDIX D

EPA METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed in Table 1 for soil and sediment samples. Some common elements are not listed in Table 1 because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). They are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed in Table 1 are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF.

1.2 Detection limits depend on several factors, the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. General instrument detection limits for analytes of interest in environmental applications are shown in Table 1. These detection limits apply to a clean matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (600-second) count times. These detection limits are given for guidance only and will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of field performance-based detection limits is presented in Section 13.4 of this method. The clean matrix and field performance-based detection limits should be used for general planning purposes, and a third detection limit discussed, based on the standard deviation around single measurements, should be used in assessing data quality. This detection limit is discussed in Sections 9.7 and 11.3.

1.3 Use of this method is restricted to personnel either trained and knowledgeable in the operation of an XRF instrument or under the supervision of a trained and knowledgeable individual. This method is a screening method to be used with confirmatory analysis using EPA-approved methods. This method's main strength is as a rapid field screening procedure. The method detection limits (MDL) of FPXRF are above the toxicity characteristic regulatory level for most RCRA analytes. If the precision, accuracy, and detection limits of FPXRF meet the data quality objectives (DQOs) of your project, then XRF is a fast, powerful, cost effective technology for site characterization.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use sealed radioisotope sources to irradiate samples with x-rays. X-ray tubes are used to irradiate samples in the laboratory and are beginning to be incorporated into field portable instruments. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This later process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples: the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α) or beta (β), which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.7 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments: in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly

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proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF: Field portable x-ray fluorescence.

3.2 MCA: Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS: Site specific calibration standard.

3.4 FP: Fundamental parameter.

3.5 ROI: Region of interest.

3.6 SRM: Standard reference material. A standard containing certified amounts of metals in soil or sediment.

3.7 eV: Electron Volt. A unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One and Chapter Three for additional definitions.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup, the analyte concentration measurement will be higher than if the fine particles are not

mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_β line of element Z-1 with the K_α line of element Z. This is called the K_α/K_β interference. Because the K_α:K_β intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_α and K_β energies are 4.95 and 5.43 keV, respectively, and the Cr K_α energy is 5.41 keV. The Fe K_α and K_β energies are 6.40 and 7.06 keV, respectively, and the Co K_α energy is 6.92 keV. The difference between the V K_β and Cr K_α energies is 20 eV, and the difference between the Fe K_β and the Co K_α energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

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4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_α/lead (Pb) L_α and sulfur (S) K_α/Pb M_α. In the As/Pb case, Pb can be measured from the Pb L_β line, and As can be measured from either the As K_α or the As K_β line; in this way the interference can be corrected. If the As K_β line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_α line. If the As K_α line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in no As being reported regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis by an EPA-approved method.

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as SW-846 Method 3050, or a total digestion procedure, such as Method 3052 is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project data quality objectives.

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method, the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r^2 often exceeding 0.95, except for barium and chromium. See Table 9 in Section 17.0). The critical factor is that the digestion procedure and analytical reference method used should meet the data quality objectives (DQOs) of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make

periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Section 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10 to 20°F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operators manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. Licenses for radioactive materials are of two types; (1) general license which is usually provided by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) specific license which is issued to named persons for the operation of radioactive instruments as required by local state agencies. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals. A copy of the radioactive material licenses and leak tests should be present with the instrument at all times and available to local and national authorities upon request. X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. Finally, an additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

5.2 Radiation monitoring equipment should be used with the handling of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs should be worn in the area of most frequent exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

5.3 Refer to Chapter Three for guidance on some proper safety protocols.

6.0 EQUIPMENT AND SUPPLIES

6.1 <u>FPXRF Spectrometer</u>: An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-

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ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation Sources: Most FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron (Fe)-55, cadmium (Cd)-109, americium (Am)-241, and curium (Cm)-244. These sources may be contained in a probe along with a window and the detector; the probe is connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum required for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of accelerating voltage is governed by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample Presentation Device: FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For most FPXRF instruments operated in the intrusive mode, the probe is rotated so that the window faces upward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors: The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon Si(Li). The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The Si(Li) detector must be cooled to at least -80 °C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a Si(Li) detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 liter. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2-270 eV; silicon pin diode-250 eV; Si(Li)–170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data Processing Units: The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in parts per million on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 100 to 500 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the units or from PCs. Once the data–storage memory of an FPXRF unit is full, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery chargers.

6.3 Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (µm) thick.

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6.5 Mortar and pestle: glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers: glass or plastic to store samples.

6.7 Sieves: 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels: for smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags: used for collection and homogenization of soil samples.

6.10 Drying oven: standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Pure Element Standards: Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if required for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.2 Site-specific Calibration Standards: Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.2.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of ten samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.2.2 Each sample should be oven-dried for 2 to 4 hours at a temperature of less than 150°C. If mercury is to be analyzed, a separate sample portion must remain undried, as heating may volatilize the mercury. When the sample is dry, all large, organic debris and nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be ground with a mortar and pestle and passed through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.2.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 grams of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 grams of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.3 Blank Samples: The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the method detection limits. These

samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.4 Standard Reference Materials: Standard reference materials (SRM) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, Inorganic Analytes.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance protocols. All field data sheets and quality control data should be maintained for reference or inspection.

9.2 Energy Calibration Check: To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting, which would indicate drift within the instrument. As discussed in Section 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (> 10 to 20°F).

The energy calibration check should be run at a frequency consistent with manufacturers recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.1 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak. The intensity and channel number of the pure element as measured using the radioactive source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank Samples: Two types of blank samples should be analyzed for FPXRF analysis: instrument blanks and method blanks. An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window.

9.3.1 The instrument blank can be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An

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instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the method detection limits should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. To be acceptable, a method blank must not contain any analyte at a concentration above its method detection limit. If an analyte's concentration exceeds its method detection limit, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration Verification Checks: A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ±20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision Measurements: The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent.

The equation for calculating RSD is as follows:

$$RSD = (SD/\text{Mean Concentration}) \times 100$$

where:

RSD	=	Relative standard deviation for the precision measurement for the analyte
SD	=	Standard deviation of the concentration for the analyte
Mean Concentration	=	Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the detection limit, but decreases sample throughput.

9.6 Detection Limits: Results for replicate analyses of a low-concentration sample, SSCS, or SRM can be used to generate an average site-specific method detection and quantitation limits. In this case, the method detection limit is defined as 3 times the standard deviation of the results for the low-concentration samples and the method quantitation limit is defined as 10 times the standard deviation of the same results. Another means of determining method detection and quantitation limits involves use of counting statistics. In FPXRF analysis, the standard deviation from counting statistics is defined as $SD = (N)^{\frac{1}{2}}$, where SD is the standard deviation for a target analyte peak and N is the net counts for the peak of the analyte of interest (i.e., gross counts minus background under the peak). Three times this standard deviation would be the method detection limit and 10 times this standard deviation would be the method quantitation limit. If both of the above mentioned approaches are used to calculate method detection limits, the larger of the standard deviations should be used to provide the more conservative detection limits.

This SD based detection limit criteria must be used by the operator to evaluate each measurement for its useability. A measurement above the average calculated or manufacturer's detection limit, but smaller than three times its associated SD, should not be used as a quantitative measurement. Conversely, if the measurement is below the average calculated or manufacturer's detection limit, but greater than three times its associated SD. It should be coded as an estimated value.

9.7 Confirmatory Samples: The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r^2) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r^2 is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

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10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Calibration: Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental Parameters Calibration: FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are required, although site-specific samples with confirmed and validated analytical results for all elements present could be used.
- Cost is reduced because fewer confirmatory laboratory results or calibration standards are required.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Section 7.2. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective Energy FP Calibration: The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the

line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_0 - C_t) / C_t) \times 100$$

where:

%D = Percent difference
C_t = Certified concentration of standard sample
C_0 = Measured concentration of standard sample

10.2.2 BFP Calibration: BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical Calibration: An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Section 7.2; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards

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by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is required. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are required to perform an adequate empirical calibration. The number of required standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton Normalization Method: The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline interference. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_α peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later on in analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, one requirement is that any large or nonrepresentative debris be removed from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Another requirement is that the soil surface be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide data for this method, this modest amount of sample preparation was found to take less than 5 minutes per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for *in situ* analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on required detection limits.

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11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 grams or 250 cm^3, which is enough soil to fill an 8-ounce jar. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Section 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the homogenization procedure using the fluorescein dye required 3 to 5 minutes per sample. As demonstrated in Sections 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, it can be used without the more labor intensive steps of drying, grinding, and sieving given in Sections 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps must be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 grams) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hours in the convection or toaster oven at a temperature not greater than 150°C. Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 minutes per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle, and sieves must be thoroughly cleaned so that any cross-contamination is below the MDLs of the procedure or DQOs of the analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in parts per million and can be downloaded to a PC, which can provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation.

13.0 METHOD PERFORMANCE

13.1 This section discusses four performance factors, field-based method detection limits, precision, accuracy, and comparability to EPA-approved methods. The numbers presented in Tables 4 through 9 were generated from data obtained from six FPXRF instruments. The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States.

The soil samples contained several of the target analytes at concentrations ranging from nondetect to tens of thousands of mg/kg.

13.2 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.3 All data presented in Tables 4 through 9 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.4 Field-Based Method Detection Limits: The field-based method detection limits are presented in Table 4. The field-based method detection limits were determined by collecting ten replicate measurements on site-specific soil samples with metals concentrations 2 to 5 times the expected method detection limits. Based on these ten replicate measurements, a standard deviation on the replicate analysis was calculated. The method detection limits presented in Table 4 are defined as 3 times the standard deviation for each analyte.

The field-based method detection limits were generated by using the count times discussed earlier in this section. All the field-based method detection limits were calculated for soil samples that had been dried and ground and placed in a sample cup with the exception of the MAP Spectrum Analyzer. This instrument can only be operated in the in situ mode, meaning the samples were moist and not ground.

Some of the analytes such as cadmium, mercury, silver, selenium, and thorium were not detected or only detected at very low concentrations such that a field-based method detection limit could not be determined. These analytes are not presented in Table 4. Other analytes such as calcium, iron, potassium, and titanium were only found at high concentrations (thousands of mg/kg) so that reasonable method detection limits could not be calculated. These analytes also are not presented in Table 4.

13.5 Precision Measurements: The precision data is presented in Table 5. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from nondetects to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a

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sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 5 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the MDL for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 5. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the MDLs so that an RSD value calculated at 5 to 10 times the MDL was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 6 shows these results. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the detection limit of the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the *in situ* homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 6 shows that the precision dramatically improved from the *in situ* to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square, measurements of different soil samples were actually taking place within the square. Table 6 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five versus ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy Measurements: Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 7 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 7 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 7. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 7.

Table 8 provides a more detailed summary of accuracy data for one FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. Table 8 shows the certified value, measured

value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability: Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 9. Similar trends in the data were seen for all instruments.

Table 9 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. The soil types are as follows: soil 1—sand; soil 2—loam; and soil 3—silty clay. The preparation methods are as follows: preparation 1—in situ in the field; preparation 2—in situ, sample collected and homogenized; preparation 3—intrusive, with sample in a sample cup but sample still wet and not ground; and preparation 4—sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.80 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 9 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 9 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Section 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time required to dry and grind the sample for small improvements in comparability.

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Homogenization requires 3 to 5 minutes. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 minutes per sample. Lastly, when grinding and sieving is conducted, time must be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 Hewitt, A.D. 1994. "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis." *American Environmental Laboratory*. Pages 24-32.

13.8.2 Piorek, S., and J.R. Pasmore. 1993. "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer." *Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals*. Las Vegas, Nevada. February 24-26, 1993. Volume 2, Pages 1135-1151.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex. X-MET 920 User's Manual.

2. Spectrace Instruments. 1994. Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction.

3. TN Spectrace. Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.

4. Unpublished SITE data, recieved from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The pages to follow contain Tables 1 through 9 and a method procedure flow diagram.

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TABLE 1
INTERFERENCE FREE DETECTION LIMITS

Analyte	Chemical Abstract Series Number	Detection Limit in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: References 1, 2, and 3

TABLE 2
SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	458	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Reference 1, 2, and 3

TABLE 3
SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Reference 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

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TABLE 4
FIELD-BASED METHOD DETECTION LIMITS (mg/kg)[o]

Analyte	Instrument					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	55	NR	NR	NR	NR	NR
Arsenic	60	50	55	50	110	225
Barium	60	NR	30	400	NR	NR
Chromium	200	460	210	110	900	NR
Cobalt	330	NR	NR	NR	NR	NR
Copper	85	115	75	100	125	525
Lead	45	40	45	100	75	165
Manganese	240	340	NR	NR	NR	NR
Molybdenum	25	NR	NR	NR	30	NR
Nickel	100	NR	NA	NA	NA	NR
Rubidium	30	NR	NR	NR	45	NR
Strontium	35	NR	NR	NR	40	NR
Tin	85	NR	NR	NR	NR	NR
Zinc	80	95	70	NA	110	NA
Zirconium	40	NR	NR	NR	25	NR

Source: Reference 4

o MDLs are related to the total number of counts taken. See Section 13.3 for count times used to generate this table.

NR Not reported.

NA Not applicable; analyte was reported but was not at high enough concentrations for method detection limit to be determined.

PRECISION

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the MDL					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84°	NR	24.60°	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85°	NR	24.92°	20.92°	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.68°	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32°	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

Source: Reference 4

° These values are biased high because the concentration of these analytes in the soil samples was near the detection limit for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the method detection limit.

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TABLE 6
PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive-Undried and Unground	Intrusive-Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium[a]	41.2	30.6	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel[a]	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver[a]	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.75
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

Source: Reference 4

[a] These values may be biased high because the concentration of these analytes in the soil samples was near the detection limit.
ND Not detected.
NR Not reported.

TABLE 7
ACCURACY

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	–	–	–	–	–	–	–	–	–	–	–	–
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	–	–	–	–	9	18-848	168.2	262	–	–	–	–
Cd	2	99-129	114.3	NA	–	–	–	–	6	81-202	110.5	45.7	–	–	–	–
Cr	2	99-178	138.4	NA	–	–	–	–	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	78.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	88-159	102.3	28.6	6	48-84	80.4	16.2	6	28-187	108.6	52.9
Pb	11	68-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.8	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	–	–	–	–	–	–	–	–
Ni	3	88-122	109.8	12.0	–	–	–	–	–	–	–	–	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	–	–	–	–	–	–	–	–	7	88-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	103.6	34.7	11	31-199	94.6	42.5

Source: Reference 4

n Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.
SD Standard deviation.
NA Not applicable; only two data points, therefore, a SD was not calculated.
%Rec. Percent recovery.
– No data.

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TABLE
ACCURACY FOR TN 9000°

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	–	–	–	–	–	–	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	–	–	–	–	–	–	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	–	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	–	–	–	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	–	–	–	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Reference 4

° All concentrations in milligrams per kilogram.
%Rec. Percent recovery.
ND Not detected.
NA Not applicable.
– No data.

TABLE 9

REGRESSION PARAMETERS FOR COMPARABILITY[1]

	Arsenic				Barium				Copper			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	288	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

Source: Reference 4

1 Log-transformed data
n Number of data points
r^2 Coefficient of determination
Int. Y-intercept
— No applicable data

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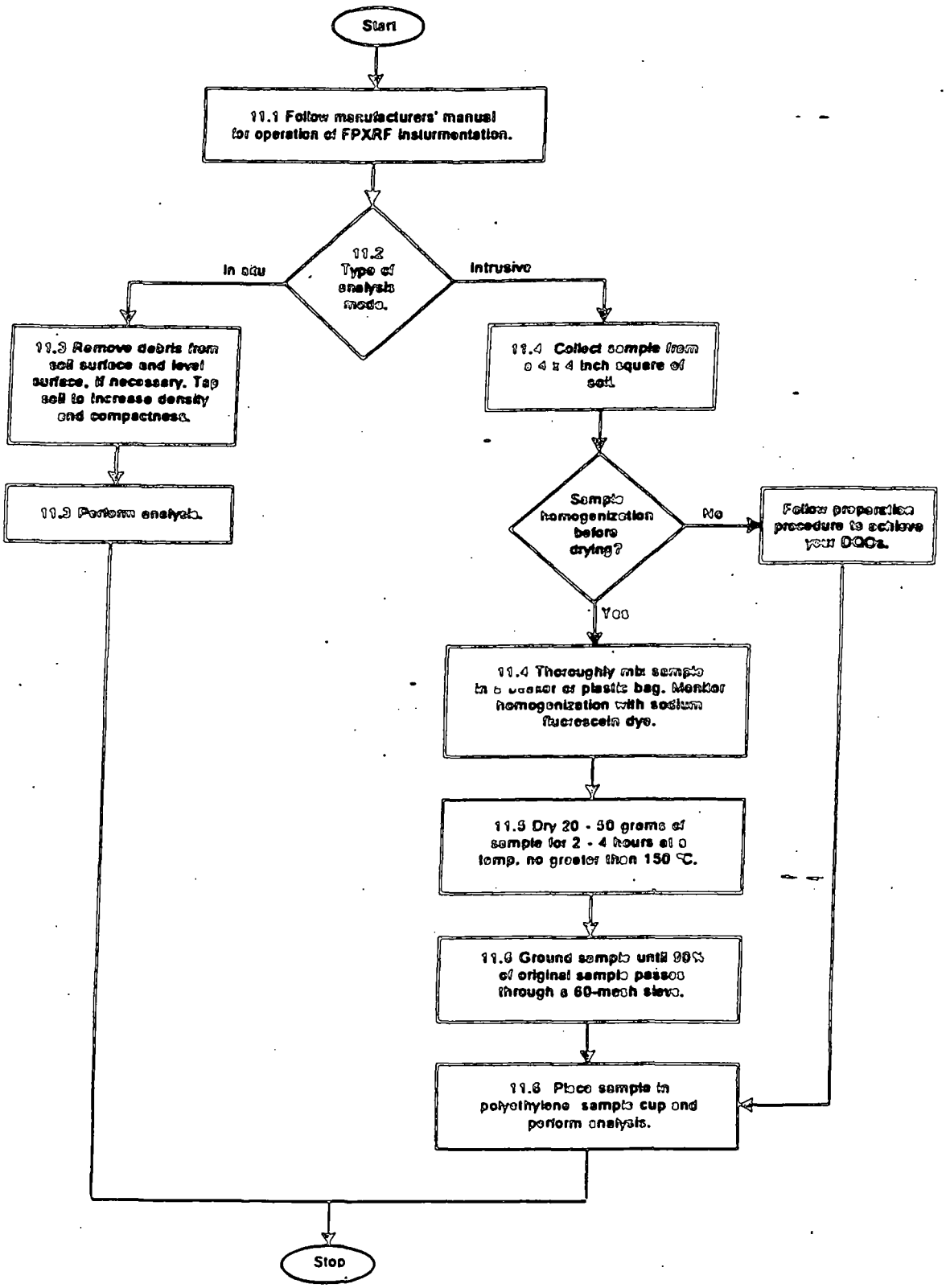
FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT



APPENDIX E

REGION 10 START-2 STANDARD OPERATING PROCEDURE FOR X-RAY FLUORESCENCE FIELD SCREENING

REGION 10
SUPERFUND TECHNICAL ASSESSMENT AND RESPONSE TEAM TWO
(START-2)

STANDARD OPERATING PROCEDURE
FOR

X-RAY FLUORESCENCE
FIELD SCREENING

May 2001

Prepared by

Region 10 START-2
Ecology and Environment, Inc.
Seattle, Washington

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1.0 INTRODUCTION

X-ray fluorescence (XRF) has proven itself to be a cost-effective and time-saving technique for metals screening and analysis on environmental sites. The Removal Program within Superfund has especially benefitted from XRF because of its ability to provide immediate information on contaminants, and its low per-unit cost of analysis. Contaminant assessment error is reduced by increasing sampling density rather than increasing the precision of the analytical method. One of the greatest advantages to XRF field screening is its ability to direct on-going removal activities, which reduces costly manpower/equipment down-time and more accurately defines the area of contamination.

This document outlines recommended and required procedures and equipment for representative collection and/or analysis of samples for XRF field screening.

2.0 METHOD SUMMARY

XRF field screening situations vary widely and therefore no universal analytical procedure can be recommended. Likewise, XRF instruments vary in technology and capability. The Sampling Plan/ Quality Assurance Project Plan should address these variables based on site characteristics and required data quality objectives.

In general, a non-fundamental parameters instrument is calibrated with site-specific samples of known concentrations (EPA CLP analysis or equivalent). If a fundamental parameters instrument is used, matrix-specific conditions are optimized. Following calibration or optimization, the sample is collected and prepared. Sample preparation of in-situ analysis may only consist of removing surface debris and placing the probe on the surface to be analyzed. However, if more precision is required to address the site data quality objectives, samples may need to be sieved, dried, crushed, and placed into x-ray cells prior to analysis.

Samples are then analyzed by exposing the matrix to the selected source for no less than 30 seconds. The instrument displays the concentration of the selected elements in the units in which they were calibrated. Depending on the application and data quality objectives, a variety of quality assurance measures are taken to ensure data validity. Additionally, a portion of the samples are sent to a laboratory or analyzed by a second fundamental parameters instrument for confirmation of results.

3.0 INSTRUMENT CALIBRATION

3.1 Site-Specific Empirical Calibration

The following instrument calibration criteria apply to non-fundamental parameters instruments incorporating empirical calibrations. The instrument should be calibrated according to vendor specifications, and the following considerations.

- Pure elements should be re-analyzed prior to each calibration.
- A minimum of 10 site-specific samples must be used for the calibration.
- The sample concentrations of site-specific samples used for the calibration should range the concentrations of interest. Several samples from the calibration suite should be at or near the decisive action level.
- The gain channel should be monitored and recorded during the initial calibration.
- Field in-situ analysis time versus the measurement time used during initial calibration is not considered critical and may vary depending on the application.
- A minimum r^2 value of 0.70 is required for an acceptable initial calibration. No criteria are established for deleting points from the initial calibration. The benefit of maximizing the r^2 value versus the detriment of reducing calibration population and eliminating some potential real matrix effects must be weighed.

3.2 Fundamental Parameters Performance Check

The performance of a fundamental parameters software-driven instrument will be monitored by analyzing a National Institute of Standards and Technology (NIST) primary standard. Available standards include Standard Reference Material (SRM) 2709, 2710, and 2711 (low, medium, and high concentration metal concentrations in environmental samples matrix). SRMs of similar concentrations to the samples will be analyzed prior to analysis, and during sample analysis at a frequency of at least every ten samples. Acceptable results will be within ± 20% of the certified values.

A site-specific sample characterized to at least 99.5% elemental constituency by a high-resolution (less than 100 KeV Mn Kα) fundamental parameters instrument is an acceptable substitute for NIST SRMs, provided that the concentrations of the analytes of interest are near the area of interest, or action level.

4.0 SAMPLE PREPARATION

Because of the impromptu and diverse nature of work performed by the Removal Program, every effort has been made to preserve the project manager's freedom to vary the degree of sample preparation to meet particular site specific data quality objectives or time/budget constraints. It should be noted that XRF field screening results can be dramatically improved if samples are dried, sieved, and crushed prior to analysis. No matter the degree of sample preparation, the samples from the site should be analyzed in the same manner as the samples analyzed during the empirical calibration.

4.1 Mandatory Sample Preparation Measures:

- Interfering surface debris will be removed for in-situ XRF measurements.
- A minimum measurement time of 30 seconds will be used.
- A single thickness plastic bag should be used as a protective layer between probe and sample for in-situ measurements. Instrument calibration should be performed with the same material between the sample and probe.
- Samples used for confirmation will be collected and homogenized as a minimum prior to XRF and laboratory analysis.

4.2 Optional Sample Preparation Measures:

- A minimum of three measurements should be taken and averaged for every grid point or measurement node to minimize error caused by microheterogeneity (nugget effect).
- Microwave drying of samples is acceptable (excluding mercury analysis).
- Sample preparation with a 10-mesh sieve will provide more precise results, but is left to the discretion of the analyst. Sieving samples must be consistent with the calibration.
- The prepared portion of the sample analyzed by XRF should be sent for laboratory confirmation to reduce the effects of microheterogeneity.

5.0 QUALITY ASSURANCE

5.1 Quality Assurance Requirements (EPA 540-R-93-071, Data Quality Objective Process for Superfund)

Screening Data (Definitive Data Without Error and Bias Determination) objectives are met by XRF analysis if either fundamental parameters modeling is used, or if the Kα and Kβ peak, or the Lα and Lβ peaks are positively identified for the element in question. Ten percent of the samples must be sent for independent laboratory confirmation to meet Definitive Data criteria. On larger sites, once calibration confirmation has been established, the number of samples sent for confirmation may taper off to 5%.

Definitive Data criteria are met if in addition to meeting the Definitive Data objectives, error analysis is determined per matrix by analyzing 8 samples in replicate, and variance is calculated.

5.2 PARCC Parameter Criteria

- Precision will be established by measuring an action-level concentration standard and a low concentration standard at a frequency of 10% of all field measurements. An acceptable level of ±20% Relative Percent Difference (RPD) from the XRF value after initial calibration (not laboratory assay value) was established. If this control limit is exceeded, all samples from the last acceptable measurement must be reanalyzed. The low concentration sample would be used to establish the detection limit and the analytical quantitation limit as defined by 3 times the standard deviation of this measurement.

- Accuracy will initially be established by sending high, medium and low concentration field samples for verification analysis. Following this initial accuracy check, calibration accuracy will be monitored by sending 10% of the project samples for laboratory confirmation.

- Representativeness will heavily depend on project-specific data quality objectives and will not be monitored directly. It should be noted in the project-specific quality assurance plan that representativeness can be increased by reducing the geostatistical error associated with sample point density.

- Comparable data will be generated if the continuing calibration results are within ±20% RPD. It is critical to note that XRF results will be comparable to the method used in assaying the initial calibration samples. For most purposes, samples will be analyzed by atomic absorption spectroscopy or inductively coupled plasma emission spectroscopy following the EPA 3050 nitric acid/peroxide digestion, and the results by XRF are more representative of the digestion procedure than the actual analytical method.

- Completeness will be established by a simple percentage of the number of measurements taken compared to the number of measurements planned.

5.3 Replicate Sample Analysis

Eight replicates of one sample are to be collected and analyzed whenever error determination is required. No duplicate sample analysis (2 replicates) is required.

5.4 Performance Evaluation Samples

Performance Evaluation (PE) sample analysis is appropriate for fundamental parameter instrumentation. However, because empirical calibration models are highly matrix dependent, PE sample analysis is not suggested.

6.0 DATA VALIDATION

Throughout sample analysis, a mid-range or action level concentration standard will be analyzed at a frequency of 10%. This analysis will serve as a continuing calibration check and results must be within ±20% of the true sample value, as determined by the appropriate EPA CLP-style analysis. If sample results from this analysis lie outside the ±20% control limit, all samples from the last successful continuing calibration check must be re-analyzed following adjustment of the appropriate instrument parameters.

In addition, a low concentration standard will be analyzed at the same frequency. From the standard deviation of this measurement, the detection limit and analytical quantitation limit will be calculated as defined by 3 times the standard deviation of this low concentration standard.

Following the field project, a follow-up data summary report will be written evaluating XRF conformance to standard operating procedures and appropriate quality control criteria. Additionally, from laboratory confirmation analyses and XRF results, a correlation coefficient will be calculated. Data sets above the upper calibration range may be eliminated due to model bias (heteroscidasticity), unless an even distribution is apparent. Data pairs below XRF and analytical detection limit will be left to the discretion of the reviewer. A correlation coefficient of 0.70 or greater must be obtained for data to be considered acceptable.

7.0 HEALTH AND SAFETY

All field-portable XRF units either incorporate radioactive sources, or X-ray tubes. All site-specific health and safety precautions as outlined in the site safety plan must be adhered to when performing XRF field screening analyses.

In addition to site chemical hazards is the consideration of radiological activity of the source(s). A current wipe test certification should accompany the instrument, dated no more than 6 months from the current date. A real-time monitoring instrument should be used periodically to monitor the instrument with the shutter closed to ensure adequate shielding. Site personnel will not be allowed to operate XRF instrumentation unless they are equipped with a dosimetry monitoring device (e.g., TLD badge, pocket dosimeter).

Prior to the removal of the instrument from the hot zone, or from the site, the instrument should be thoroughly decontaminated with a mild solution of soap and water.

8.0 POTENTIAL SOURCES OF ERROR

The following are recognized sources of error when using XRF for field screening applications. Some sources of error are unavoidable, or may cause error within acceptable limits as established by the data quality objectives. Although they will not be discussed in detail, the user should be aware of these error sources.

- Sample matrix causes the greatest potential for error during analysis, especially with instruments with high pressure gas proportional detectors (resolution greater than 800 eV). Causes of error include factors such as chemical, geomorphology, surface texture, particle size, density (average Z number), microheterogeneity, and moisture content.

- High energy sources and radio frequency generators may cause software and detector interferences. Interfering sources may include site radios and video monitors.

- Certain unavoidable site characteristics are inherent for error in XRF analysis such as particle size, temperature, humidity, and geomorphology.

- Chemical interferences may lead to increased error especially with proportional gas detectors. More common chemical interference may include Pb and As, Fe with Cr and Ni, Pb with Bi and Rb.

- Instrumental factors may lead to error such as battery loss, gain control, drift, and temperature fluctuations.

APPENDIX F

NITON CORPORATION USER'S GUIDE VERSION 5.0, CHAPTERS TWO AND THREE

NITON Corporation

XL-309

&

*700*series

User's Guide Version 5.0 (HTML) Chapter 2

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Chapter 2: Operating your NITON

NITON XL-309 and 700 Series Spectrum Analyzers are hand-held, portable XRF detectors, designed to make fast, accurate measurements. The XL-309 measures concentrations of lead, while 700 Series instruments measure concentrations of many different elements simultaneously. NITON instruments measure the precision of each reading, store up to 3,000 readings with complete x-ray spectra, and download data quickly to a PC.

NITON designed the radioactive source and shielding of our analyzers with one guiding principle in mind: properly used, these will not expose the NITON user to levels of radiation significantly above natural background levels.

Note: The accuracy and precision of the data you collect with your NITON XRF will largely depend on your familiarity with the instrument and your knowledge of the media you are testing.

Our free factory training is designed to give you the basic tools to use our instruments. This User Guide supplements our training. You can use it as both a quick reference and a detailed operating manual for any of our XRF analyzers.

This is your NITON XRF Spectrum Analyzer

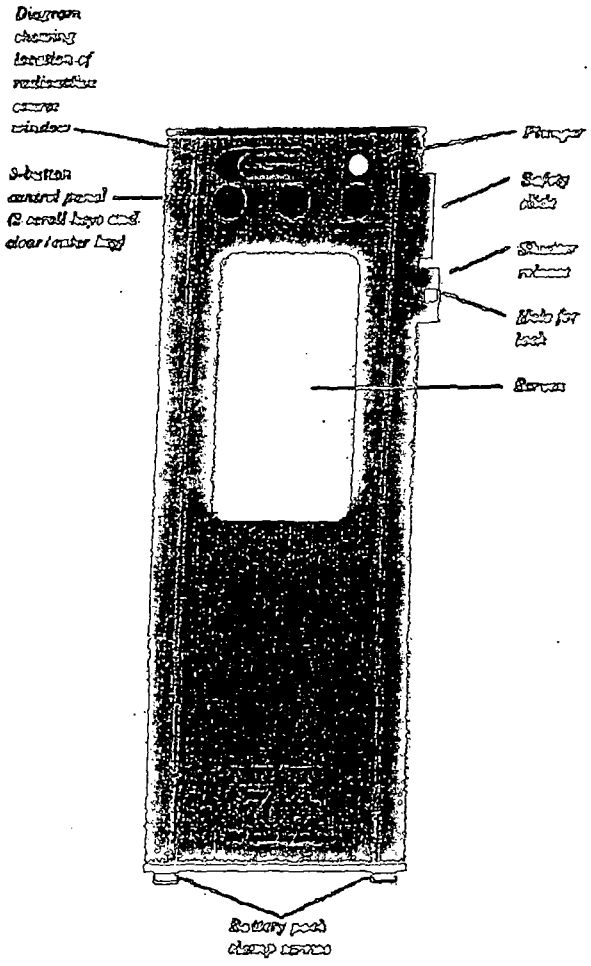
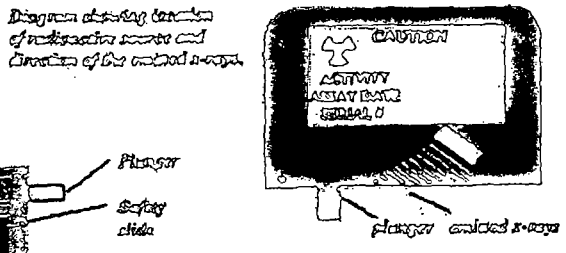


Fig. 2.01 Front view of the NITON 700.

Fig. 2.02
Top view of the your NITON.



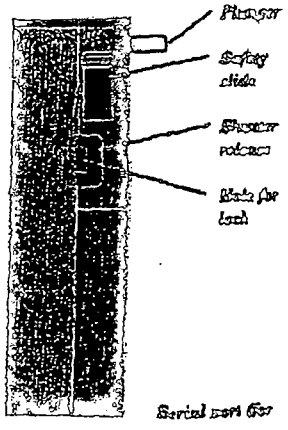


Fig. 2.03
Right side view of your NITON.

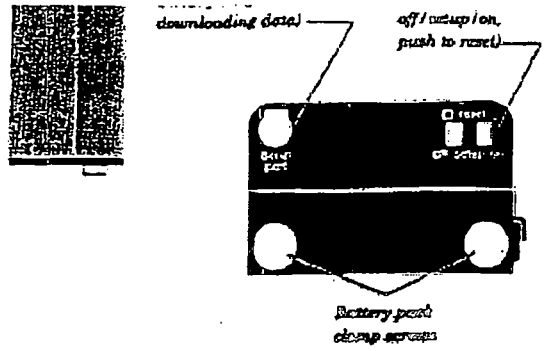


Fig. 2.04
Back view of your NITON

NITON Spectrum Analyzers operate in the following modes:

Modes of operation, by model

Model	Bulk Mode	Thin Sample Mode	Paint Modes
701	No	Yes	No
701-A	No	Yes	Yes
702	Yes	No	No
702-A	Yes	No	Yes
703	Yes	Yes	No
703-A	Yes	Yes	Yes
XL-309	Opt (lead only)	Opt (lead only)	Yes

Turning on your NITON

1. Turn on the instrument. Depress and slide the On/Off switch on the bottom of the instrument to the on position (Figure 2.04). Sometimes the instrument's battery saving features momentarily delay start up. If your NITON does not turn on immediately, turn it off, wait a few seconds, and turn it on again. Each time the NITON is turned on, the Main menu appears (Figure 2.05).

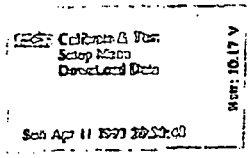


Fig. 2.05 Main Menu

2. The control panel consists of three buttons (Figure 2.06). These buttons allow you to navigate all of your NITON's screens and menus. Press the Clear/Enter button to *select* the function indicated by the screen arrow. When you turn on your NITON, the Screen arrow is on

Calibrate & test.

Note: You can begin to test <u>immediately</u> in whatever mode you *last* tested in by pressing the Clear/Enter button.

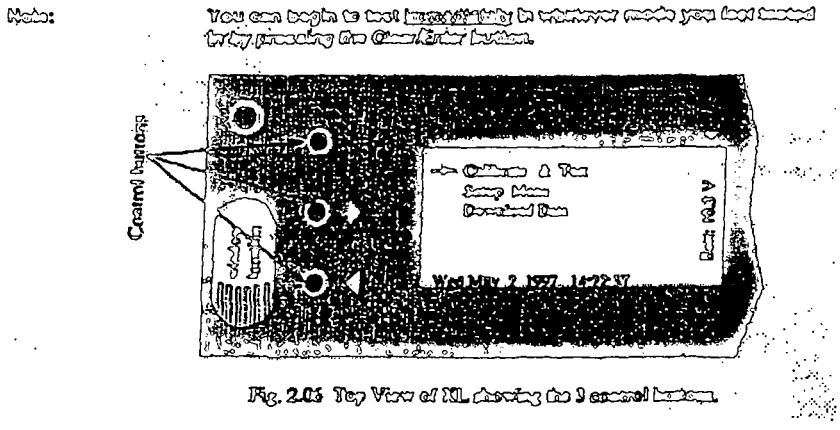


Fig. 2.06 Top View of XL showing the 3 control buttons.

Getting started

The XL-309 and 700 Series Instruments are highly sophisticated, electronic spectrum analyzers. The more familiar you are with your NITON's operation, the better your measurements and reports will be. Here, in brief, is an outline of how to do various kinds of testing using your NITON. More detailed information is offered in subsequent chapters.

1 Turn on the instrument. When testing in Bulk Sample or

1. Turn on the instrument. When testing in Bulk Sample or Thin Sample modes, leave your NITON on for fifteen minutes prior to testing. *This is not necessary if you are going to test in any of the Paint Modes.* Go to the Setup Menu (Figure 2.07) and set the .mode you wish to test in.

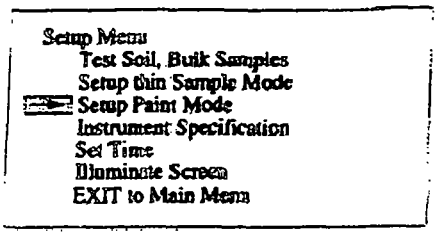


Fig. 2.07 Setup Menu

2. Press Clear/Enter to begin self-calibration.

3. When the NITON beeps, calibration is complete. You are now ready to test. For instructions on how to take a measurement, depending on the nature of the media you will be measuring, turn to one of the following chapters: Chapter 3: Analyzing Bulk Samples; Chapter 4: Analyzing Thin Samples; or Chapter 5: Analyzing Lead Paint.

Note: Check your instrument's calibration with testing standards before and after testing and at least once per hour during testing.

The Setup Menu

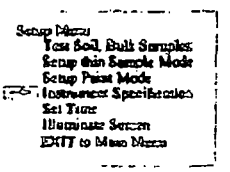


Fig. 2.05 Setup Menu

Press Clear/enter to bring up the Instrument Specification screen.

Use the Setup Menu (Figure 2.08) to check your instrument specification: to set the date and time: to illuminate the screen

continously; or to select a different testing mode. Select the Setup Menu from the Main Menu with the Arrow buttons; enter the Setup Menu by pressing Clear/Enter.

Instrument Specification

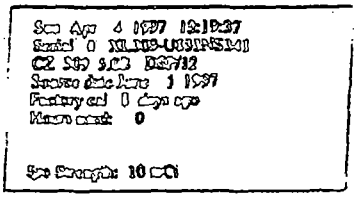


Fig. 2.09 Instrument Specifications

To check the source strength of your instrument and other useful information, select the Instrument Specification screen (Figure 2.09) from the Setup Menu with the Arrow buttons. Press Clear/Enter. The screen displays the following information:

1. The Day, Month, Date, Year and Time (hours, minutes and seconds).

2. The Instrument Serial Number

3. The instrument Model; and the versions of Firmware and DSP software installed on the instrument.

4. The Source Date, the assay date of the cadmium$_{109}$ source.

5. The number of days since the last factory calibration of the instrument.

6. The Hours used, the number of hours the instrument has been used since the last factory calibration.

7. The Source Strength, the current strength of the

instrument's cadmium$_{109}$ source, in millicuries (mCi).

To exit the Instrument Specification screen to the Main Menu, press the Clear/Enter button.

Setting the time and date on your NITON

NITON sets the date and time (EST) on each instrument before it is shipped. Reset as needed when changing time zones, daylight savings time begins and ends, or whenever the time or date is wrong.

Caution: Check the Date and Time displayed on the Ready to Test screen. If they are not correct, reset them <u>before</u> taking any measurements. Your readings will not be accurate unless the date and time are correct.

To reset the date and time from the Setup Menu, do the following steps:

1. Use the the Arrow buttons to scroll to Set Time (Figure 2.10 a,b).

2. Press Clear/Enter to select it. The Date and Time appear as follows:

Month-Day-Year-Hour-Minute-Second

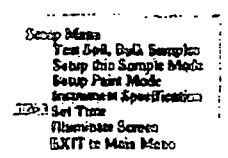


Fig. 2.10a Setup Menu Set Time

Press Clear/Enter to bring up the Set Time screen.

Sat Apr 3 1997 13:31:44

Fig. 2.10b Set Time screen

Month-Day-Year-Hour-Minute-Second

The cursor starts at Month and moves to the right. To change the time and date, move from left to right on the screen. For example, To change the hour and seconds:

1. Press Clear/Enter three times to move the cursor to Hour.

2. Use the Arrow buttons to change the hour to the desired hour. Press Clear/Enter.

3. The cursor automatically moves to the next field: Minute. Use the Arrow buttons to change the minutes to the desired minutes. Press Clear/Enter again to move the cursor to Second.

4. Use the Arrow buttons to change the seconds to the desired seconds. Press Clear/Enter.

5. After selecting Seconds, the Main Menu screen is again displayed, set to Calibrate & Test.

Note: If the year is incorrect, set it first. Use Clear/Enter to move to the year position and the Arrow buttons to set the year. Then press Clear/Enter *five* more times and set the remaining fields as described above.

Lighting the LCD screen

In its default mode, your instrument's LCD screen remains back-lit for 15 seconds after any of the three buttons is pressed. You can light the screen any time the instrument is turned on by pressing any of the three buttons. When working in a dark place, you also have the option of lighting the screen continuously.

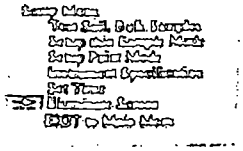


Fig. 2.11 Setup Menu
Illuminate Screen

Take the following steps to either light the screen continuously, or turn off continuous screen lighting if it is currently activated:

1. Use the Arrow buttons to select Illuminate Screen from the Setup Menu (Figure 2.11).

2. Press the Clear/Enter button to turn continous screen lighting on or off. The instrument will then return automatically to the Main Menu.

Overview of test modes

The Setup Menu allows you to choose the pre-programmed test mode best suited for the type of testing that you will be doing. A full chapter is devoted to each mode later in this User's Guide.

Note: The Setup Menu shows all NITON analyzer modes for all instruments. If you select a test mode which is not available on your NITON instrument, a reminder message will be displayed on the sceen.

Please contact NITON instrument sales at (800) 875-1578 or your local NITON sales representative to enquire about upgrading your NITON analyzer to add capabilities.

Use the Arrow buttons to select the mode you wish to test in. Press **Clear/Enter** to select the mode.

The Bulk Sample mode

Bulk Sample Mode can be used to measure concentrations of contaminants in any fairly homogeneous, fine-grained medium such as soil, ground-up paint chips, a liquid or many other kinds of bulk materials.

To test in Bulk Sample Mode:

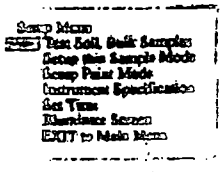


Fig. 2.12 Setup Menu Bulk Samples

1. Use the Arrow buttons to select

Test Soil, Bulk Samples

from the Setup Menu (Figure 2.12). Press the **Clear/Enter** button.

2. The instrument will return to the Main Menu ready to Calibrate & Test in Bulk Sample Mode. Press the Clear/Enter button.

3. The instrument will initiate self-calibration. This will take one to two minutes. When self-calibration is complete, the instrument will beep and display the Ready to Test screen for Bulk Sample Mode (Figure 2.13).

4. See Chapter 3: Testing Bulk Samples for details on how to test particular kinds of bulk samples.

Sun May 11 1997 20:39:22
Serial # XL309-U833NS0341

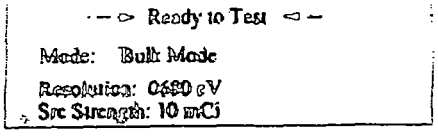


Fig. 2.13 Ready to Test. Bulk Mode

The Thin Sample modes

Thin Sample Modes can be used to measure concentrations of contaminants in a variety of thin layers, including deposits on dust wipes, filters and many other substrates, including, for example, thin layers of uranium on concrete.

Caution: The Standard Thin Sample Mode should not be used for quantitative lead-paint testing. Use only the three Paint Testing modes to test lead-based paint.

There are five Thin Sample Testing modes, each designed for a different type of test media:

1. 37 mm CE Filters: Used for 37 mm diameter filters (fiberglass or cellulose-ester) used in personal exposure monitoring. This mode can also be used for 37 mm filters used to analyze dust in Dust Vacuum Methods. In this Thin Sample Mode, three measurements are taken, weighted, and summed for each filter.

2. TSP/PM Filters: Used for the larger filters to monitor the concentration of metals in air. In this mode, the instrument averages the measurements you take on the filters.

3. Dust Wipes: Used for dust wipes to take samples by wiping surfaces following HUD guidelines for risk assessment and clearance testing for lead in dust.

4. Standard Thin Sample: Used for taking single measurements of samples or coatings. In this mode, results are displayed, in micrograms/cm^2.

5. User-Definable Thin Samples: User-definable testing gives you the flexibility to specify custom thin sample measurement protocols.

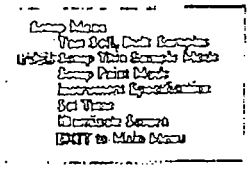


Fig. 2.14 Samp Menu
Thin Sample Mode

Testing in the Thin Sample Modes:

1. Use the Arrow buttons to select

Setup Thin Sample Mode

from the Setup Menu. Press **Clear/Enter**.

2. The **Choose Operation Mode for Thin Samples** screen will appear (**Figure 2.14**)

3. Use the **Arrow buttons** to select the mode appropriate for the kind of thin samples you are going to test. Press **Clear/Enter**.

4. The **Choose Operation Mode for Thin Samples** screen will *highlight* the thin sample mode you have selected and the cursor will move to **Exit to Main Menu (Figure 2.15)**. Press the **Clear/Enter** button to return to the Main Menu. Press the **Clear/Enter** button again to initiate **Calibration & Testing** in the thin sample mode you have selected.

5. The instrument will initiate self-calibration. This takes one to two minutes. When calibration is complete, the instrument will **beep** and display the **Ready to Test** screen for the thin sample mode you have selected (**Figure 2.16**).

6. See **Chapter 4: Testing Thin Samples**, for details on how to test thin samples.

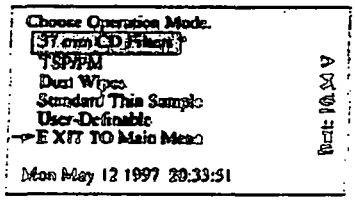


Fig. 2.15 Operation Mode
Select 37 mm CD Filters

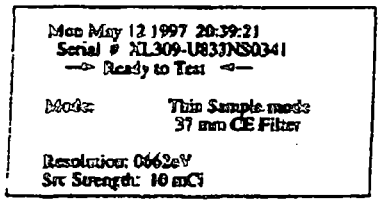


Fig. 2.16 Ready to Test
37 mm Filter Mode

The Paint modes

All three Paint Modes can be used interchangeably to measure lead concentrations in paint in mg/cm^2. In all paint modes, NITON analyzers simultaneously measure and analyze both K-shell and L-shell lead x-rays to determine (1) the numerical value of the lead in mg/cm^2 present in the sample; (2) the 95% confidence interval; and (3) whether the sample has a lead concentration that is greater-than-or-equal-to ("Positive") or less-than ("Negative") the lead **Action-level** (in mg/cm^2) that has been entered.

been entered.

Standard Paint Mode

In Standard Paint Mode, the instrument reads until a 95% confident reading of "Positive" or "Negative" versus the Action-level is achieved. Then the instrument displays either Positive or Negative, the Result in mg/cm^2, and displays Surface lead for all Positive readings where the lead is not shielded by overlying layers of non-leaded paint.

In Standard Paint Mode, testing times will vary somewhat from sample to sample. The instrument will measure *only* until a 95% confident reading of "Positive" or "Negative" (versus the Action-level you have set) has been attained. Most readings take 10 seconds or less.

Standard Mode + Spectra

Standard Mode + Spectra is identical to Standard Paint Mode except that the x-ray spectrum is displayed with each reading.

K & L + Spectra Mode

In K & L + Specra Mode, the instrument displays the complete test information *continuously*, from the beginning of each reading, including the K-shell reading with two-sigma confidence interval, the L-shell reading with two-sigma confidence interval, the combined reading (Pb) with two-sigma confidence interval, and the full x-ray spectrum. With each reading, a Null result is displayed until a Positive or Negative result is determined.

In K & L Mode + Spectra, you may continue readings indefinitely after a "Positive" or "Negative" result is obtained, until you have attained a desired measurement time or degree of precision.

Note: In all paint testing modes, if a test is stopped *before* a "Positive" or "Negative" determination has been made, you will get a "Null" test result.

Testing in the Paint Modes:

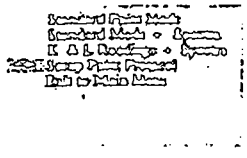


Fig. 2.19 Setup Paint Mode Arrow to Paint Present

1. Use the Arrow buttons to select

Setup Paint Mode

from the Setup Menu. Press Clear/Enter. The Setup Paint Mode menu screen will appear (Figure 2.17)

2. Use the **Arrow buttons** to select

Set up Paint Protocol

. Press **Clear/Enter**. The **Paint Protocol** screen will appear (**Figure 2.18**)

3. Use the **Arrow buttons** to adjust the times for the **1st beep**, the **2nd beep** and the **3rd beep** signals for **K & L Mode + Spectra** and to set the **Action level**. Use the **Clear/Enter** button to enter each selection.

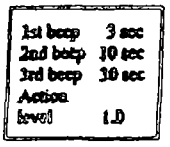


Figure 2.18: Paint protocol screen

4. When the **Action-level** has been entered, the **Setup Paint Mode** screen will re-appear (**Figure 2.17**). Now use the **Arrow buttons** to select a Paint Testing Mode. Press **Clear/Enter**.

5. The **Main Menu** will appear, with the instrument ready to **Calibrate & Test** in the paint mode you have selected. Press**Clear/Enter**.

6. The instrument will self-calibrate in one to two minutes. When self-calibration is complete, the instrument will beep and display the **Ready to Test** screen for the paint mode you have selected (**Figure 2.19**).

7. See **Chapter 5: Testing Paint Samples**, for detailed descriptions of all three paint testing modes.

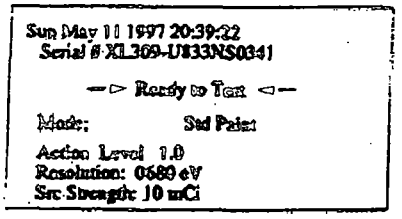


Fig. 2.19 Ready to Test. Paint Mode

Calibrating your NITON

Your NITON has been thoroughly calibrated at the factory. To further assure the best Quality Assurance/Quality Control, your NITON performs a second self-calibration check every time you turn on or reset the instrument.

In addition, NITON has provided you with several standard samples so you may check both

calibrations. These tests against known standards insure that the instrument is functioning properly and buttress your results with a permanent record of regular calibrations.

Instrument self-calibration

When the screen arrow (->) is on Calibrate & test, press Clear/Enter to start the self-calibration process (Figure 2.20). Self-calibration takes one to two minutes. When it is completed, the instrument will beep and the Ready to Test screen will appear.

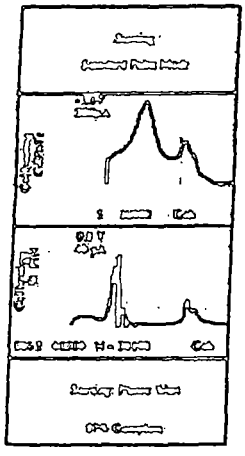


Fig. 2.20 [illegible]

Caution:

The ready to test screen

The Ready to Test screen (Figure 2.19) displays the following fields:

1.The current Date and Time.

Caution: Check the Date and Time. If they are not correct, reset them before taking any measurements (see page 10). Your readings will not be accurate unless the date and time are correct.

2. The instrument Serial Number.

3. The indication that the instrument is Ready to Test

4. The testing mode the instrument is ready to test in.

5. The Action-level the instrument will use to make either a "Positive" or "Negative" determination of lead in paint testing. The Action-level is only used in paint testing modes.

6. The Energy Resolution. The lower the number (in eV), the better the instrument will perform.

Caution: If you try to calibrate the instrument and it does not calibrate successfully, push the Reset Button on the bottom of the instrument and recalibrate. If your NITON does not calibrate successfully in three attempts, please call the NITON Service Department at (401)

294-1234.

7. The Source Strength (Src Strength). The Source Strength indicates the current activity of the cadmium$_{109}$ source in your instrument, in millicuries. Your NITON compensates automatically for the decay of the source.

Re-calibrating your NITON during testing

To insure the accuracy and precision of your NITON, it is recommended that you re-calibrate hourly during testing. To recalibrate:

Press the reset button on the bottom of your NITON.

or turn the NITON off, then on, and press the **Clear/Enter** button.

Note: Occasionally, your NITON may refuse to take further readings and the screen will display the following message:

YOU MUST RECALIBRATE.

Typically, this will occur when there is a sudden, very large change in the ambient temperature. When this occurs, recalibrate and continue testing.

How to use your NITON standard samples

NITON provides sets of standard samples for each testing mode. These are used to check the calibration of the instrument:

1. For Bulk Sample Mode, there is a set of three NIST soil standards

2. For Thin Sample Mode there is a set of three thin film standards: lead, copper, and iron.

3. For Lead Paint Mode, there is a set of government-traceable lead paint films.

Note: Although the standards do not contain every element our multi-element analyzers test for, when an instrument correctly measures the standards you have have received with your 700, your NITON will correctly measure the other elements.

Test the standards regularly. First, immediately after the instrument finishes self-calibration. Then test the standard samples appropriate to the type of tests you are conducting, and once every 1-2 hours thereafter.

Warning: Tampering with the 5,500 ppm lead-in-soil standard may cause exposure to lead dust. Keep all standards out of reach of children.

Caution: Never tamper with Test Standards. They should not be used unless they are completely intact.

Soil and Thin Film standards

To test soil or thin film standards, place the sample in the test platform receptacle and proceed to test as with any prepared sample. The NITON standard soil samples provided with your instrument contain known amounts of several elements. Do not contaminate the thin film samples with your fingerprints. Handle them by the edges with clean hands.

Lead paint standards

1. Place the NITON standard with the colored side face up. Choose the RED strip labelled 1.0 +/- 0.1. Take a reading of that standard. Place the instrument on the standard so that the instrument window is fully on the standard. Your NITON should display a value between 0.9 and 1.1 mg/cm^2 and should indicate Surface lead.

2. Place the same standard with the colored side down. Take a reading of the standard (buried beneath the equivalent of 5-6 coats of non-lead paint). Your NITON should still display a value between 0.9 and 1.1 mg/cm^2 and should not display Surface lead.

Note: If your instrument is testing high on Standard samples, check the surface the Standards are resting on. The surface may contain lead.

When you test the Standard samples, your instrument should give readings which approximate the certified values. Your instrument should give consistent readings for each sample.

Downloading data

Your NITON stores up to 3,000 measurements plus their spectra. You can download this data to a computer for reporting or insertion in a database.

Note: Downloading data does not erase readings. To make room for the next set of data, erase readings after verifying that the data was downloaded successfully (see next section).

The RS-232 port, on the back of your NITON, accommodates a 4-pin LIMO connector. A LIMO to 9-pin RS-232 connector cable is provided with your NITON. Your NITON can communicate with either a "dumb" or an "intelligent" terminal, such as a VT100 connected to a mainframe computer or a PC-compatible computer.

Fast data dump

You can download up to 3,000 measurements, their descriptions, and spectra (4-90 keV) in *minutes* using the high-speed compressed format, NITON/Mid-Hudson Downloading Software, provided with your instrument.

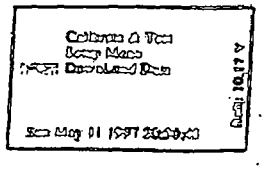


Fig. 2.21 Main Menu
DownLoad Data

1. Connect your NITON to your computer with the RS-232 port cable that is provided.

2. Using the Arrow buttons, select **Download Data** from the **Main Menu** and press **Clear/Enter** (**Figure 2.21**).

3. Select **Fast Data Dump** from the **Download Data menu (Figure 2.22)**and press **Clear/Enter**. Select the first to the last readings you wish to download. The **default** setting will download all readings currently stored in memory.

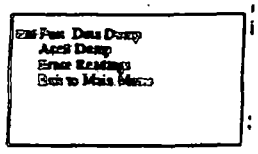


Fig 2.22 Download Data
Fast Data Dump

4. When the instrument finishes downloading, it will return to the **Main Menu**.

ASCII data dump

For users who wish to download data in ASCII format, the NITON can dump its data as an ASCII file to any terminal emulator program.

1. Connect the NITON to your computer with an RS-232 cable.

2. In the **Download Data** screen, press the **Arrow buttons** to scroll to **ASCII dump** (**Figure 2. 23**). Press **Clear/Enter**.

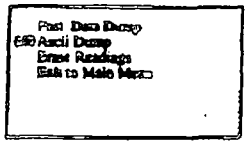


Fig. 2.23 Download Data
Ascii Dump

3. When the instrument finishes downloading, it will return to the **Main Menu**.

Erasing readings

If you do not erase your data, the NITON will continue to record data until the memory is completely

full. Then the NITON will start to overwrite older data. Any data that is overwritten in this way will be lost.

Your NITON can store data on up to 3,000 measurements in all Paint modes, or 1,000 readings in Bulk Sample or Thin Sample modes.

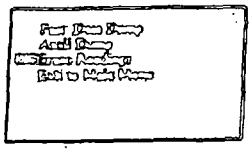


Fig. 2.24 Download Data
Erase Readings

Note: Download your data <u>before</u> the memory is completely full. Clear the memory after downloading.

The erase readings function is designed to protect you from <u>accidentally</u> erasing readings. To erase readings:

1. In the Download Data menu, use the Arrow buttons to scroll to Erase Readings (Figure 2.24). Press Clear/Enter.

2. The Erase Readings screen (Figure 2.25) appears with the following choices:

ERASE all readings

-> CANCEL do not erase

EXIT to Main Menu

The screen arrow defaults on Cancel do not erase, so that if you select it by mistake, you will not erase any readings.

3. To Erase Readings, use the Up-Arrow button to go to ERASE all readings. Then press Clear/Enter. When you enter either ERASE all readings or CANCEL do not erase your instrument will return to the Main Menu, ready to take and store more readings.

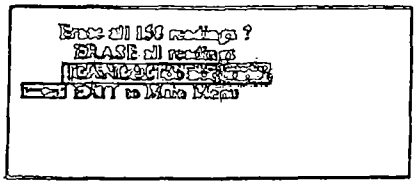


Fig. 2.25 Erase Readings Screen
Default is: CANCEL, do not erase

Battery packs and battery charger

Fully charged, each Nickel Metal Hydride battery pack gives eight or more hours of continuous use. It takes about 2.5 hours to fully recharge a spent battery pack if the batteries have been recently used. If the NITON has not been used for several weeks, or if the batteries are completely discharged, they must be pre-charged before they can be recharged. See **Battery Charger**, below.

NITON Battery packs can be recharged at least 500 times. They are warranted to be free of defect when shipped. They are not further covered by manufacturers' warranty. When they need to be replaced, new battery packs may be purchased from NITON.

Note: Before beginning a test, be certain the battery pack has sufficient charge. It is always a good idea to carry a spare battery pack.

Caution: NITON's Nickel Metal Hydride battery packs discharge at a rate of about 2% per day when not in use.

Battery pack routine maintenance

Some guidelines:

* Don't leave battery packs on the charger *all* the time. Overnight recharging is recommended.

* For longest battery lifetimes, use a battery until completely discharged, and then recharge.

* Don't recharge a fully charged battery pack. If you want to charge a partially charged battery, run the Discharge cycle before recharging.

* Store the charger and battery packs in a cool, but not cold, place, away from direct sunlight.

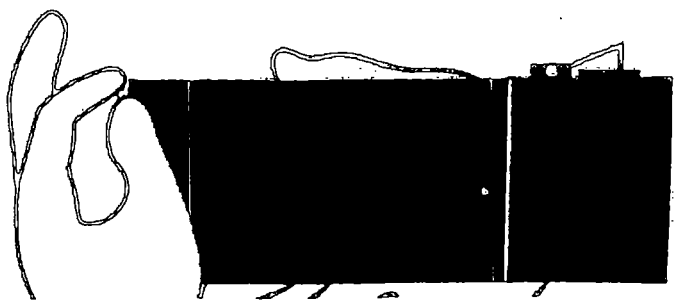
* When a battery pack is not used for a long period of time, it will lose its charge completely. Fully recharge it before use.

Note: The lithium battery inside your NITON will prevent any loss of data if you need to change the battery pack before downloading readings.

Changing battery packs

Removing a battery pack

Fig. 2.26.
Loosen clamp screws to remove the battery pack. The clamp screws stay attached to the battery pack.



1. Avoid changing the battery pack outdoors. Moisture and dirt can damage a battery.

2. Rest the NITON on a clean surface.

3. Loosen the (2) clamp screws. They do not come off (Figure 2.26).

4. Pull the battery pack away from the instrument by grasping the knurled screws and gently rocking the battery pack from side to side while removing it.

Installing a battery pack

1. Rest the NITON on a clean surface, as before.

2. Slip the notch at the bottom of the battery pack into the wide slot.

3. Gently push the battery pack in, taking care that the battery pack connector is seated properly to the instrument.

4. Tighten the (2) knurled screw clamps that fit into holes on the NITON. If the screw clamps do not tighten, the connectors are not lined up properly. These screw clamps must be tight for a secure connection.

Recharging battery packs

Recharging with the AC adapter

1. Lay the battery pack on top of Battery Charger. Fit connectors together snugly (Figure 2.27).

2. Plug one end of the AC adapter into the power port on the bottom of the charger. Push the plug in, making sure it seats fully.

3. Power up the charger: Plug the other end of the AC adapter into a 110V outlet. The yellow *Power* light will come on and stay on throughout. The green *Charge* light will also come on. It will blink slowly at first, indicating that the battery is on Pre-charge, and then stay on with a steady light, indicating that the battery is on Full Charge.

4. In Full Charge mode, the green *Charge* light will stay on with a steady light while the battery is being charged. It is normal for the charger to make some noise in Full Charge mode.

5. In Trickle Charge mode: When the battery is fully charged, the charger will automatically switch

to Trickle Charge mode and the green *Charge* light blinks rapidly.

Caution: Do not leave battery packs on the Battery Charger longer than necessary.

Battery charger

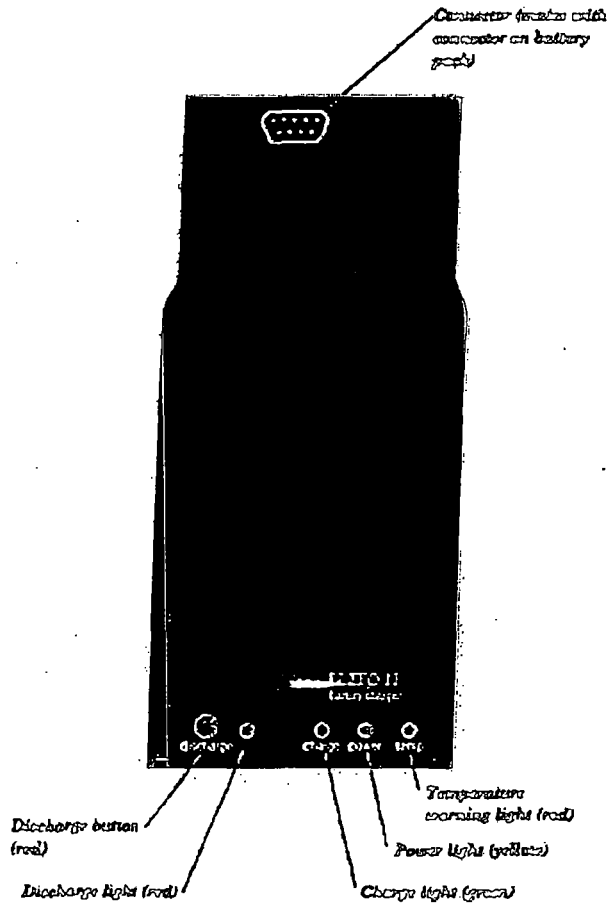


Fig. 2.27. Front view of the battery charger.

Discharge cycle

Put battery packs on the Discharge Cycle only if they are not holding a charge; or, if they are partially charged, run the Discharge Cycle before recharging. It takes about eight hours to fully discharge a battery pack. To discharge a battery pack, place it on the charger and:

1. Press the red Discharge button. The red Discharge light goes on, and the green Charge light blinks slowly, showing charger is in Discharge mode.

2. After a full Discharge cycle, the charger automatically recharges the battery.

3. The red Discharge light goes out and the green Charge light will blink rapidly, showing it is in the Trickle Mode.

Pre-charge

If your NITON battery packs run all the way down, they must be pre-charged before they can be re-charged. The process can take up to 5 hours. A battery is pre-charging when the green Charge light on the battery charger is blinking slowly, and the Discharge and Temperature lights are off.

Overheating during charge

Caution: If the red Temp light comes on repeatedly when a battery pack is on the battery charger in the Full Charge cycle, call NITON Customer Service at (401) 294-1234.

Caution: Do not store the battery packs or battery charger in direct sunlight.

Using your vehicles 12V DC outlet

[yen] A 12V DC Adapter is provided with your NITON. Instructions are the same as for using the 110V AC Adapter. When you have seated all connections well, the yellow Power light will come on.

[yen] Do not use the Discharge Cycle while on the DC outlet.

[yen] Secure the charger so the power cord does not get pulled out while the vehicle is in motion.

[yen] The plug of the DC Adapter has a 5A internal fuse. To check the fuse, unscrew the cap that retains the contact from the end of the plug. Replace this fuse only with a 5A fuse of the same size. If the fuse in the 12V Adapter burns out frequently, call NITON's Service Department at (401) 294-1234.

Note: Please do not throw away spent battery packs. Return spent battery packs to NITON so we can dispose of them properly.

Maintenance, cleaning and repairs

NITON Corporation welcomes any questions or comments you may have about your NITON analyzer. Please do not hesitate to call us at either our Main Office number: (781) 275-9275 or at our Rhode Island Service Facility number: (401) 294-1234.

Caution: All Service except exterior cleaning must be performed by NITON Corporation. Do not attempt to make repairs yourself. Opening the case of your NITON will void the instrument Warranty.

Keep your NITON clean, particularly the beryllium window on the bottom of the instrument. If the window is dirty, the performance of your NITON will be affected. Clean the window gently with cotton swabs. Clean the instrument's metal case with a soft cloth. Never use water, detergents, or solvents. These may damage the instrument.

Note: Never ship your NITON analyzer back to the factory for *any* reason without calling and obtaining a Return Authorization (RA) Number from NITON Corporation.

Storage, transport, and shipping

Storing and transporting your NITON

All NITON instruments come in waterproof, drop-proof carrying cases with padlocks. NITON instruments can be transported by car or plane or shipped as an ordinary package. There are no restrictions for tunnels or bridges. No notification is required for transportation except the following: There may be disclosure and/or licensing requirements if you take your NITON instrument across state or national boundaries. Please check with the appropriate agencies for details.

No special labelling is required on the outside of case or packaging. A compliance statement must be kept with the instrument case. *Always* transport the unit in its carrying case, and keep the NITON in its case whenever it is not being used. Store the instrument, in its case, in a secure area.

Shipping your NITON

All NITON instruments must be packed in their original padded carrying cases for shipment. Pack the NITON in its carrying case and ship in either the original carton and packing material or their equivalent.

Caution: Do not ship your instrument back to NITON for any reason without first notifying NITON Corporation and receiving a Return Authorization Number.

Caution: If you return your NITON without the carrying case you will void the instrument warranty. You will also be billed for a replacement case plus any repairs resulting from improper shipping.

Always enclose a copy of a current leak test certificate when you ship your instrument back to NITON.

Caution: NITON's license prohibits repairing or upgrading any XRF instrument without a current leak test certificate. If you return an instrument without a current leak test certificate, NITON will perform a leak test and bill you for the leak test.

Note: Keep a copy of the following statement in the NITON case whenever the instrument is shipped:

THE NITON SPECTRUM ANALYZER CONFORMS TO THE CONDITIONS AND LIMITATIONS SPECIFIED IN 49 CFR 173.422 FOR EXCEPTED RADIOACTIVE MATERIAL, INSTRUMENTS AND ARTICLES, N.O.S. UN-2910. THIS PACKAGE CONTAINS NO MORE THAN 50 mCi $CADMIUM_{109}$ IN A PLATED, SOLID, SEALED SOURCE INSTALLED IN AN X-RAY FLUORESCENCE ANALYZER.

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NITON Corporation

XL-309

&

700series

User's Guide Version 5.0 (HTML) Chapter 3

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3: Analyzing bulk samples

Overview

The NITON XL-309 may be used to test lead in soil and ground-up paint chips if equipped with optional Lead In Soil Analysis software and hardware. 702, 702-A, 703 and 703-A Model Spectrum Analyzers are multi-element analyzers for bulk media, thick samples of materials such as soil, sludge, and various liquids. Applications include:

- in-situ soil testing,
- in-situ materials testing (e.g., contaminated concrete)
- bagged soil sample testing
- testing sludge, sediments, liquids, and dust in cups,
- testing prepared soil samples.

Choose the Bulk Sample mode from the Setup screen (Figure 3.01).

Note: Before testing in Bulk Sample mode, turn your NITON on at least 15 minutes prior to

testing. This will give you more precise measurements.

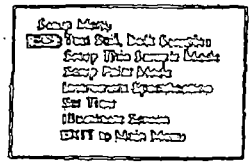


Fig. 3.01 Setup Menu: Test Soil, Bulk Sample

In general, testing methods for bulk media are of two types: Field screening and testing prepared samples. Understanding the difference between these two types of analysis is crucial to getting good data.

Field screening should be used to profile an area, to locate sources of contamination, to determine the boundaries of contamination, or to gather data that will subsequently be used to design a sampling plan. Field screening is usually only approximate; field screening will correlate very well with lab analysis for a highly-homogeneous sample, but may correlate extremely poorly for a non-homogeneous sample.

Note: For performance evaluation of field XRF results by comparing them to laboratory results (done to justify XRF usage), never use in-situ testing; always gather samples and prepare them before testing.

When comparing field screening to laboratory analysis, try to compare the same samples. For best results, collect a large sample in a zipper locking storage bag. Shake the bag to mix the sample. Test the bagged sample several times using the NITON and average the readings. Then compare this average reading with lab results.

If you must test in-situ for performance evaluation, take several XRF readings bracketing a spot. Then take a sample for laboratory testing from that spot. For further discussion of field screening, see EPA Method 6200, "Field Screening Using a Field-Portable XRF." Contact NITON for a copy. The EPA accepts field screening using the NITON if the screening is performed using Method 6200. Most states accept EPA Method 6200.

The measurement screen

On NITON XL-309s with optional Lead in Soil Analysis, *only* lead is displayed in bulk sample testing. On 700 models, only the two highest-concentration elements are displayed (in ppm, with the two-sigma confidence intervals) on the first Measurement screen (Figure 3.02a), with the x-ray spectrum. The black bars on the spectrum display highlight the presence or absence of lead or iron in the sample. The test time is also displayed in nominal (source) seconds.

The summary screen

When you end a reading, the Measurement Screen is replaced by the Summary Screen (Figure 3.02b). On 700 models, results are displayed for 14 elements. The elements are divided into two groups: elements that were detected in the sample, and elements that were not detected. Press the Arrow buttons to scroll through the elements.

Detection Limit: For an element to be detected by the NITON in a given sample, the measured concentration of the sample must be at least three times the standard deviation of the measurement. This detection limit will depend on the composition of the sample.

Precision: The measurement precision for each element displayed appears to the right of the measured concentration, under the heading "+-". The precision of each measurment is two times the standard deviation (sigma). An element is classified detected if the measured concentration (in ppm) is at least 1.5 times the precision.

Detected elements are displayed as in the Measurement screen. Non-detected elements are shown as < xx, where xx is the detection limit for that sample. The detection limit for each element is calculated from each sample.

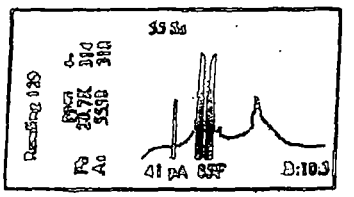


Fig. 3.02a Measurement Screen
Bulk Mode. In-progress screen.

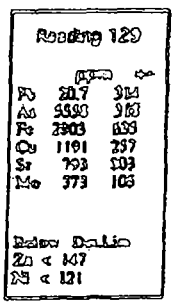


Fig. 3.02b Bulk Mode
Summary Screen

In-situ surveys

Before you take your first measurement, you must decide whether to test the bulk material

- in-situ (in-place),
- as bagged samples (or, for liquids and sludge, in cups) with a minimum of preparation, or
- in an XRF cup after careful preparation.

Note: More sample preparation (drying, milling and sieving) will yield greater accuracy. The drier, finer, and more homogeneous the particles, the better the measurements.

If you are primarily interested in determining whether an element is present (rather than in accurately measuring how much is present), direct measurement is the quickest, simplest way to proceed. Even if you intend to take samples, preliminary direct measurements will help you to survey the site. The analysis of bagged samples is another screening technique.

The NITON test guard

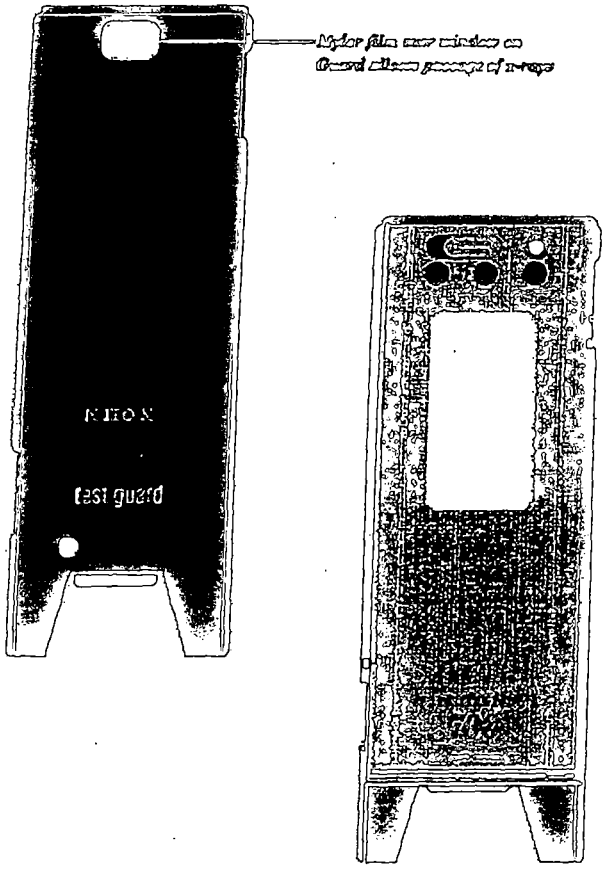


Fig. 3.03
The NITON
Test Guard

The NITON Test Guard (Figure 3.03) is a formed metal plate designed to be placed directly between the ground or other bulk media and the NITON. Use the Test Guard for surveys of bulk media *in-situ* or for testing bulk samples in bags. The Test Guard shields the unit from contamination and damage.

Testing in-situ

Warning: When taking samples from a site where toxic chemicals may be present, always use gloves and respiration equipment for your own protection.

1. Select a measurement site. Lead-in-soil from paint, for instance, will be concentrated within a few feet of the painted structure. Valid results will depend on a sufficient and appropriate selection of sites to sample.

2. Clear any surface debris or vegetation. Use a flat area so that the NITON will contact the test medium. The finer and more homogeneous the material, the more accurate the measurement. (You can increase your accuracy when testing soil by loosening the soil and letting it dry in the sun before testing.)

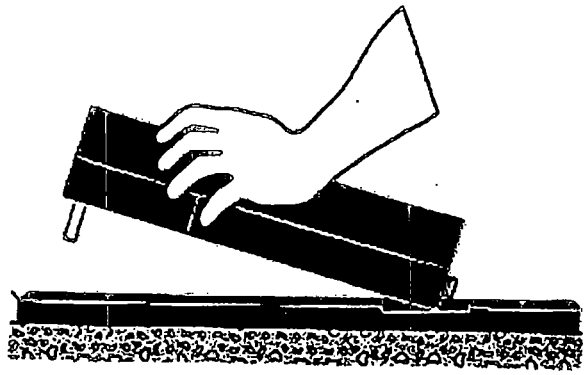


Fig. 3.04a
Place your NITON on the Test Guard.

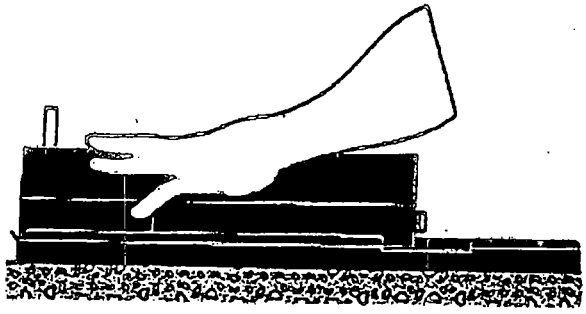


Fig. 3.04b
Firmly press your NITON flat against the surface.

3. Place the test guard on ground. Keep the top of the test guard clean.

4. Hold the NITON in one hand.

Warning: Always treat radiation with respect. Do not put your hand on the end plate of the NITON while measuring. Never point the NITON at yourself or anyone else when the shutter is open.

5. Push the safety slide (that locks the shutter release) out from under the shutter release. If the slide is still tucked in, you cannot press in the release nor will the instrument fit on the test guard correctly.

6. Place the NITON on the test guard so that the rectangular opening on the test guard is under the window of the NITON, squeeze the shutter release, and firmly press the instrument flat against the surface of the test guard (Figure 3.04 a,b). If you don't squeeze the shutter release, the plunger will not depress. If the plunger is not fully depressed, the window is not fully open and the NITON cannot measure accurately. The back of the unit must be flush with the test guard.

Note: During the measurement, you do not need to squeeze the shutter release continuously. Hold the NITON firmly against the test guard surface and it will continue to read. Once you lift the instrument, the plunger will back out the bottom, the shutter will close, and the test will be finished.

7. Watch for indications to decide when the test has reached the desired level of accuracy. A typical screening test will last 20-30 source seconds.

Warning: In the unlikely event that the plunger gets stuck in the open position, simply push it closed. Then call the NITON Service Department at (401) 294-1234.

In-situ depth profiling

An XRF soil test examines only the top millimeter or so of soil. To do depth profiling, simply remove a vertical slice of soil and test several samples from different depths. Doing so rapidly yields information about the depth of contamination.

Analysis of bagged bulk samples

Sometimes it is convenient to collect samples in plastic bags. Without further preparation of the sample, you can screen the site by testing each bag. Because you are testing through a bag, test results will tend to be 5-10% lower than test results obtained from direct analysis.

Taking bagged samples

1. Before sampling a site, size it up for differences in soil characteristics. Valid results depend on a sufficient and appropriate selection of sites to sample. Consider the site's topography, texture, drainage, color of topsoil, and past use.

2. Take a composite sample from each predetermined area. Do not combine samples from areas with different compositions or history. A composite sample made up of samplings from two distinctly different areas is not representative of either area.

Mix the sample. If it is too large, reduce the sample. Some techniques for reduction and homogenization are described in the section on analysis of prepared samples.

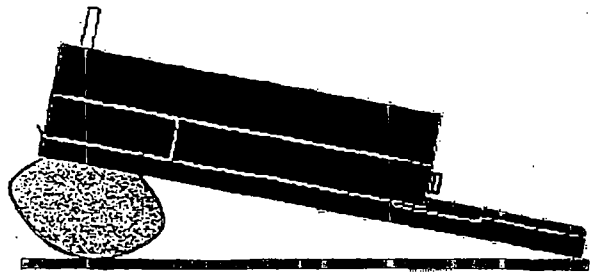
3. Fill a clean plastic bag with 50-100 grams of soil and close it securely (with a twist tie). The accuracy of your measurements will be limited by the thickness of the plastic in the bag you use. 1 mil-thick Polyethylene bags offer a reasonable compromise between accurate readings and bag durability. Be sure to label each bag with your name and the location of the sample site.

Testing samples in bags

Shape the bag of soil to form a continuous uniform layer of at least 1 cm. (0.4 inch) thickness. Place the NITON test guard on the bag (Figure 3.05). Then follow testing in-situ instructions.

Warning: Do not hold bagged bulk samples in your hand during testing.

Fig. 3.05
To test a bag of soil, firmly press your NITON plus Test Guard flat against the surface of the bag (which should rest on a firm surface – not on your hand).



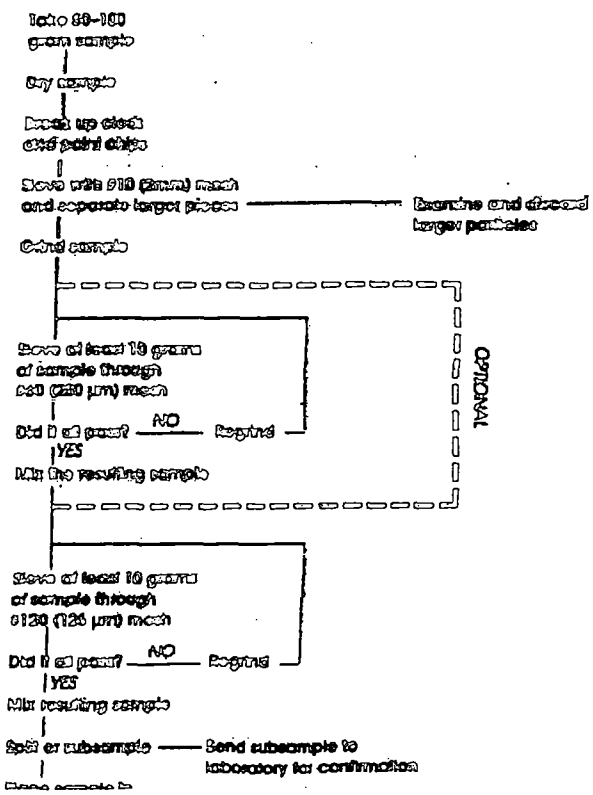
Analysis of prepared bulk samples

Prepared sample analysis is the most accurate method for determining the concentration of elements in a bulk medium using your NITON. Sample preparation will minimize the effects of moisture, large particle size and variations in particle size.

Warning: For your protection, when taking samples from a site where toxic chemicals may be present, always use gloves and respiration equipment.

NITON recommends a specific sample protocol. Following this protocol for preparing and testing samples is vital for achieving a level of accuracy comparable with laboratory results. See Figure 3.06 for a flow chart of the protocol.

Fig. 3.06 Flow chart of sample preparation protocol recommended by NITON.
Use of the #10 mesh sieve is optional.



Taking bulk samples

Note: When testing for lead-in-soil in a residential setting, it is standard practice to sample the top 4 to 6 inches of soil.

The soil probe or sampling tube is a very convenient sampling tool. It not only allows speed but it makes more accurate composite samples than any other tool as it may always be inserted to a marked depth and it removes the same amount of soil at each insertion. There are core sampling devices that remove an intact cylinder of undisturbed material.

A shovel, spade, dibble, narrow (1-1/2 inch) garden trowel, or other sampling tool can do the job. Take a half-inch soil slice. A satisfactory soil auger may be made by welding a 1-1/4 or 1-1/2 inch wood bit into a 1/2 inch pipe equipped with a T-handle.

Take 50-100 gram sample to insure that you have a sample large enough to be representative and unbiased after mixing, grinding, and straining it.

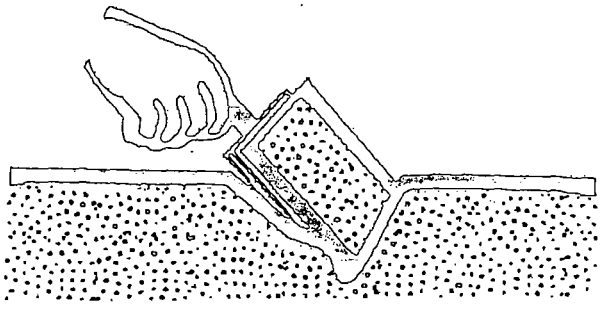
1. Before sampling a site, evaluate it for differences in soil characteristics. Valid results depend on a sufficient and appropriate selection of sites to sample. Test results may be worthless, even highly misleading, unless the samples tested actually represent the area.

Consider topography, texture, drainage, color of topsoil, and past use. Lead, for instance, is usually concentrated near a building with lead paint (within 4-6 feet).

2. If the individual samplings are taken with a spade or trowel, (Figure 3.07) reduce the samples by taking a vertical slice (so it is representative of the entire spadeful) about one inch wide.

Place the reduced samples in a clean pail. Then mix the sample thoroughly by stirring and by rotating the pail at an angle of 45 degrees. Don't shake. (You do not want to stratify the sample by weight).

Fig. 3.07
Use a spade, trowel or garden dibble to take a half-inch thick slice of soil.



3. Take a composite sample from each predetermined area. Do not combine samples from areas with different compositions or history. A composite sample made up of samplings from two distinctly

different areas is not representative of either area.

From each predetermined area, prepare a composite sample by taking several samplings consisting of vertical columns of material approximately 1 inch in diameter. The length of each column should be about 6 inches. Lead from paint is usually concentrated within the top 1-4 inches. The elements you wish to measure and the local history will determine how deep you need to sample.

Package samples from the following areas separately: samples close to painted structures, close to roads, samples close to where various types of waste have been stored, or near pressure-treated lumber.

4. Fill a clean plastic bag and close it securely (with a twist tie). Be sure to label it with the date, the site and the location where you took the sample

Preparing bulk samples

The equipment you need to prepare samples is included in your kit. Among these are a mortar and pestle (for the XL-309 with lead-in-soil-analysis), an electrically powered grinding mill (included with 700s), and several sized-sieves.

Caution: Keep all test equipment clean to prevent contaminated samples.

The mortar, pestle, and grinding mill may be cleaned with dry paper towels. Water will also clean the mortar, pestle, and the mill's container, but be sure each is absolutely dry before you use them on another sample. The mortar and pestle may be cleansed by grinding clean dry sand in the mortar. Use the short bristle brushes (included in your Bulk Testing Kit) to clean the sieves. When Soil Grinder blades wear out, unbolt the worn blades and replace.

Cone and quartering

At various times while preparing a sample you may need to divide it. Cone and quartering is a method for splitting the sample into homogenous quarters. Slowly and carefully pour the dry material onto a flat sheet or pan forming a symmetrical cone. Using a flat thin-bladed tool, such as a knife or ruler, divide the cone into equal piles. Divide these in half again. Now you have four samples, each one-quarter the size of the original and each more homogenous than the original.

1. If the sample is moist and cohesive, dry it. To best prepare a sample for presentation to the XRF, the material should be dry and well homogenized. Ideally, the entire sample should be dried to constant weight, sieved to remove gravel and debris, and ground or milled to a fine powder.

The sample can be dried in any of several ways. Choose one of the following: Oven dry the sample for approximately 2 hours at 150° C., until the sample reaches a constant weight; air dry the sample overnight at room temperature in a shallow pan; gently stir and warm the sample in a pan over a hot plate or burner.

Oven drying is inappropriate when volatile compounds may be present in the sample. For example, lead present as tetraethyl lead would be driven off by the heat of drying. Some forms of mercury and arsenic are volatile. Air drying will preserve more of these volatile substances.

2. Grind the sample to break up dirt clods and/or paint chips.

3. Sieve with the #10 (2mm) mesh and separate out the larger pieces (stones, organic matter, metallic objects, etc. Examine the larger particles by eye (look for paint chips), but do not include in the sample.

4. Grind the sample so its particles will be finer and more homogenous. Use mortar and pestle, or an electrically powered grinding mill.

Warning: Grinding-and-sieving dried samples produces dust. Even clean soil contains silica, which may be hazardous when airborne. Prepare all samples in a ventilated area; wear a mask, gloves, and an apron; and spread a drop cloth.

5. Sieve at least 10 grams of the sample through #60 (250 um) and #120 (125 um) mesh. Re-grind the unpassed material until the required fraction is able to pass.

6. Mix the resulting sample.

Putting the sample in an XRF sample cup

The container holding the sample affects the accuracy of the measurement. Use a container with as thin-walled a window as is convenient and use the same kind of container and window for each sample. Consistency and careful attention to detail are keys to accurate measurement.

Note: The sample container should be a sample cup of a type that can be filled from the rear; that is, the side opposite the window (e.g. Chemplex #1330). NITON recommends using a 1/4 mil mylar film window (Figure 3.08). A supply of cups and windows are included.

1. Place a circle of mylar film on top of an XRF sample cup. The window goes on the end of the cup with the indented ring. Note that the window may be prepared ahead of time.

2. Secure the film with the collar. The flange inside the collar faces down and snaps into the indented ring of the cup. Inspect the installed film window for continuity and smooth, taut appearance.

3. Set the cup, window-side down, on a flat surface. Fill it with at least three grams of the prepared sample (no more than half-full). Take care that there are no voids or layering.

4. Placing the cup film-side down on a flat surface, tamp the sample into the cup. The end of the pestle makes a convenient tamper. If you intend to re-use the sample, you can, alternatively, place a filter-paper disk on the sample before tamping it.

5. Fill the cup with polyester fiber stuffing to prevent sample movement. Use aquarium filter or pillow filling as stuffing. A small supply of stuffing comes with your bulk sample kit.

6. Fasten the cap on the cup (Figure 3.09). Using an indelible pen, write an identifying number on the cup. Keep a record of the sample number, the site and location, the date of the sample, and any other relevant comments.

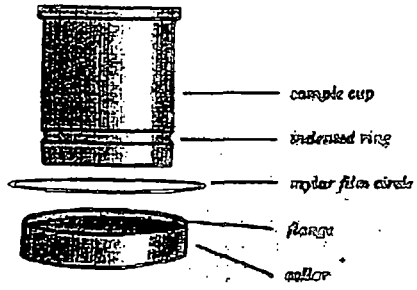


Fig. 3.03 Secure the film by snapping the collar on to the cup.

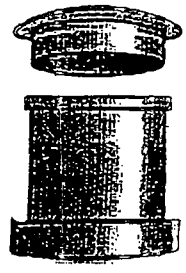


Fig. 3.04 Fasten the cup on the cup.

Preparing samples of liquids, sludges or dust

Liquids:

Fill an XRF sample cup with the liquid to be tested (Use no cotton). It is best if some overflows when the cap is put on, since the cup must be full.

Sludge:

Sludge can be placed directly in an XRF cup for screening. This is considered in-situ testing because no attempt has been made to prepare the sample. For more accuracy, the sludge can be dried, sieved, and ground.

Screening dust:

Use large dust samples taken from a home vacuum cleaner bag. Remove fibers, hairs, and debris. At least three grams of dust are needed to assure accurate analysis. Samples as small as one or two grams may be measured with less accuracy. Even smaller samples (0.3 to 1.0 grams) can be analyzed by applying a weight correction factor and by using a funnel to place the sample in the center of the sample cup.

Prepare in an XRF sample cup and test the same way you would with a soil sample. For risk analysis, it is advisable to use a 60-mesh sieve to isolate and test only fine particles.

The bulk testing platform

The test platform (Figures 3.10a,b) is an accessory fixture for holding bulk samples (such as soil or ground paint chips) in standard film-window XRF cups. This fixture snaps quickly and securely to your NITON instrument.

The platform latch screws underneath for storage. Before using the test platform, unscrew the latch and rescrew it on the end of the platform nearest the receptacle for the sample cup.

The test stand securely holds the XRF sample cup in place.

Testing the sample;

Set the NITON test platform on a flat, solid surface. Place the sample cup in the receptacle of the sampler. Included in your kit are some foam disks that you can put in the receptacle under the cup for firmer contact between the NITON and the sample cup window. Attach the NITON to the test stand and follow in-situ bulk sample instructions (Figures 3.11 a,b).

Fig. 3.10a
The Niton
Test Platform

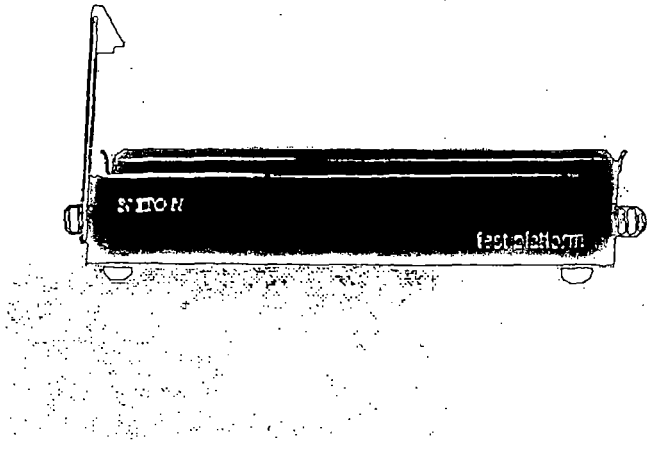
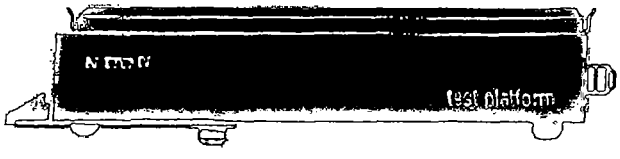


Fig. 3.10b
The Niton
Test Platform
with its latch in
the stored position.



NITON Back to the Table of Contents

1. Introduction

Liquid and solid environmental samples are routinely collected by E & E during field surveys, site investigations, and other site visits for laboratory analysis. Unless the samples have anesthetic, noxious, or other properties that could inhibit the ability of a flight crew member to perform his or her duty or are known to meet the established U.S. Department of Transportation criteria for hazardous material (i.e., explosive, corrosive, flammable, poisonous), they are not regulated as hazardous materials.

This Standard Operating Procedure (SOP) describes the packaging procedures to be used by E & E's staff to ensure the safe arrival of the samples at the laboratory for analyses. These procedures have been developed to reduce the risk of damage to the samples (i.e., breakage of the sample containers), promote the maintenance of sample temperature within the cooler, and prevent spillage of the sampled material should a container be broken.

In the event the sample material meets the established criteria of a DOT hazardous material, the reader is referred to E & E's Hazardous Materials/Dangerous Goods Shipping Guidance Manual (see H&S 5.5).

2. Scope

This SOP describes procedures for the packaging of environmental samples in:

- Coolers;
- Steel, aluminum and plastic drums; and
- 4GV fiberboard boxes.

The Hazardous Materials/Dangerous Goods Shipping Guidance Manual will complete the information needed for shipping samples by providing guidance on:

- Hazard determination for samples which meet the USDOT definition of a hazardous material;
- Shipping profiles for "standard" shipments;
- Shipping procedures for "non-standard" shipments;
- Marking of packages containing hazardous materials;

- Labeling of packages containing hazardous materials; and
- Preparation of shipping papers for hazardous materials shipment.

3. Sample Packaging Procedures

3.1 General

It is E & E's intent to package samples so securely that there is no chance of leakage during shipment. This is to prevent the loss of samples and the expenditure of funds for emergency responses to spills and the efforts necessary to re-obtain the sample.

Over the years, E & E has developed several "standard" package configurations for the shipping of environmental samples. These standard package configurations are described below.

Liquid samples are particularly vulnerable. Because transporters (carriers) do not know the difference between a package leaking distilled water and a package leaking a hazardous chemical, they will react to a spill in an emergency fashion, potentially causing enormous expense to E & E for the cleanup of the sample material. Therefore, liquids are to be packed in multiple layers of plastic bags and absorbent/cushioning material to preclude any possibility of leaks from a package. This section defines the standard packaging configurations for environmental samples.

3.2 Liquid Environmental Sample Packaging Procedures

Liquid environmental samples should be collected and preserved as outlined in the Standard Operating Procedures (SOP) for Surface Water Sampling (ENV 3.12), and Groundwater Well Sampling (ENV 3.7). Preserved water samples are not considered to meet the HM/DG definitions of Class 8 (Corrosive) and are therefore considered to be nonhazardous samples. Liquid environmental samples may be shipped using an 80-quart cooler or an outer package consisting of either a steel or aluminum drum. Because the steel and aluminum drums provide little insulating capability, they should not be used for samples that require icing.

Packaging Liquid Environmental Samples Using the 80-Quart Cooler

- Label and seal all water sample bottles according to appropriate sampling SOPs;
- Secure the bottle caps using fiberglass tape; and
- Place each amber, poly, and volatile organic analysis (VOA) bottle in a sealable plastic bag. Mark the temperature blank VOA bag for identification.

If a foam block insert is used:

- Line the cooler with two plastic bags;

- Place a foam insert (with holes cut to receive the sample bottles) inside the plastic bag;
- Place the bottles in the holes in the foam block;
- Fill void spaces with bagged ice to the top of the cooler;
- Fold over the plastic bags lining the cooler and secure shut with tape;
- Place Chain-of-Custody (C-O-C) form in a sealable bag and tape it to the inside of the cooler lid; and
- Secure the cooler with strapping tape and custody seal. Cover the custody seals with clear tape.

If vermiculite is used:

- Place 1 inch of inert absorbent material (vermiculite) in the bottom of the cooler;
- Line the cooler with two plastic bags;
- Place each sample bottle inside the inner bag;
- Fill the void spaces around the bottles with vermiculite to about half the height of the large bottles;
- Fill the remainder of the void spaces with bagged ice to within 4 inches of the top of the cooler, making sure the VOAs are in direct contact with a bag of ice;
- Fold over the plastic bags lining the cooler and secure shut with tape;
- Fill the remaining space in the cooler with vermiculite to the top of the cooler;
- Place C-O-C form in a sealable bag and tape it to the inside of the cooler lid; and
- Secure the cooler with strapping tape and custody seal. Cover the custody seals with clear tape.

Alternate Packaging Using 1A2/1B2 Drum

- Place 3 inches of inert absorbent material (vermiculite) in the bottom of the drum;
- Line the drum with two plastic bags;

- Place each sample bottle inside the inner bag;
- Fill the void spaces around the bottles with vermiculite to the height of the larger bottles;
- Fold over the plastic bags lining the drum and secure shut with tape;
- Fill the remaining space in the drum with vermiculite to the top of the drum;
- Place C-O-C form in a sealable bag and tape it to the inside of the drum lid; and
- Secure the drum with closing ring and apply custody seals. Cover the custody seals with clear tape.

3.3 Soil/Sediment Environmental Sample Packaging Procedures

Soil/sediment environmental samples should be collected as outlined in the SOP for Soil Sampling (ENV 3.13), and SOP for Sediment Sampling (ENV 3.8). Soil/sediment environmental samples may be shipped using an 80-quart cooler, a 4GV fiberboard combination package, or an outer package consisting of either a steel or aluminum drum. Because the steel and aluminum drums provide little insulating capability, they should not be used for samples that require icing.

Packaging Soil/Sediment Environmental Samples

- Label and seal each sample container according to SOPs;
- Secure the bottle caps using fiberglass tape;
- Place each sample bottle inside a sealable plastic bag and place it in its original shipping box or in individual fiberboard boxes. Mark the temperature blank bag for identification; and
- Secure the original shipping box with strapping tape, place shipping box in a plastic bag, and secure the plastic bag with tape.

If an 80-quart cooler is used:

- Place bubble pack or similar material on the bottom and sides of an 80-quart cooler;
- Place the bagged shipping boxes in the cooler with a layer of bubble pack between each box;

- Fill the void spaces with "blue ice" or ice in baggies to the top of the cooler;
- Place C-O-C form in a sealable baggie and tape it to the inside of the cooler lid; and
- Secure the cooler with strapping tape and custody seal. Cover the seals with clear tape.

If a 1A2/1B2 drum is used:

- Place 3 inches of inert absorbent material (vermiculite) in the bottom of the drum;
- Line the drum with two plastic garbage bags;
- Place the boxes inside the inner bag;
- Fill the space around the samples with vermiculite;
- Fold over the plastic bags lining the drum and secure shut with tape;
- Fill the remaining space around the bags with vermiculite to the top of the drum;
- Place C-O-C form in a sealable bag and tape it to the inside of the drum lid; and
- Secure the drum with the closing ring and apply custody seals. Cover the custody seals with clear tape.

Note: If a small number of samples are being shipped, it may be more practical to package them using the vermiculite or foam block configurations used for shipping liquid samples.

4. Shipping Procedures

Environmental samples are to be shipped as nonhazardous cargo. Unless the samples have anesthetic, noxious, or other properties that could inhibit the ability of a flight crew member to perform his or her duty or are known to meet the established U.S. Department of Transportation criteria for a hazardous material (i.e., explosive, corrosive, flammable, poisonous), they are not regulated as hazardous materials. When preparing the containers (i.e., cooler, drum, or box) for shipment, E & E staff must remove all labels from the outside container. Labels indicating that the contents may be hazardous are misleading and are not appropriate. Markings indicating ownership of the container, destination, and chain of custody labels are acceptable and can be attached as required.

When completing the paperwork for shipment, the standard nonhazardous forms must be used. Do not use the hazardous materials/dangerous goods airbills, either in total or in part; these-forms are coded and their use will invite unnecessary questions. This will only serve to confuse Airborne or Federal Express' terminal personnel and will cause much frustration and the delay of sample shipment.

Environmental sample packages can be shipped overnight by both Airborne and Federal Express. When choosing between the two, cost should be considered. It is normally much cheaper to ship Airborne. For work conducted and paid for by E & E, it is E & E's policy that you must first attempt to ship by Airborne before considering Federal Express. In addition, Airborne tends to have remote locations open later in the evenings than Federal Express, which may be helpful when trying to complete a full day's sampling effort and still make the flights on time. Although both companies offer pickup of samples at the site, it is advisable to call ahead and ensure that this service is offered beforehand. In almost all cases, both companies will deliver to the laboratory of your choice on Saturdays. When planning for sampling activities, check with the companies in advance to verify pick-up and delivery schedules.

When dropping off the sample packages at Airborne or Federal Express, do not indicate that these samples may be hazardous. This will only raise questions and delay the shipment. Although you may have an idea of the kind of contamination that is contained in the samples, you must remember that you are sending these samples to a laboratory for analyses to determine the amount and type of contamination, if any. Until that is determined, the presumption is that the samples are environmental in nature and not subject to the hazardous materials restrictions.

APPENDIX G

JEROME® 431-X™ MERCURY VAPOR ANALYZER OPERATION MANUAL

JEROME®
431-X™
MERCURY VAPOR ANALYZER

OPERATION MANUAL

September, 2000

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JEROME 431-X

Mercury Vapor Analyzer

Operation Manual

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FOR THOSE WHO CAN'T WAIT TO USE YOUR JEROME 431-X BEFORE READING THIS MANUAL

CAUTION: The Jerome 431-X is intended for vapor use only. DO NOT allow the probe or the instrument's intake to come in contact with liquids, dust or other foreign material.

Please read the manual for added details that will optimize the results and the life of your instrument or if your application requires use of dosimeters, special voltage inputs or data output. Also, refer to the manual for complete details on operation, maintenance and troubleshooting.

The Jerome 431-X is easy to operate and ready for use upon receipt from the factory. Follow these brief steps to use your instrument.

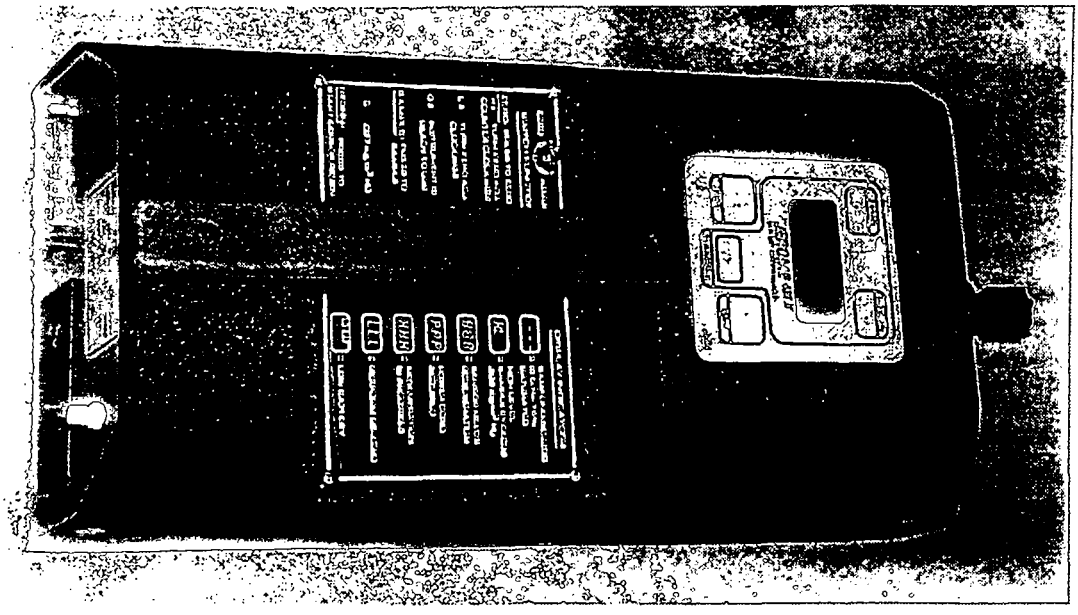
- Remove the instrument from the packing material. Check for any damage and confirm receipt of all parts on your packing list. Contact Arizona Instrument Customer Service at 800-528-7411 or 602-470-1414 if you have any questions.
- Press the ON button. In less than one second the display should read 000. Note that a LO BATT message appears briefly in the upper left corner. If the LO BATT light persists, charge the battery. See page 13 for details.
- Check the voltage setting (110 or 220 VAC) on the back of the instrument. Ensure that it is set to the correct voltage. If the voltage must be changed, turn the knob. However, it may also be necessary to change the frequency setting; see page 18 for details.
- Perform a sensor regeneration by following these steps:
  - ➢ Plug the line cord into the instrument using the plug in the back and to an AC power outlet.
  - ➢ Power the instrument ON and press the REGEN button. The instrument will begin a 10 minute regeneration cycle, indicated by .H.H.H flashing on the display. Do not interrupt this cycle. For a complete description of this process, see page 10.
  - ➢ If any error message, such as .H.L.P or .L.L.L appears on the display, see the Troubleshooting section on page 20.
- Adjust the sensor zero by pressing the ZERO button and turning the zero adjust screw located under the handle. Adjust until the display reads 0.
- The instrument is now ready to sample. Note that as the instrument measures mercury, the ZERO will display H. Do not adjust the ZERO after the instrument has measured mercury and before the next regeneration. (Occasionally the ZERO may drop to L (for low) between the initial zeroing and the first sample. It is OK to readjust the ZERO if the instrument has not measured mercury.)
- The instrument is designed for work space air monitoring. Press the SAMPLE button to start a 12 second sampling cycle.
- DO NOT allow the probe or the instrument's intake to come in contact with liquids.
- Note that the instrument is not explosion proof.
- After the day's survey, again perform a sensor regeneration. When complete, store the instrument with the zero air filter in the intake.

If you have any questions call AZI Customer Service, or your Technical Sales Representative, at 1-800-528-7411 or 1-602-470-1414

2 INTRODUCTION

The Jerome 431-X Gold Film Mercury Vapor Analyzer is designed for the easy and accurate analysis of mercury vapor in the workplace environment and for the location of mercury spills. The 431-X is easy to operate and has few maintenance requirements however, please take a moment to read this manual before attempting operation.

The Jerome 431-X is an ambient air analyzer with a range of 0.001 to 0.999 milligrams per cubic meter (mg/m^3 Hg). If you have any questions about your application or operation, please call AZI Customer Service at 800-528-7411 or 602-470-1414 for assistance.



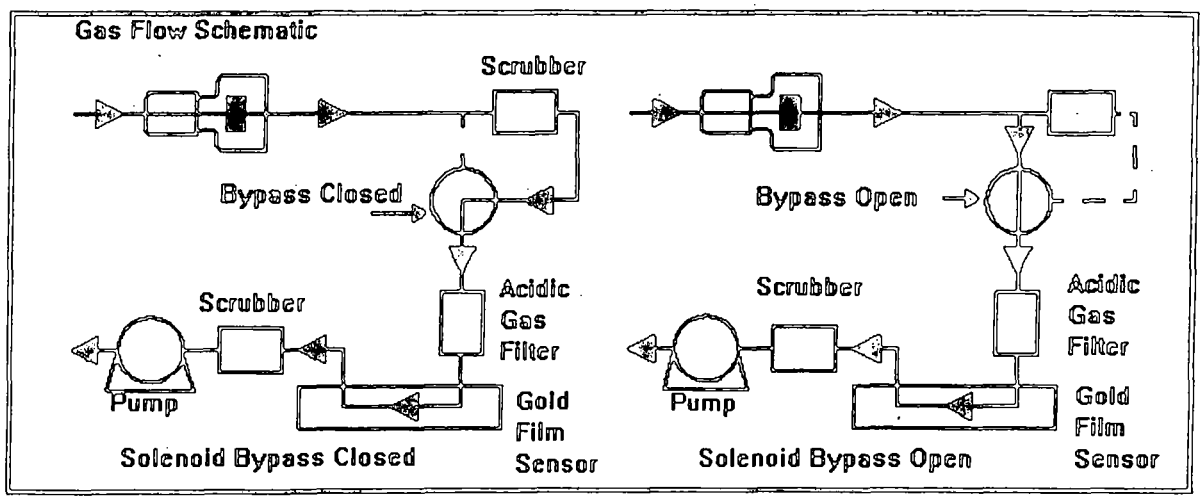
2.1 Features of the 431-X

- Automatic sensor regeneration when equipped with the communications option and used with the Jerome Communication Interface Software (JCI) program and the Jerome data logger.
- Regulated film heat voltage during sensor regeneration. This allows the sensor to clean properly with voltages from 100-130 VAC (or 200-260 VAC).
- Survey mode can be locked in.
- DIP switch setting can change the digital meter readings from mg/m^3 Hg to nanograms (ng) of Hg (see page 18).
- The Jerome 431-X can be operated from 100-130 or 200-260 VAC. To change the default voltage range, refer to Setting the Input Voltage, page 18.

3 PRINCIPLES OF OPERATION

Mercury is unique in its ability to alter the resistance of a gold film. The 431-X sensor consists of two thin gold films, a reference and a sensor, configured in a Wheatstone Bridge Circuit, which detects very small changes in electrical resistance. The reference film is sealed and not exposed to mercury. The sensor film is exposed to mercury resulting in resistance changes, which are measured by the circuit. A microprocessor computes the concentration and displays the results.

Activating the SAMPLE mode starts an internal pump which draws air through a scrubber filter and into the flow system. After 2 seconds, the sample solenoid bypass opens, closing off the scrubber filter from the flow system. The sample air passes through a filter (removing any acidic gases which interfere with the sensor's response to mercury) and is drawn over the gold film sensor. The sensor adsorbs and integrates the mercury vapor. Nine seconds after starting, the sample solenoid bypass closes and the remainder of the sample is drawn through the scrubber filter and the flow system. The measured concentration is then displayed on the digital meter in milligrams per cubic meter (mg/m^3) of mercury. An internal DIP switch can be used to change the digital meter display from mg/m^3 to nanograms of mercury (see page 18).



The instrument's microprocessor automatically rezeroes the digital meter at the start of each sample cycle and retains the meter reading until the next sample cycle begins, thus eliminating drift between samples.

During the sample cycle, bars on the digital meter represent the percentage of sensor saturation. Approximately sixty-five samples containing 0.1 mg/m^3 Hg may be taken before the sensor reaches saturation. After absorbing approximately 500 nanograms of mercury, the sensor becomes saturated and needs to be cleaned. This is accomplished by a manually activated 10 minute heat cycle, or sensor regeneration which burns the mercury from the sensor. This mercury is absorbed on internal filters to prevent any external contamination. The solenoid bypass closes during the sensor regeneration cycle, causing the air to pass through the scrubber filter, providing clean air for the regeneration process. The flow system's final scrubber prevents contamination to the atmosphere from the desorbed mercury.

After a sensor regeneration, it is necessary to bring the two gold films back to a similar resistance. The ZERO button, along with the ZERO ADJUST potentiometer, are used to reset the sensor's reference film and sensor film to the same baseline. The sensor may exhibit some low level thermal drift after the regeneration cycle, due to heat generated during sensor regeneration. To ensure maximum sample accuracy, wait 30 minutes after a regeneration and then check the ZERO adjustment. If the display reads 0 when the ZERO button is pressed, the adjustment has been accomplished. If the display reads H or L, simply turn the ZERO ADJUST pot with the trimmer tool or small screwdriver to complete the adjustment.

Only adjust the ZERO pot after a regeneration. It is not necessary to rezero between samples since the instrument automatically erases the previous reading. If the ZERO ADJUST pot is manually turned between samples, the results will be slightly lower than the actual concentration. However, this is not a permanent problem and is corrected with a sensor regeneration.

4 INSTRUMENT OPERATION

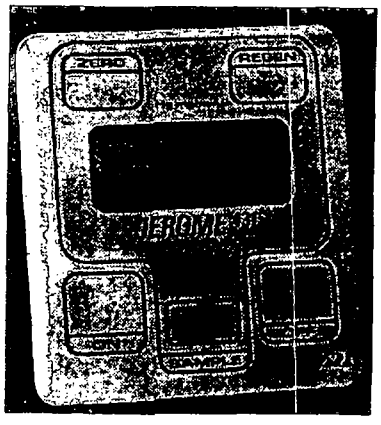
4.1 Digital Meter Display Codes

METER DISPLAY	EXPLANATION
000	Ready to sample
.000	Lack of mercury reading
00.0	Lack of mercury reading, display in nanograms (see page 18)
.8.8.8	Perform sensor regeneration (refer to page 10)
.H.H.H	Sensor regeneration in progress (.H.H.H flashes)
.L.L.L	Perform re-zero (refer to page 10)
.P.P.P	Power cord required or low line power, <100 VAC (or 200 VAC)(see page 19, Changing the Fuse, if .P.P.P remains on after the cord is connected.)
.H.L.P	High line power, greater than 130 VAC (or 260 VAC)
.LO BAT	Recharge batteries (refer to page 13)
.E.E.E	Same as LO BAT, automatically shuts off
.HL	High level, sample exceeded maximum sample limit (.999)
DURING SAMPLING	
.-	0-25% sensor saturation
.--	25-50% sensor saturation
.---	50-75% sensor saturation
-.---	75-100% sensor saturation

DURING SAMPLING	USING THE SURVEY MODE
-	Survey sampling (minus sign flashes continuously)
WHEN ZERO IS DEPRESSED	Adjust to 0 only after sensor regeneration. It is normal for the display to read H after sampling has started.
0	Zero, ready to sample
H	High, turn Zero pot counterclockwise
L	Low, turn Zero pot clockwise

4.2 Daily Operations

Before each day's use of the Jerome 431-X, perform the following four steps to verify proper instrument operation:



- Press the power ON button.
  - ➢ The digital meter displays 000. (Disregard the digital meter's initial momentary reading.) Recharge or replace the battery pack if the LO BAT indicator REMAINS ON. Refer to pages 13 and/or 17 for the procedure.
  - ➢ To ensure the instrument's electronics have stabilized, allow a 1 minute warm up before beginning the next step.
- Perform a sensor regeneration. Refer to page 10 for the procedure. Thirty minutes after sensor regeneration is complete, rezero the instrument.

NOTE:	For maximum accuracy, such as when testing with the Functional Test Kit, wait thirty minutes after the sensor regeneration cycle to rezero the unit. For emergency response, such as for spill cleanup, the unit can be rezeroed immediately after sensor regeneration

- Press the SAMPLE button.
  - ➢ During the sample cycle, the digital meter displays a bar (-) which indicates the amount of sensor saturation.
- At the end of the 12 second cycle, read the digital meter.
  - ➢ The number shown on the digital meter is the mercury concentration in mg/m^3. This value remains on the display until the next sample is taken. The digital meter automatically zeroes at the start of each sample.
- At the end of each day's use perform a sensor regeneration. DO NOT ALLOW MERCURY CONTAMINATION TO STAY ON THE GOLD FILM SENSOR OVERNIGHT.

4.3 Sensor Regeneration Instructions

A sensor regeneration is needed to clear the 431-X sensor of any accumulated mercury and to prolong the life of the sensor. This simple procedure should be done:

- At the beginning of the day on which the instrument is to be used.
- During the mercury survey, if the sensor becomes saturated.
- At the end of the day's survey, before storage.
- At a minium of every 30 days while the instrument is in storage. Regeneration once each month will prolong the life of the gold film sensor.

See the Principles section on page 7 for more details on the gold film sensor and the sensor regeneration.

AC power must be between 100-130 VAC or 200-260 VAC for the sensor to clean properly. If AC power is not between these limits, an .P.P.P or .H.L.P may appear in the display (see page 8). Refer to page 18 for voltage and frequency settings.

CAUTION: Once a sensor regeneration is initiated, DO NOT interrupt the cycle.

- Attach the power cord to the 431-X and plug it into AC power. AC power is required to thermally regenerate the sensor.
- Press the power ON button.
- Press the REGEN button.
  - ➢ The digital meter flashes .H.H.H for the duration of the 10 minute cycle and displays .0.0.0 when the cycle is completed.
  - ➢ DO NOT INTERRUPT THIS CYCLE. Wait until the cycle is completed before continuing with the next step.

NOTE:	The digital meter will read .P.P.P after REGEN is activated if the power cord is not plugged in or if the instrument's fuse needs replacing. Plug in the power cord, or if necessary, replace the fuse according to the procedure on page 19.

- While pressing the ZERO button, turn the ZERO ADJUST potentiometer using the trimmer tool until the digital meter reads 0. See the illustration on page 6 for the location of the ZERO ADJUST potentiometer.
  - ➢ If the meter reads H, turn the ZERO ADJUST counter-clockwise;
  - ➢ If the meter reads L, turn the ZERO ADJUST clockwise.

NOTE:	A minimum 30 minute wait after the sensor regeneration cycle is complete ensures maximum sample accuracy. However, the unit can be used immediately following the sensor regeneration if necessary. When the sensor regeneration is complete, press ZERO and adjust the ZERO ADJUST pot until 0 appears on the display. Install the zero air filter in the intake and take several samples or lock the instrument into survey mode (see page 12). After approximately one minute, stop sampling and check the ZERO. Adjust to 0. Repeat sampling through the zero air filter until sensor remains on 0.

NOTE: Depending upon internal configuration, a number between 00 and 100 may appear on the display, instead of H, L, or O when zero is pressed. See Internal Dip Switch Settings, page 18, for details. **IMPORTANT: Do not turn the ZERO ADJUST potentiometer between samples.** Turn the ZERO ADJUST only after a sensor regeneration cycle otherwise invalid readings will result.

- Press the power OFF button and disconnect the power cord.
- The Jerome 431-X is ready for sampling.

4.4 Sample Mode

This mode, used for standard operation, produces optimum accuracy (+/-5% at 0.100 mg/m^3 Hg) with the Jerome 431-X.

- Press the power ON button.
  - ➢ The digital meter displays 000. If the unit is set to display in ng, the digital meter displays 00.0. (Disregard the digital meter's initial momentary readings.) Recharge or replace the battery pack if the LO BAT indicator REMAINS ON. Refer to pages 13 and/or 17 for the procedure.

- To ensure the instrument's electronics have stabilized, allow a 1 minute warm up before beginning the next step.

- Press the SAMPLE button.
  - ➢ During the sampling cycle, the bar (or bars) shown on the digital display indicate the current percentage of sensor saturation. (Refer to Meter Display Codes, page 8, for code descriptions.)
  - ➢ The bar (or bars) flash after 2 seconds and again after an additional 7 seconds. This flashing signals the opening and closing of the solenoid sample bypass. (See the Principles of Operation on page 7 for details.)

- At the end of the 12 second cycle, read the digital meter.
  - ➢ The number shown on the digital meter is the mercury concentration in mg/m^3 (or ng). This value remains displayed until the next sample is taken. The digital meter automatically zeroes at the start of each sample.

- When the sensor is completely saturated, the digital meter displays .8.8.8 instead of a value. No further operation is possible until a sensor regeneration is performed. (Refer to page 10 for the Sensor Regeneration procedure.)

- Press the power OFF button when not in use. Install the zero air filter in the instrument intake during storage.

4.5 Sampling Notes

- The Jerome 431-X is intended for vapor use only. DO NOT allow the probe or the instrument's intake to come in contact with liquids, dust or other foreign material. Moisture or liquids drawn into the instrument can damage the sensor and flow system.
- The Jerome 431-X operates a minimum of 6 hours on a fully charged battery.
- Use the probe (AZI P/N1400-2002) to locate mercury vapor in hard to reach places. Plug the probe directly into the instrument's intake.

4.6 Survey Mode

The survey mode takes samples every 3 seconds automatically. Use this mode to locate mercury spills or to assess areas of potentially high mercury concentrations. Sampling in the survey mode is not as accurate. Due to the decreased sample volume, the accuracy of the instrument is reduced to +/- 20% @ .100 mg/m^3.

- Press the power ON button.
  - ➢ The digital meter displays 000. If the unit is set to display in ng, the digital meter displays 00.0. (Disregard the digital meter's initial momentary readings.) Recharge or replace the battery pack if the LO BAT indicator REMAINS ON. Refer to pages 13 and/or 17 for the procedure.
  - ➢ To ensure the instrument's electronics have stabilized, allow a 1 minute warm up before beginning the next step.

- Press and hold the SAMPLE button.
  - ➢ The instrument takes a normal 12 second sample, displays the concentration at the end of the cycle and then goes into the survey mode sampling every 3 seconds. The display flashes the measured concentrations at the end of each 3 second sample cycle.

- When you are finished surveying, release the SAMPLE button.
  - ➢ The final survey value remains displayed until the next sample is taken.

NOTE:	Approximately 65 samples at .1 mg/m^3 may be taken before a sensor regeneration is required.

- To lock the instrument in a survey mode:
  - ➢ Hold the SAMPLE button down until the sensor status indicator bar(s) "_" begins flashing on the display.
  - ➢ Press the ZERO button, then release the SAMPLE button.
  - ➢ The pump should continue to run and the display should update every 3 seconds.
  - ➢ The instrument remains in the survey mode until one of the following occurs:
    - ➢ The sensor is saturated
    - ➢ A LO BAT (low battery) signal is encountered
    - ➢ An HL (high mercury level) is encountered
    - ➢ The instrument is turned OFF.

- Press the power OFF button when not in use.

4.7 Operating on AC Power or Generator

For stationary use, the 431-X may be operated on AC power. Operating the instrument only on AC power eliminates the need for the battery pack and its necessary maintenance. If preferred, the battery may be unplugged or removed completely.

When using a generator to power the Jerome 431-X, it is important that the generator is capable of maintaining a constant voltage output. This is especially true during the sensor regeneration. Use a high quality line conditioner or voltage regulator to prevent damage to the electronic components and the sensitive gold film sensor.

4.8 Operating on Internal Battery Power

Battery power allows use of the Jerome 431-X as a portable instrument. If battery power is necessary for use, please be aware of the following:

- A fully charged battery pack (AZI P/N Z4000-0907) provides power for a minimum of 6 hours of operation.
- For operating more than 6 hours, an extra fully charged battery pack is needed.
- Complete battery recharging takes 14 hours. Refer to page 13, Charging Batteries for instructions if needed.
- The 431-X use a rechargeable NiCad battery. Dispose of the old battery properly when it is replaced with a new one.
- External battery power: A special version of the Jerome 431-X is available that can be operated from a secondary DC source, such as a battery used in conjunction with solar panels. Contact AZI for additional information.

4.9 Charging Batteries

- Press the power OFF button.
- Attach the power cord to the 431-X and plug it into AC power.
- *Complete battery recharging takes 14 hours.*
- *The 431-X contains a trickle charger so it may be continually plugged into an AC power source without damaging the battery pack.*

NOTE:	To charge the batteries outside of the instrument, use the IDC Battery Charger (AZI P/N 4000-1011, for 115 VAC, P/N 4000-1012, for 230 VAC).

4.10 Obtaining Maximum Battery Life

There are certain inherent limitations to NiCad (Nickel Cadmium) batteries. The primary limitation is a memory effect that occurs when the batteries are partially discharged and then recharged, repeatedly. This memory leads to a drastic reduction in the usable battery life. To prevent this memory effect, periodically allow the battery pack to discharge completely, then recharge the battery pack.

- For maximum battery life, follow these 3 steps:
  - ➢ At least once a month wait until LO BAT appears on the digital meter before recharging the battery pack.
  - ➢ Charge the battery pack when the LO BAT indicator comes on. Excessive discharge can damage the battery pack. Before storing the instrument verify the power is OFF.

- When batteries fail to hold a charge, the battery pack should be replaced. Battery life under normal usage is approximately 1 year, depending on the number of charge and discharge cycles.

5 MAINTENANCE

5.1 Preventive Maintenance Calendar

To keep the Jerome 431-X operating at peak performance, follow the maintenance schedule below. Use this schedule as a guideline only, as maintenance is more a function of application and amount of use, rather than time.

ALWAYS install the zero air filter into the instrument's intake during storage.

PART/COMPONENT	MAINTENANCE CYCLE	PAGE
Charge batteries	At least once per month, after one month's storage, or when LO BAT appears	13
Change .25mm fritware	Weekly or as needed	15
Change internal filters*	After 6 months of use or as needed.	16
Replace zero air filter*	Annually	16
Factory calibration	Annually	19
Calibration check	Monthly or as needed	29
Replace batteries	Annually or as needed The battery pack contains NiCad batteries. Dispose of them properly.	17

* C/M filters contain Mallcosorb™, Scrubber filters and zero air filters contain Resisorb™. For safety information, see the supplier's Material Safety Data Sheets (MSDS) or call AZI Customer Service at 1-800-528-7411 or 1-602-470-1414 for assistance in obtaining the MSDS. Dispose of all filters properly.

5.2 Flow System

The Jerome 431-X's flow system is the crucial link between the sensor and the sample. For the instrument to perform correctly, the flow system must be properly maintained. The user maintainable components of this system are the intake filter (.25 mm fritware), a C/M filter, two scrubber filters and connecting tubing.

Check the Preventive Maintenance Calendar, page 14, for a suggested schedule for changing filter disc and filters. The Tygon™ tubing in the system must be free of crimps for proper flow.

- Replace the .25mm Fritware once each week. In dusty environments, the fritware may need replacement as often as once a day. Replacement .25mm Fritware are available from AZI, Technical Sales, 1-800-528-7411 or 1-602-470-1414, (see Accessories & Maintenance Parts on page 25).
  - ➢ Unscrew and remove the intake from the instrument.
  - ➢ Push the old fritware disc out using your trimmer tool.
  - ➢ Use tweezers to insert the new fritware. Avoid touching the new fritware disc with fingers.
  - ➢ Use the blunt end of the trimmer tool to seat the fritware disc firmly against the inner ledge of the intake.
  - ➢ Screw the intake back on the instrument.

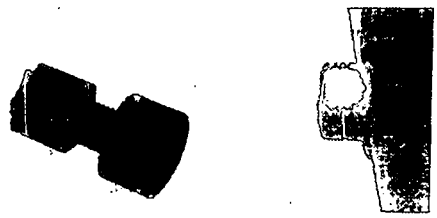


Figure 4 Remove Intake

Figure 6 Remove Fritware

Figure 5 Insert Fritware

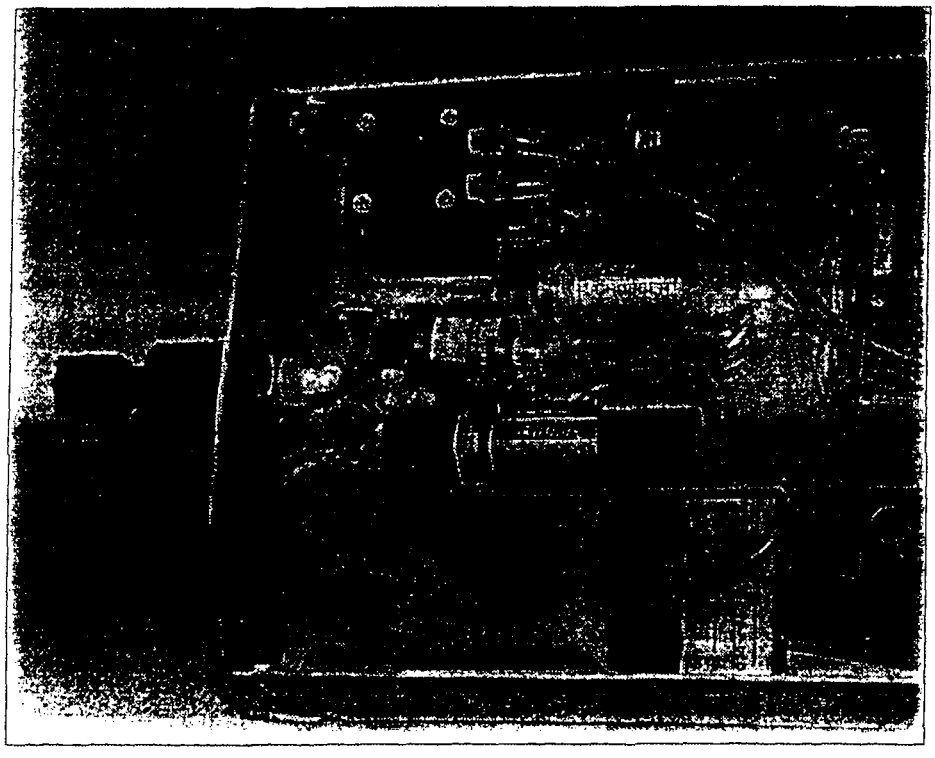
CAUTION

The air flow intake, on the front of the instrument, must be securely held in place. If it is loose, the tubing inside the instrument can become twisted when the intake housing is replaced. On older instruments, it may be necessary to open the instrument and tighten the retaining nuts from the inside. Call AZI Customer Service at 1-800-528-7411 or 1-602-470-1414 if you have any questions.

5.3 Internal Filters

Replace the internal filters system (one C/M filter and two scrubber filters) after six (6) months of use, or as needed. (See Troubleshooting section, page 20.)

- Press the power OFF button and unplug the power cord.
- Remove the 2 side screws from the intake end of the instrument and open the case.
- Carefully disconnect the Tygon™ tubing from both ends of the filters and discard the old filters.



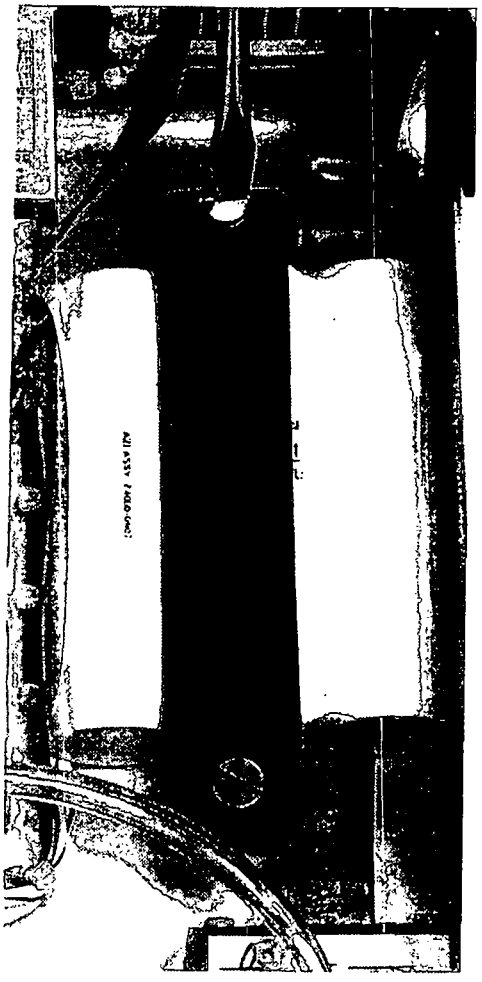
CAUTION:

The Series C/M filter contains Mallcosorb™ and the Scrubber Filters contain Resisorb™. Both types contain trace amounts of mercury. For safety information, see the supplier's Material Safety Data Sheets (MSDS) or call AZI Customer Service at 1-800-528-7411 or 1-602-470-1414 for assistance in obtaining the MSDS. Use proper disposal methods for all filters.

- Connect the new filters to the Tygon™ tubing, ensuring all filter straight nipples point toward the intake and elbows point according to the illustration.
  - ➢ Push the Tygon™ as far as it will go onto the filter fittings.
- Push the filters into the mounting clips.
- Remove any crimps in the tubing and ensure that tubing connections are secure.
- Close the case and replace the screws.
- Dispose of all filters in accordance with State and Federal EPA Regulations.

5.4 Replacing Battery Pack

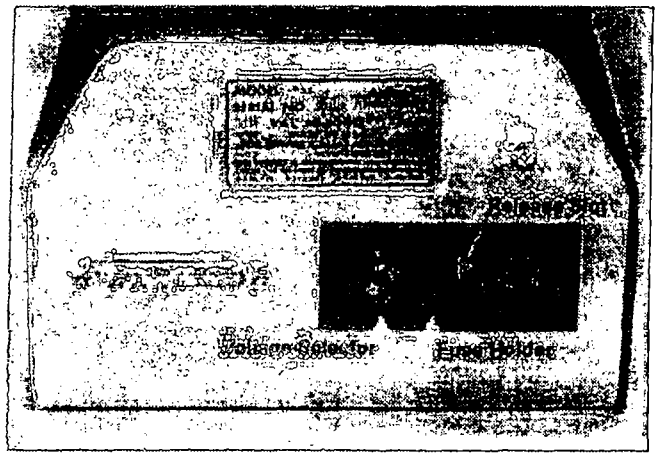
- Press the power OFF button.
- Unplug the power cord.
- Remove the screws, one on each side, from the intake end of the instrument and open the case lid.
- Disconnect the battery connector from the board.
- Loosen the two (2) captive screws holding the battery bracket and remove the bracket.
- Remove the old battery pack and replace it with a new battery pack.
- Replace the battery bracket and tighten the captive screws.
- Connect the new battery connector to the board.
- Close the case and replace the screws.
- Dispose of the old NiCad battery properly, in accordance with State and Federal EPA Regulations.



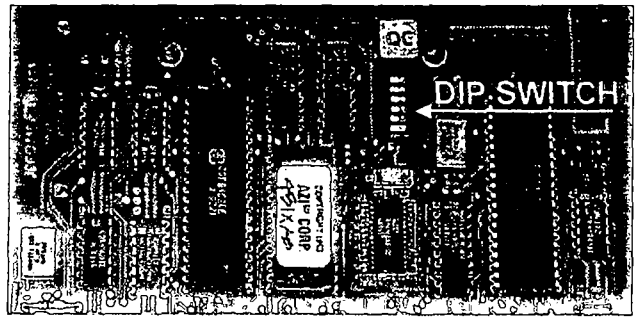
6 INTERNAL DIP SWITCH SETTINGS

6.1 Input Voltage and Frequency

This instrument has been factory set and calibrated to use the input power setting, either 110 VAC, 60 Hz, or 220 VAC, 50 Hz, requested when the instrument was ordered. The settings are easily changed using the switch in the Power Inlet Module for voltage and the internal DIP switch setting for frequency.



- Ensure the instrument is turned OFF and unplugged.
- Locate the power receptacle on the rear of the instrument.
- Insert a small screwdriver in the voltage selection slot and turn the selector until the arrow points toward your setting choice and a click is heard.
- Turn the instrument off.
- Remove the two screws near the front of the instrument and open the lid.
- Locate DIP Switch SW2 at the top of the main circuit board. See figure at right.
- Set DIP Switch SW2 position #1 and #6 as follows.



	60Hz	50Hz
Position #1	OFF	OFF
Position #6	OFF	ON

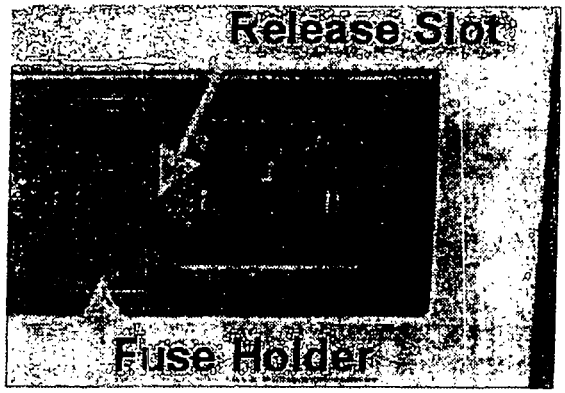
6.2 Displaying Nanograms or Milligrams/Cubic Meter

The instrument is factory set to display mg/m^3 (milligrams per cubic meter) Hg (.XXX). For some applications, including dosimeter analysis, the instrument's display can be converted to display nanograms.

- Turn the instrument off. Remove the two screws near the front of the instrument and open the lid.
- Locate DIP Switch SW2 at the top of the main circuit board. See figure above.
- Place position #2 to OFF for Nanogram display.
- Return position #2 to ON for Milligram display.

6.3 Changing the Fuse

If the instrument display reads .P.P.P when the instrument is connected to AC power or when REGEN is pressed, or if the battery will not charge, the fuse may need to be replaced. The AC line power could also be less than 100 VAC (220 VAC). Check the fuse with an ohm meter and/or the AC line power with a voltage meter.



- Locate the power receptacle on the rear of the instrument.
- Insert a small screwdriver in the slot and gently slide the fuse compartment out.
- Check the fuse held in the open sided clip and if it is bad, replace it with the spare fuse held in the sliding spare fuse compartment. Discard the bad fuse.
  - ➢ When it is convenient, replace the spare fuse with another 1A 250V Fast-Blo fuse (AZI P/N 5100-1012).
- Replace the fuse compartment in the power receptacle.

7 CALIBRATION

The Jerome 431-X's gold film sensor is inherently stable and does not require frequent calibration. The interval between calibrations depends upon the application and frequency of use; however, the recommended minimum or maximum interval is every 12 months.

The Jerome 431-X has been factory calibrated using NIST traceable permeation tubes. In order to calibrate the Jerome 431-X, a sophisticated calibration system is required that ensures stability of the calibration gas source, eliminates any pressure in the calibration gas stream and controls the temperature of the calibration environment. Calibration also requires special proprietary software. This system has an estimated uncertainty of ±3.5%.

We strongly recommend you take advantage of our calibration and maintenance service at Arizona Instrument. A certificate of calibration is issued by Arizona Instrument when your instrument is factory calibrated to 0.100 mg/m^3. Two additional calibration points, 0.010 and 0.025 mg/m^3, are available upon request at additional charge. Contact Arizona Instrument Customer Service at 1-800-528-7411 or 1-602-470-1414 for assistance in obtaining calibration.

7.1 Verification of Calibration and Quality Control

The Functional Test Kit, AZI P/N 4431-0902, is used to determine if your instrument is within calibration tolerances between recommended annual factory calibrations. If your application requires frequent verification of instrument function, this test demonstrates the unit's operation, calibration, and function. This test verifies proper instrument operation through the introduction of a known mass of mercury into the Jerome analyzer and allows you to have complete confidence in the sample results.

Recording the Functional Test Kit results in an instrument log provides a quality control/quality assurance record of instrument function between regular calibrations. As long as the test results fall within the expected range, you may assume the instrument is functioning correctly.

THIS FUNCTIONAL TEST DOES NOT CALIBRATE THE INSTRUMENT.

A complete description of the Functional Test Kit procedures is contained in Appendix A, beginning on page 29.

To order the kit, contact Arizona Instrument Technical Sales at 1-800-390-1414 or 1-602-470-1414.

8 431-X TROUBLESHOOTING

Symptom	Possible Cause	Solution
Power Problems		
Unit does not turn ON. LCD displays 000 when connected to power cord and ON button is pressed.	Dead battery	Recharge battery (minimum 14 hours) refer to page 13. Replace battery, refer to page 17.
Unit does not turn on when connected to AC power cord.	Fuse Insufficient power	Replace fuse, refer to page 19 . Be sure there is power to the AC outlet using a volt meter.
Regeneration & Zero problems		
LCD displays .8.8.8.	Sensor saturated	Do not attempt to rezero. Unit must be regenerated. See page 10 for information.
LCD displays .L.L.L when taking first sample.	Changes in temperature	Readjust zero pot. See page 6 for information .
LCD displays H at finish of sensor regeneration	Internal contamination may redeposit mercury from flow system onto gold film sensor.	Remove and replace intake filter disk, Tygon™ tubing and internal C/M filter. Check tubing for kinks or crimps. Repeat regeneration cycle. See page 10 for information.
Zero adjust pot cannot be adjusted to 0	Pot not turned sufficiently	Turn zero adjust up to 20 times to reach the end. Pot will "click" softly.
Display still unchanged	Sensor may be ruptured or pot may be broken	Turn pot slowly in opposite direction till display reads 0. If still unchanged, call AZI Customer Service.

Symptom	Possible Cause	Solution
Sampling Problems		
Air flow is restricted during the sensor regeneration cycle, causing possible permanent damage.	Kinks and crimps in the Tygon™ tubing.	Periodically check the Tygon™ tubing inside the instrument.
High erratic results	Internal mercury contamination	1. Install zero air filter in intake and tighten intake nut. Press SAMPLE button. After 3 samples, if readings are over .003 mg/m^3, replace intake filters and Tygon tubing. 2. Perform a REGEN with zero air filter in intake. See page 10 for information. Retest if necessary. Replace intake filters and Tygon™ tubing.
High/erratic results Readings vary more than 0.05 when in survey mode.	Film connection	Press and hold SAMPLE button for 12 seconds in clean area or with zero air filter in intake. Move unit from side to side, or up and down during sample cycle. Call AZI Customer Service.
Low response or erratic readings after a long period of non-use	May need a second regeneration cycle.	Wait 20 minutes between regeneration cycles. Test with FTK. See page 29 for information. If still unresponsive, call AZI Customer Service.
False readings, may go to .8.8.8 or .L.L.L	Extremely cold or extremely warm air sampled into unit	If sampling under these conditions, install zero air filter in intake. Sample until display reads .003 mg/m^3 or less. This equilibrates sensor temperature with the temperature of the sample air stream. Remove filter and take samples.
High/erratic results	Intake and internal filters may get clogged and need replacement when sampling in a dusty area	Open instrument to check for pinched, crimped or disconnected internal tubing. In extreme conditions a particle filter may be installed on intake.

9 JEROME 431-X TECHNICAL SPECIFICATIONS

Range	0.001 to 0.999 mg/m^3
Sensitivity	0.003 mg/m^3 Hg
Precision	5% relative standard deviation @ 0.100 mg/m^3 Hg
Accuracy	+/- 5% @ 0.100 mg/m^3 Hg
Response time-sample mode	12 seconds
Response time-survey mode	3 seconds
Flow rate	750cc/min (0.75 liters/min)
Power requirements	100-130 VAC or 200-260 VAC, 50 Hz or 60 Hz, 115 watts maximum
Batteries	Rechargeable Nickel Cadmium
Fuse	1A 250V 5 X 20 Fast Blo
Construction	Aluminum alloy
Dimensions	15 cm x 33 cm x 10 cm (6" w x 13" l x 4" h)
Weight	3.18 kilos (7 pounds)
Digital meter	Liquid crystal display (LCD)
Operating environment	0° - 40°C, non-condensing, non-explosive

9.1 Optional "Communications" Version

Alarm output	30V DC, 100mA
Dosimeter power output	For dosimeter analysis
Data output	1. Digital, Serial, RS232, Baud Rate 1200 for use with Data Logger, Base Station, and/or JCI program software 2. Digital, Serial, RS232, data format, but with driver for 20mA capability and 0 & 20mA logic levels; Baud Rate 1200 (special industrial applications)

"OPTION BOARD"

Data output	0 - 2V or 4 - 20 mA
Auto sample interval	5, 15, 30, 60 minutes
Auto regeneration interval	6, 24 or 72 hours

9.2 Instrument I/O Interface

The 431-X I/0 port (25 pin D-sub) has six functions:

- Serial communication channel, RS-232

Pin 1	Protective ground	➢ Interface type RS-232C full duplex, DCE
Pin 2	Data in	
Pin 3	Data out	➢ Communication parameters - 1200 Baud, 1 start bit, 8 data bits, 2 stop bits, no parity
Pin 7	Data ground	

- Serial communication channel, 20mA current loop

Pin 1	Protective ground	➢ Interface type: 20mA current loop, full duplex
Pin 4	Data out (+)	
Pin 5	Data in (+)	➢ Communication parameters - 1200 Baud, 1 start bit, 8 data bits, 2 stop bits, no parity
Pin 14	Data out (-)	
Pin 16	Data in (-)	

- Alarm output

Pin 9	Switched battery+	➢ Maximum voltage, 30 VDC
Pin 10	Alarm output (open collector, active low)	➢ Maximum current 100mAmp
Pin 7	Battery ground	
Pin 23	Battery ground	

- Dosimeter power

Pin 22	Dosimeter enable	➢ Voltage, 24 - 28 volts AC
Pin 23	Battery ground	➢ Connecting pin 22 to 23 enables the dosimeter desorption cycle.
Pin 12 & 24 Tied together	Dosimeter power	
Pin 13 & 25 Tied together	Dosimeter power	

- Switched battery connection for data logger

Pin 9	Battery +
Pin 7	Battery ground
Pin 23	Battery ground

- Unswitched battery connection for external battery pack pin assignments

Pin 15	Battery +
Pin 19	Battery +
Pin 7	Battery ground
Pin 23	Battery ground

NOTE: Pins 6, 8, 11, 17, 18, 20 and 21 should not be connected.

9.3 Potential Interferences

Potential interferences to the Jerome mercury vapor analyzers are rare and most of these can be eliminated with proper maintenance procedures. However, erroneously high readings can sometimes occur. Here are a few things to be aware of when using the instruments.

The gold film sensors used in the Jerome mercury vapor analyzers do not respond to the following compounds:

- Hydrocarbons
- CO, CO_2, and SO_2
- Water vapor (Note that water vapor condensation on the gold film can cause irreparable harm to the sensor and must be avoided.)

The acidic gas filter, contained in the internal filter system, removes the following compounds that cause the gold film sensor to respond:

- Chlorine
- NO_2
- Hydrogen Sulfide (H_2S)
- Most mercaptans (organic sulfur compounds or "thiols")

In areas containing these highly volatile compounds, the filter can become quickly saturated. In such situations, it is recommended that these gases be allowed to dissipate before sampling for the less volatile, more persistent mercury vapor. A special filter designed to remove chlorine gas is available from Arizona Instrument and may be ordered as Chlorine Filter, AZI P/N Z2600-3940. Collection of air samples with Jerome gold coil dosimeters for analysis by the Jerome mercury vapor analyzers will also eliminate interferences.

Ammonia in very high concentrations can cause an offgassing of accumulated acidic fumes from the internal acidic gas filter, resulting in positive readings on the instrument. In these cases, the ammonia odors are very strong. Again, either allow the vapors to dissipate or use the dosimeters. Filter replacement at regular intervals, or when unexpectedly high readings are encountered in areas of these potential interferents, may resolve these problems.

Volatile mercury compounds in general will cause the gold film to respond. Alkyl organic mercuries such as methyl mercury (and other "straight chained" compounds) are typically extremely volatile and change the electrical resistance of the gold film sensor. Any such responses should be considered "qualitative," not quantitative. The instruments are designed and calibrated to elemental mercury vapor only.

Inorganic mercury salts such as mercuric chloride are not very volatile. They may, however, generate some minute level of elemental mercury vapor to which the instruments will respond. This response, again, should be considered a qualitative response only.

10 ACCESSORIES & MAINTENANCE PARTS

Accessory Kit, Y431-0901

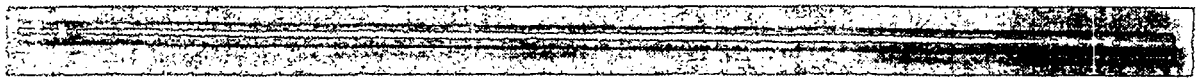


Figure 12 Probe, 1400-2002

Figure 15 Tubing Adaptor, 1400-3010

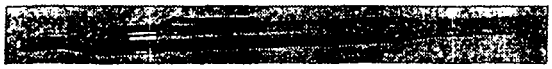


Figure 14 Trimmer Tool, 2300-0001

Figure 13 Fritware, .25", 2600-3039

Figure 16, 115 VAC Line Cord, 6000-4003

This Manual, SS-086

Functional Test Kit, Y431-0902

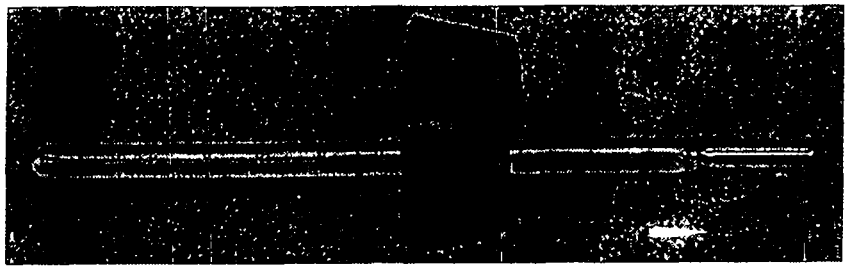


Figure 18 Stopper Assembly, A2600-0902

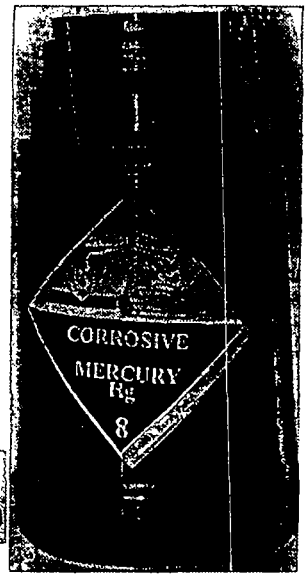


Figure 17 Calibration Vessel, Thermos, 2600-0030

Figure 19 Mercury Vial, A2600-0904

Figure 20 Septa (20), 3200-0011

Figure 21 Septum Holder, Z2600-3914

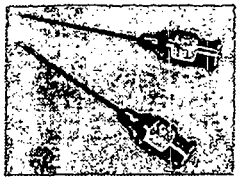


Figure 23 Syringe Needles, 22 Ga. Reusable, 2600-0022

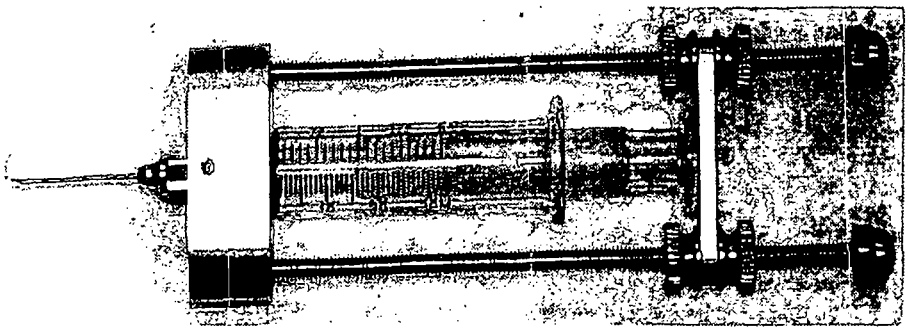


Figure 22 Syringe Assembly, A2600-0903

Maintenance Kit, Y431-0903

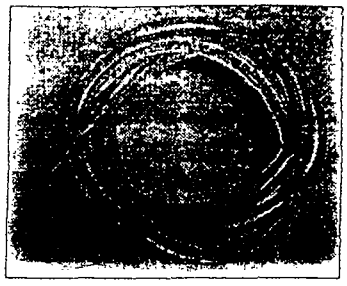


Figure 25
Tygon Tubing, 1/8",
2500-3001

Figure 26 Fritware, .25
Dia. 2600-3039

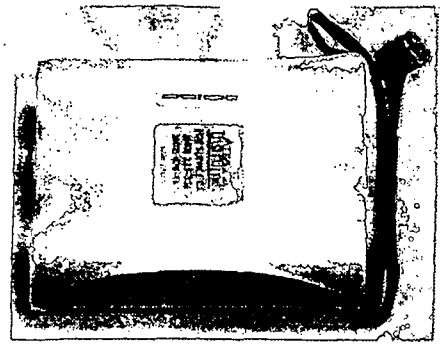


Figure 24 021 Battery Pack,
Z4000-0907

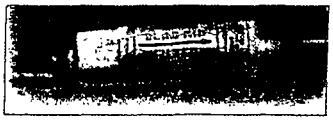


Figure 28 Zero Air Filter,
Z2600-3905

Figure 29 Scrubber Filter,
Z2600-3930

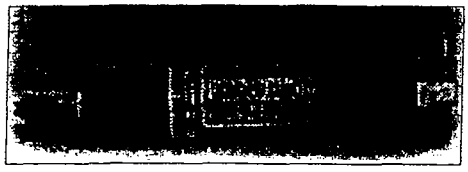


Figure 27 C/M Filter, Z2600-3926

Dosimeter Analysis Kit, Y431-0905

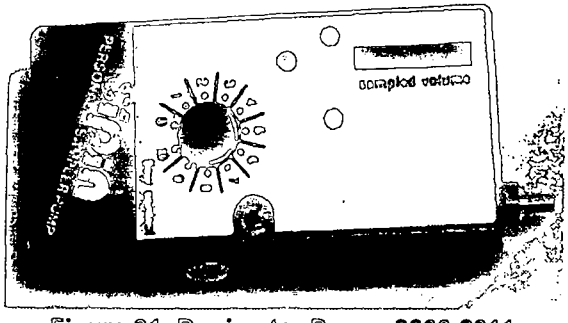


Figure 31 Dosimeter Pump, 2600-2011

Figure 32 Dosimeter
Pump DC Supply
4000-1023 - 115VAC
4000-1024 - 230 Euro

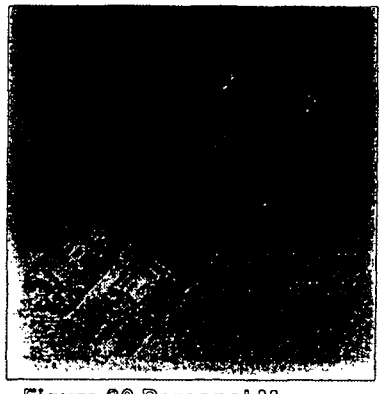


Figure 30 Personal Mercury
Dosimeter,
X4321-0901

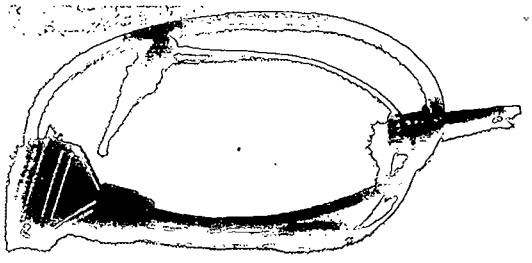


Figure 34 Dosimeter Lead Set,
2100-6017

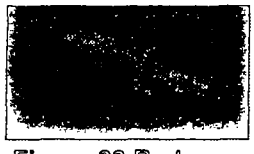


Figure 33 Reducer,
1/8" X 3/16"
1300-0031

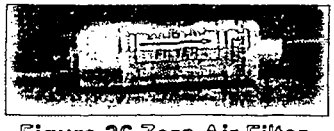


Figure 36 Zero Air Filter,
Z2600-3905

Figure 35 Tygon Tubing,
3/16", 1', 2500-3010

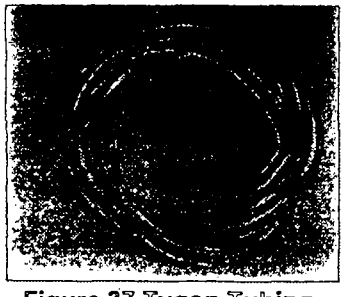


Figure 37 Tygon Tubing,
1/8", 2', 2500-3001

In addition to the parts listed in kits, which may be ordered individually, the following accessories and replacement parts are available.

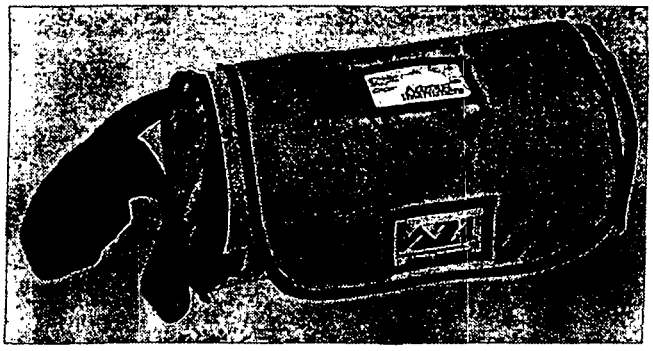


Figure 38 Field Carrying Case, Y1400-0052

The custom designed Field Carrying Case holds the Jerome 431-X, Personal Mercury Dosimeters, Filters, Tygon Tubing, Probe, and Power Cord in foam padded safety. Openings allow full use of the instrument without removing it from the case.

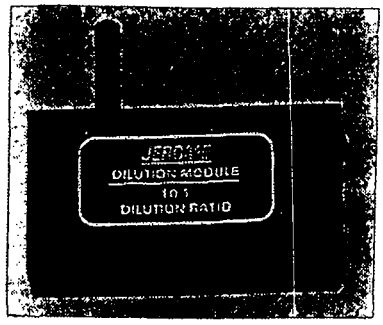


Figure 39
10:1 Dilution Module, Z2600-3911

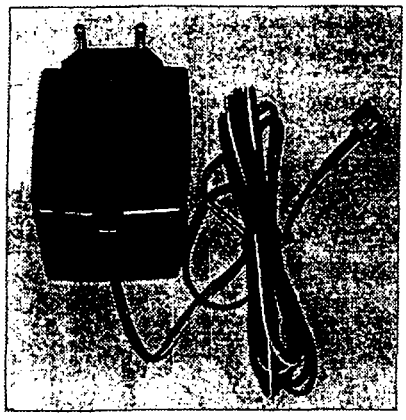


Figure 41
230 VAC Battery Charger, 4000-1012

Figure 40
115 VAC Battery Charger, 4000-1011

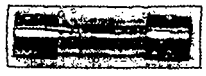


Figure 42
Fuse, 1 Amp, 250V, 5100-1012

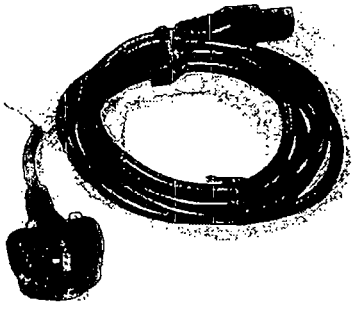


Figure 43 Line Cord, 220 VAC, British, 200-0003

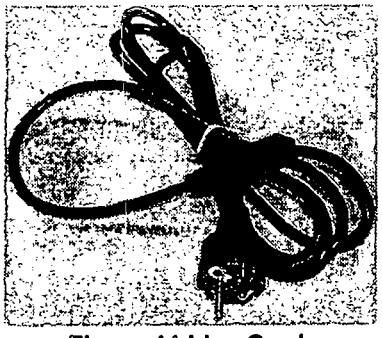


Figure 44 Line Cord, 220 VAC, Continental Europe, 200-0008

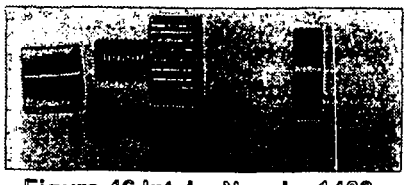


Figure 46 Intake Nozzle, 1400-3009

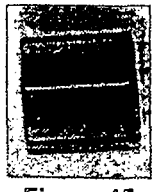


Figure 45
Tube Nut
PS-151

Figure 47 Chlorine Filter, Z2600-3940

Available but Not Shown

Y411-0904	031/411 Carrying Case Assembly, Holds the Jerome 431-X, Personal Mercury Dosimeters, and Accessories in foam padded safety
Y031-0902	DC Power Kit - available on special order

For current prices and delivery information, call AZI Jerome Sales at 602-280-1414 or 800-528-7411.

11 FACTORY CALIBRATION SERVICE

Service includes filter replacement, component check and calibration and instrument calibration to NIST traceable standards. A certificate of calibration is issued by Arizona Instrument when your instrument is factory calibrated to 0.100 mg/m^3. Two additional calibration points, 0.010 and 0.025 mg/m^3, are available upon request at addition charge.

Loaner instruments are available upon request. For pricing, availability, and scheduling, call AZI Customer Service at 800-528-7411 or 602-470-1414. You may use e-mail at support@azic.com

12 APPENDIX A , 431-X FUNCTIONAL TEST

If your application requires frequent verification of instrument functionality, this test will benefit you. If the test results fall within the expected range, you may assume the instrument is functioning properly. This test does not calibrate the instrument.

NOTE: Perform the functional test ONLY after a sensor regeneration.

The 431-X Functional Test Kit contains all accessories necessary to perform the functional test. See the complete list on page 25 and verify that all the parts to the kit are present.

CAUTION:

The vial and thermometer contain liquid mercury and are possible sources of mercury contamination. Follow the instructions for handling or transferring the mercury into the Functional Test Kit Vessel carefully.

For safety information, see the supplier's Material Safety Data Sheets (MSDS) or call AZI Customer Service at 1-800-528-7411 or 1-602-470-1414 for assistance in obtaining the MSDS.

12.1 Preparation

- Carefully unpack and inspect the parts of the kit.
- ENSURE that the mercury shipping container and mercury filled thermometer are not broken.
- In a ventilated area, preferably under a fume hood, remove the mercury vial from its shipping container.
- Place the functional test kit vessel and the mercury vial close to each other and open the mercury vial.

CAUTION:

The edge between the plastic case and the glass inner vessel of the functional test kit vessel are not sealed enough to prevent mercury from entering the area between the inner and outer vessels. ENSURE the mercury, handled in the next step, does not come in contact with the seal where the glass and plastic portions join.

NOTE: The vessel may be disassembled to transfer the mercury and better prevent contamination of the outer portion of the vessel. Instructions to disassemble the vessel can be found on page 30.

12.2 Mercury Transfer

- CAREFULLY pour the mercury into the center of the functional test kits vessel's opening.
  - ➢ ENSURE that no mercury residue is on the outside of the vessel. See the supplier's Material Safety Data Sheets (MSDS) or call AZI Customer Service at 1-800-528-7411 or 1-602-470-1414 for for clean-up instructions.

- INSTALL the stopper assembly into the functional test kit vessel carefully, to prevent breakage of the thermometer.
  - ➢ PRESS the stopper assembly into the vessel to achieve a good seal.

- USE the 431-X instrument to verify that the outside of the vessel is not contaminated and the mercury vapor emission level, if any, is below the OSHA TLV for mercury.

- ALLOW the kit to adjust to room temperature for at least two (2) hours before using.
  - ➢ The temperature range for the test is 18-22°C. Avoid temperature fluctuations.

> CAUTION:
> Do not use the calibration vessel as a portable container. If the calibration vessel is upset or greatly agitated, mercury droplets will cling to the thermometer stem, the rubber stopper, the mouth of the calibration vessel and the needle guide.

12.3 Vessel Disassembly

> CAUTION:
> The inner portion of the vessel is made of glass. Handle the vessel carefully to prevent breakage.

- LOOSEN, BUT DO NOT REMOVE the base of the vessel. The base unscrews from the body.

- SET the vessel on a firm surface.

- HOLD the base stationary and unscrew the body from the base.

- HOLD the base and the inner glass vessel with one hand while removing the body and gasket with the other hand.

- After the mercury is transferred into the glass inner vessel, reassemble in the reverse order.

12.4 Replacing Mercury

An oxide coating will form on the drop of mercury and will cause lower readings in your testing. Gently swirl the vessel to disturb the outer oxidized surface of the droplet. If this does not restore higher readings, it may be necessary to replace the mercury.

- Carefully remove the stopper assembly from the calibration vessel.

> CAUTION:
> **BE SURE NEEDLE GUIDE IS FREE OF LIQUID MERCURY.**

- Carefully pour the mercury into a disposal vessel. Refer to Vessel Disassembly Instructions on page 30.
  - ➢ Mercury can become trapped between the plastic calibration vessel and the glass inner-liner.

- Replace the oxidized mercury with approximately ½ cc fresh mercury. (AZI P/N A2600-0904)
  - ➢ Do NOT use the syringe for measuring liquid mercury. Dispose of oxidized mercury properly.

- Reassemble the calibration vessel.

- Reinstall the stopper assembly.

12.5 Syringe Technique

- Pull and hold the syringe plunger against the bar-stop.
- Verify that the black mark on the syringe plunger aligns with the 1cc mark on the syringe barrel.
  - ➢ If it does not, the holder assembly must be adjusted. Call AZI customer service at 602-281-1745 or 800-528-7411 for assistance.

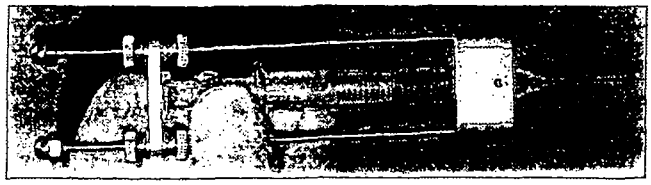


Figure 48 Check Calibration

- Insert the needle into the needle-guide of the bottle stopper.

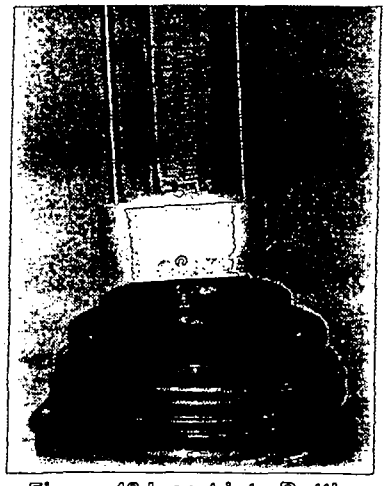


Figure 49 Insert into Bottle

- Operate the plunger two or three times to pump mercury vapor into the syringe. On the final stroke, pull and hold the plunger against the bar-stop.
- Holding the plunger against the bar-stop, remove the syringe from the bottle and move it to the septum attached to the instrument intake.

Figure 50 Hold plunger against Bar-Stop

- Continue to hold the plunger against the bar-stop and insert the syringe needle into the septum.

- Press "SAMPLE" on the instrument.

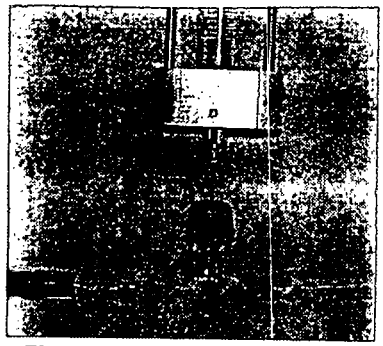


Figure 51 Insert needle into septum

- When the display flashes, release the plunger and allow gravity to feed the mercury vapor into the airstream. If the plunger stops, gently press it completely closed.

- Remove the syringe needle from the septum.

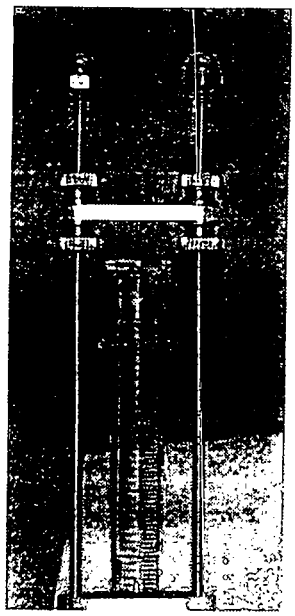


Figure 52 Gravity pulls plunger down

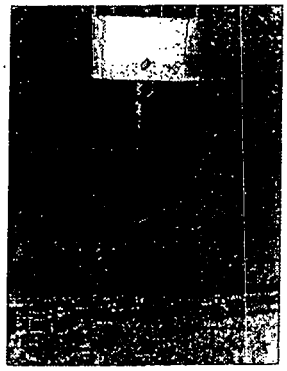


Figure 53 Remove Needle from septum

12.6 Functional Test Procedure

NOTE: Perform the functional test ONLY after a sensor regeneration.

- Allow the calibration vessel to remain stable at room temperature for at least 2 hours.
  - ➢ The temperature range for the test is 18° - 22°C.
  - ➢ Temperature fluctuations during the test procedure will produce erratic results.

- Replace the .25mm fritware.
  - ➢ Refer to page 15 for instructions.

- Replace the septum on the septum holder assembly.

- Plug the tubing adapter end of the septum assembly into the instrument's intake and tighten the intake tube nut.

> NOTE: To check for a tight seal, gently pull on the septum holder assembly. If it comes out of the intake, it may be necessary to remove the intake tube from the instrument and firmly press the tubing adapter through the intake. Tighten the intake tube firmly to the intake stem.

- Attach a zero air filter to the septum assembly.

- Press power ON.

- Take 3 samples.
  - ➢ If the average meter reading is greater than .005, stop here. The instrument may be contaminated. See the Troubleshooting section, page 20.
  - ➢ If the average meter reading is less than .005, continue to the next step.

- Note the temperature of the calibration vessel.

- Press the SAMPLE button, wait 2 seconds and when the display flashes, inject 1 cc of mercury vapor according to the syringe technique described on page 32. Be sure all mercury vapor has been injected before the solenoid closes (second click and display flash).

> CAUTION:
> Carefully follow these instructions to minimize error.

- Record the meter reading.

- Repeat the instructions for mercury injection three more times.
  - ➢ The readings obtained for the last three 1cc injections should be within +/- 5% of each other.

- Refer to the Temperature Conversion Chart, page 35, for the acceptable range.
  - ➢ The average of the last three readings should fall within the range shown on the chart.

If the average is within range, the JEROME 431-X is functioning properly.

- If the last three readings are not within +/-5% of each other,

  - ➢ Perform a sensor regeneration. Press ZERO and turn the ZERO ADJUST (refer to page 10 in the 431-X manual for the complete sensor regeneration procedure).
  - ➢ Wait 1 hour before proceeding to the next step.
  - ➢ Repeat.the mercury injection test procedure.
  - ➢ If the average of the last three readings is still not within range, refer to page 35 Functional Test Troubleshooting.

431-X Temperature Conversion Chart

Temperature in °C	Digital Meter Response
16	.091 to .123
17	.100 to .135
18	.108 to .146
19	.118 to .159
20	.129 to .174
21	.138 to .187
22	.151 to .204
23	.164 to .222
24	.177 to .240

12.7 Functional Test Troubleshooting

If you don't achieve good results with the functional test procedure, check the following:

Results	Solution
Too low	Be sure to inject the Hg vapor ONLY after the display flashes (approximately 2 seconds after SAMPLE is pressed).
Typically too high	Ensure the calibration vessel temperature is stable.
Too low	Ensure there is no oxidation on the mercury drop in the calibration vessel. Gently swirl the mercury drop in the calibration vessel. Replace if necessary.
Too low	Ensure the instrument's intake is not blocked with foreign matter. Check flow with a flow meter.
Too low	Ensure syringe is calibrated to 1cc. Use a new syringe needle. Straighten or replace crimped or blocked internal tubing.

If you find the above does not solve your problem, please call AZI Customer Service at 800-528-7411 or 602-470-1414.

13 APPENDIX B - PERSONAL MERCURY DOSIMETER

The gold coil personal mercury dosimeter is a unique collection device for mercury vapor. The Jerome 431-X Gold Film Mercury Vapor Analyzer and the Personal Mercury Dosimeter determine personal exposure levels and ambient air concentrations, as well as low levels of mercury in natural and stack gases

For personal sample collection, the dosimeter is worn as close to the wearer's breathing zone as possible and is connected by tubing to a pump usually worn on a belt. The dosimeter can also be used for multiple point area monitoring by placing a dosimeter, with pump attached, in various strategic locations.

We recommend a pump flow rate of 2 cc/minute for the most accurate results when sampling in an atmosphere that for eight hours may contain an average of .5 mg/m^3 Hg. If you are considering using any other flow rate, see page 40, Nonstandard Flow Rates.

After sample collection is completed, the dosimeter is inserted in the Jerome 431-X's intake. A dosimeter lead set is connected between the dosimeter and the DB-25 connector on the back of specially equipped instruments. The instrument supplies power to volatilize the accumulated mercury from the dosimeter to the gold film sensor. The Jerome 431-X determines the mass of mercury collected by the dosimeter in a 17 second analysis. The dosimeter is ready for immediate re-use after a mercury measurement has been performed.

13.1 Dosimeter Technical Specifications*

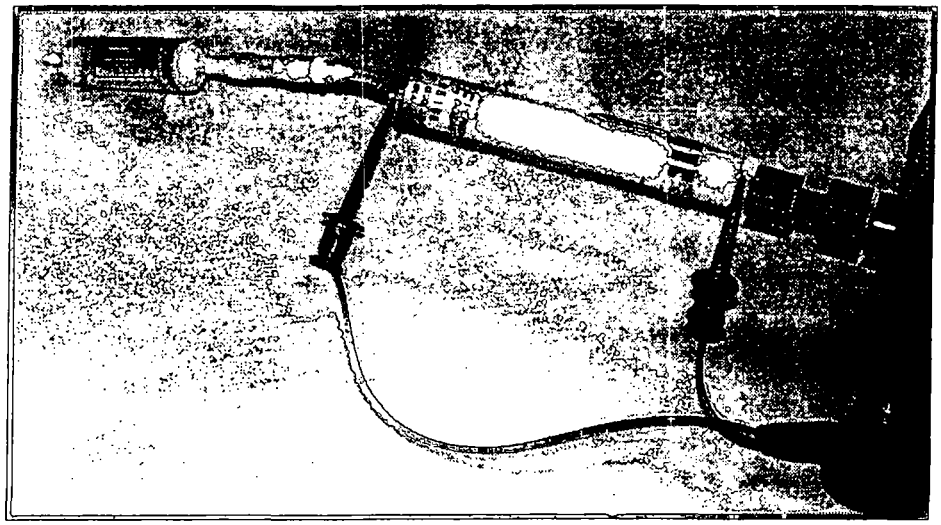
Sensitivity	< 0.5 x 10^{-9}g Hg	
Precision	15% RSD @ 0.100 mg/m^3 Hg	
Accuracy	15% @ 0.100 mg/m^3 Hg	
Recommended flow rate	2 cc/min (0.002 liters/min) for atmospheres of 0.5 mg/m^3	60 cc/min for 100% collecting efficiency in atmospheres with lower concentrations
Construction	Nylon/Glass	
Weight	1.5 ounces	
Dimensions	0.5" dia. x 4.5" l	
Capacity	1000 X 10^{-9}g Hg	
Analysis Time	< 2 min	

*Based on 2 cc/min flow rate

13.2 Before Sampling with the Dosimeter

The personal mercury dosimeter adsorbs mercury vapor over a period of time. Therefore, before each day's use it is necessary to ensure the dosimeter is mercury free. Perform the following steps to remove any accumulated mercury.

- Connect the system as shown.
  - ➢ Insert the dosimeter's large end in the 431-X's intake and gently tighten the intake tube nut to ensure an airtight seal.
  - ➢ Connect the Dosimeter Lead Set clips as shown, Short red lead to the rear and long black lead to the far end.



- Attach the power cord to the 431-X and plug it into AC power. AC power is required to heat the dosimeter.

- Attach the Dosimeter Lead Set 25 pin connector to the respective 25 pin communications port.

- Press the instrument's power ON button.

- Press the instrument's SAMPLE button.
  - ➢ The digital meter reading will appear in 15 seconds.

- Wait 60 seconds and press the SAMPLE button again.
  - ➢ The digital meter should display less than 0.005, verifying all mercury has been removed from the dosimeter coil.

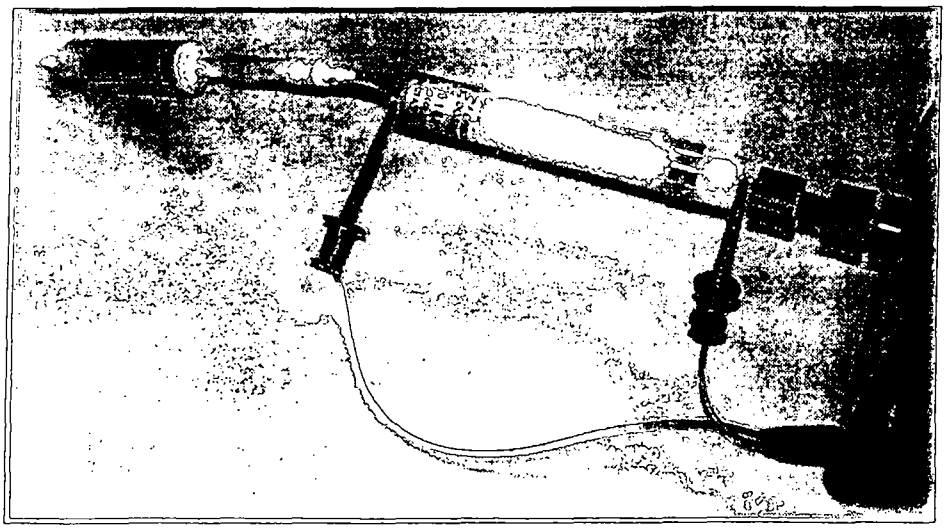
- The dosimeter is ready for sample collection.

NOTE:	For best results, dosimeter analysis should be performed immediately after collection. If analysis cannot take place immediately, place the red end caps on the dosimeter. For accurate results, perform dosimeter analysis no later than five days after sampling.

13.3 Dosimeter Analysis

NOTE: Wait a minimum of 30 minutes after a sensor regeneration before continuing.

- Connect the system as shown.
  - ➢ Insert the dosimeter's large end in the 431-X's intake and gently tighten the intake tube nut to ensure an airtight seal..
  - ➢ Connect the Dosimeter Lead Set clips as shown, Short red lead to the rear and long black lead to the far end.



- Attach the power cord to the 431-X and plug it into AC power. AC power is required to heat the dosimeter.

- Attach the Dosimeter Lead Set 25 pin connector to the respective 25 pin communications port.

- Press the instrument's power ON button

- Reading the dosimeter (dosimeter desorption)
  - ➢ Press the SAMPLE button.
    - ▷ The digital meter reading appears in 15 seconds.
  - ➢ Record the digital meter reading (include the decimal point).
  - ➢ Wait 30 seconds.
  - ➢ Press SAMPLE again and record this digital meter reading. Repeating the heating/reading process ensures complete release of mercury from the dosimeter coil.

- Add the two digital meter readings together. The sum of the two digital meter readings is the figure you will use in your calculations and is referred to as the meter response (MR).

NOTE: A third dosimeter desorption (pressing the SAMPLE button) should give a reading of .005 mg/m^3 or less.

- Perform the following calculation to obtain the mercury concentration in mg/m^3 based on a time weighted average; or alternately, DIP switch #2 can be set to OFF and the digital meter will display nanograms Hg directly (refer to diagram, page 18).

Working Formula and Units of Measure

$$\frac{\text{Meter Response X Conversion Factor}}{\text{Pump Flow Rate X Sampling Time}} = \text{Sample Concentration}$$

Where:

Meter Response	=	Total of the two digital meter readings in mg/m^3
Conversion Factor	=	87.5 $ng/mg/m^3$ (a constant which changes the meter response to nanograms of Hg)
Pump flow rate	=	2.0cc per minute (calibrated value of the supplied Sipin Pump)
Sampling time	=	Duration of the sample in minutes
Sample concentration	=	In ng/cc mg/m^3

EXAMPLE: To calculate a time weighted average during an 8 hour period:

Meter response	=	0.600 mg/m^3 (sum of the two meter response readings)
Conversion factor	=	87.5 $ng/mg/m^3$ (constant)
Pump flow rate	=	2 cc/min
Sampling time	=	8 hours (480 min)

➢ Convert the meter response (the total of the two digital meter readings) to nanograms of mercury.
  ➢ 0.600 x 87.5 = 52.5 nanograms of Hg

➢ Determine the total volume of air sampled.
  ➢ 2 cc/min x 60 min/hr x 8 hr = 960 cc

$$\frac{52.5\text{ ng}}{960\text{ cc}} = 0.055\text{ ng / cc of Hg} = 0.055\text{ mg / m}^3\text{ of Hg}$$

➢ Determine the Hg concentration (time weighted average) of the dosimeter.

- Check the sensor status after each dosimeter analysis.

IMPORTANT:
Perform a sensor regeneration as soon as the meter display shows "—" (four bars) which shows 75-100% sensor saturation to prevent the loss of a sample.

- Seal the dosimeter with caps after analysis to prevent mercury contamination during storage.

- If your readings exceed 75 nanograms or more, try the recommendations described next or call AZI Customer Service at 800-528-7411 or 602-470-1414 for assistance.

13.4 Non-Standard Flow Rates and Dilution Modules

You may use a pump with a flow rate up to 50 or 60 cc/min, but be aware that there are certain limitations. If your pump flow rate exceeds 2 cc/min and your average dosimeter analysis produces nanogram levels of 75 or more, it may be easy to collect more mercury beyond the linear range of the 431-X sensor. You thus risk over ranging your instrument and losing your collection data. Higher flow rates may also impair the capture efficiency of the dosimeter.

We recommend that you drop your flow rate or use a dilution module* (AZI P/N Z2600-3911). Lowering the flow rate to decrease the sample volume provides the greatest accuracy. Using a dilution module introduces an additional 15% inaccuracy to your analysis. As an alternative to the dilution module, sample for shorter time periods.

Dilution Module Specifications

Accuracy	+/- 15% of 10:1 ratio
Input concentration range:	
Low	0.7 mg/m^3 Hg
High	5.0 mg/m^3 Hg
Housing	Nylon
Dimensions	1" w x 2.7" l x 3" h
Weight	3.3 oz

The dilution module is factory set to a 10:1 ratio. The mass of mercury entering the dilution module is reduced by 90%, leaving a 10% (X10 dilution) concentration to be introduced into the Jerome 431-X. since this ratio can change slightly with use, it is important to occasionally determine the current dilution module ratio to ensure accurate results. For normal applications a X8 to X12 ratio is recommended. The 431-X Functional Test Kit contains all accessories necessary to determine the current dilution module ratio.

Call Customer Service at 800-528-7411 or 602-470-1414 if you have questions about flow rates or applications.

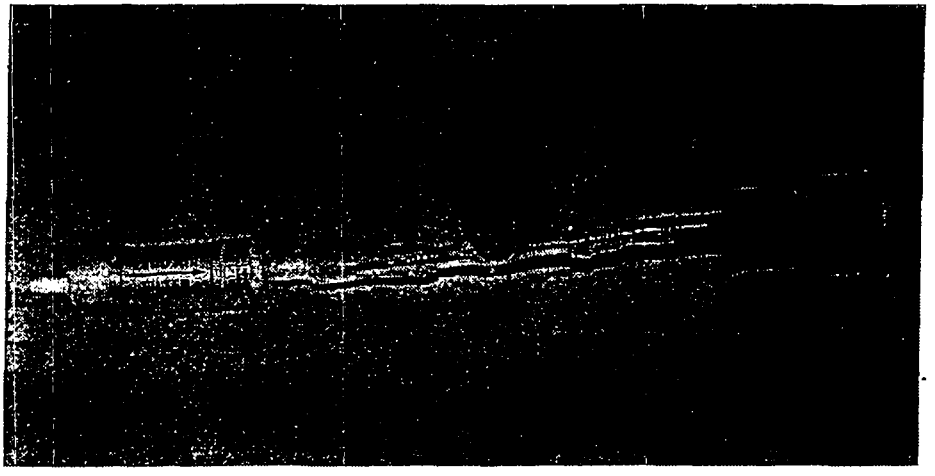
*The dilution module contains Resisorb™, mercury vapor adsorbent. For safety information, see the supplier's Material Safety Data Sheets (MSDS) or call AZI Customer Service at 1-800-528-7411 or 1-602-470-1414 for assistance in obtaining the MSDS.

13.5 Dilution Module Ratio Check

NOTE: Wait a minimum of 30 minutes after a sensor regeneration before starting this procedure.

Direct 431-X Readings:

- Connect the instrument, septum holder assembly and zero air filter, with arrow pointing to the instrument, as shown below.

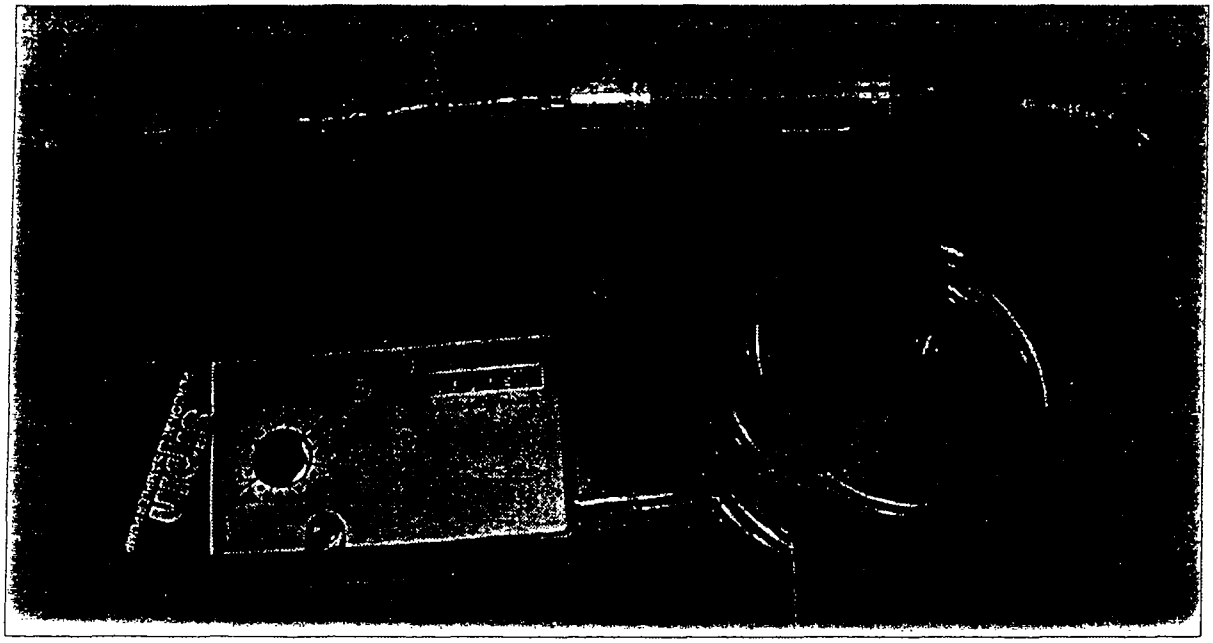


- Press the Jerome 431-X power ON button.
- Inject 1 cc of mercury saturated vapor into the septum, according to the Syringe Technique described on page 32 (431-X Functional Test, Appendix A).
- Make 3 additional 1 cc injections and record the digital meter readings (include the decimal points).
- Average the results of the last 3 injections.
- Remove the septum assembly and zero air filter from the instrument.

Most Accurate Method

Perform the above test, however attach the dosimeter, septum holder assembly and zero air filter to the sampling pump that will be used. (See diagram.) Collection efficiencies should be approximately 100% up to 60 cc/min. If high flow rates are used, the final calculation should include this collection efficiency correction factor.

- Connect your pump, dosimeter, septum holder assembly and zero air filter together with 1/8" an d 3/16" Tygon™ tubing as shown below.



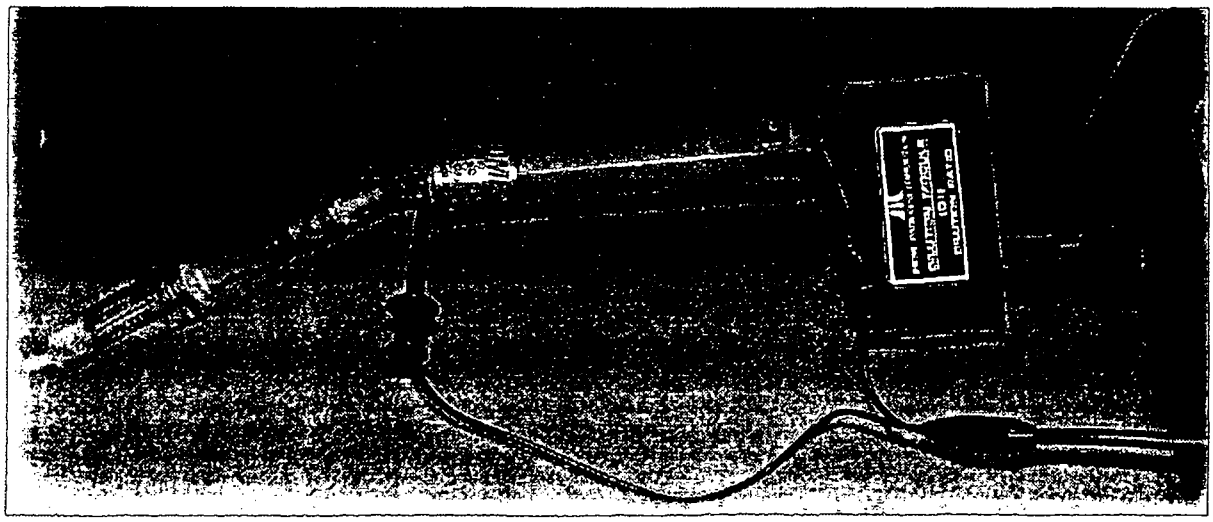
- Turn on the pump.

- Inject 10 cc's of Hg into the septum, 1 cc at a time.
  ➢ A total of ten, 1cc injections, one after another.

- Wait 30 seconds after the last injection then turn off the pump.

- Remove the dosimeter, septum assembly and zero air filter from the pump.

- Connect the instrument, dilution module, dosimeter, zero air filter and dosimeter lead set as shown below.



- Attach the power cord to the 431-X and plug it into AC power.
  ➢ AC power is required to heat the dosimeter.
- Press the Jerome 431-X Power ON button and then press the SAMPLE button.
  ➢ The digital meter reading appears in approximately 15 seconds.

- Record the digital meter reading (include decimal point). Wait 60 seconds, then press SAMPLE again and record this reading.

- Repeating the heating process ensures complete release of mercury from the dosimeter coil.

- Add the two digital meter readings together.
  ➢ The sum of the two digital meter readings is the figure you will use in your calculations and is referred to as the meter response (MR).

- Repeat this procedure two more times.

- Average the three meter responses you obtained in this section.

Dilution Module Ratio Calculations

- Multiply the average obtained for Direct 431-X Readings by 10 (the number of 1 cc injections).
  ➢ Divide this result by the average obtained in the section "Loading the Dosimeter" on page 42.

- Use the result as the dilution module ratio in your dosimeter analysis.

EXAMPLE:

Direct 431-X readings

0.102 mg/m^3
0.103 mg/m^3
0.104 mg/m^3
0.103 mg/m^3 average

Loading the dosimeter

0.120 mg/m^3
0.113 mg/m^3
0.100 mg/m^3
0.111 mg/m^3

Step 1 (above)
0.103 mg/m^3 x 10 = 1.030 mg/m^3

Step 2 (above)
$(1.030\ mg/m^3) / (0.111\ mg/m^3 = 9.4$

Dilution module ratio 9.4:1

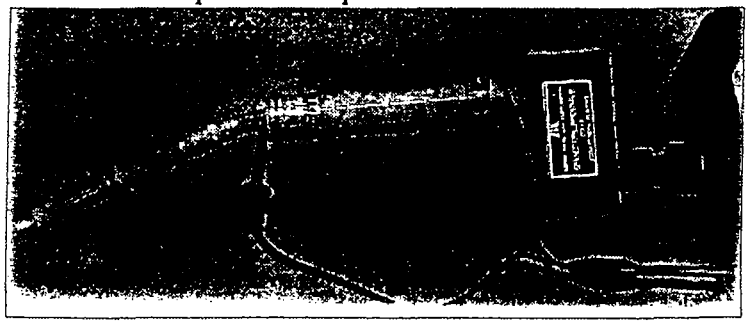
NOTE: For normal applications a X8 to X12 ratio is recommended. If your ratio is not within this range, call Customer Service at 800-528-7411 for assistance.

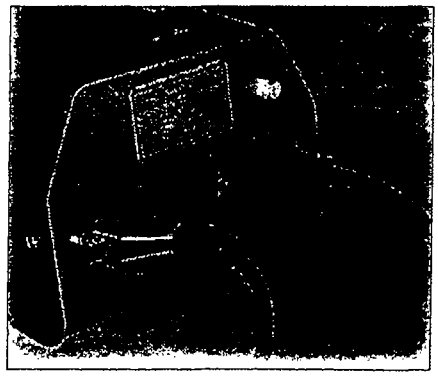
13.6 Analysis with a Dilution Module

NOTE: Wait a minimum of 30 minutes after a sensor regeneration before starting this procedure.

- Connect the system as shown in the figure below.

- Attach the power cord to the 431-X and plug it into AC power.
  ➢ AC power is required to heat the dosimeter.





- Press the Jerome 431-X power ON button and then press SAMPLE button. The digital meter reading appears in 12 seconds.

- Record the digital meter reading (include the decimal point). Wait 30 seconds, then press SAMPLE button again and record this reading.
  ➢ Repeating the heating process ensures complete release of mercury from the dosimeter coil.

- Add the two digital meter readings together.
  ➢ The sum of the two digital meter readings is the figure you will use in your calculations and is referred to as the meter response.

- The following calculations will provide the mercury concentration in mg/m^3 based on a time weighted average.

$$\text{(Meter Response converted to nanograms (Ng) of mercury)} \times \frac{\text{Dilution Module Ratio}}{\text{Sample Volume}} = \text{Sample Concentration}$$

- Alternately, DIP switch #2 can be set to OFF and the digital meter will display nanograms Hg directly.

MR (meter response)	*total of the two digital meter readings in mg/m³*
87.5 ng/mg/m³	conversion factor, a constant which changes the meter response to nanograms of Hg
DM dilution module ratio	the ratio determined on page 43
SV (sample volume)	pump flow rate (in cc/min) multiplied by sample time (in minutes)
Sample concentration	*ng/cc = mg/m³*

EXAMPLE:
Assume the following values.

Meter Response (MR)	0.600 mg/m³ (sum of the two meter response readings)
Conversion Factor	87.5 ng/mg/m³ (constant)
Dilution Module Rate	9.4
Pump Flow Rate	2 cc/min
Sampling time	8 hours = 480 min
Sample volume	2 cc/min X 480 min = 960cc

A time weighted average during an 8 hour period is calculated by:

$$\frac{(0.600mg/m3)\times(87.5ng/mg/m)}{960cc} = 0.055ng/cc$$

- Convert the meter response (the total of the two digital meter readings) to nanograms of mercury.
  - The MR (0.600) multiplied by the conversion factor (87.5) equals nanograms of mercury
  - 0.600 x 87.5 = 52.5 nanograms of Hg

- Determine the actual mass of Hg collected by the dosimeter.
  - Nanograms of mercury multiplied by the dilution module ratio.
  - 52.5 nanograms x 9.4 = 493.5 nanograms

- Determine the total volume of air sampled.
  - The pump flow rate multiplied by 60 min/hr multiplied by 8 hours.
  - 2 cc/min x 60 min/hr x 8 hr = 960 cc

- Determine the Hg concentration (time weighted average) of the dosimeter.
  - The mass of Hg collected by the dosimeter divided by the total volume of air *sampled.*
  - 493.5 nanograms divided by 960 cc = 0.0514 ng/cc of Hg = 0.0514 mg/m³ of Hg

- Check the sensor status after each dosimeter analysis.

- Seal the dosimeter with tubing after analysis to prevent excessive mercury contamination during storage.

> IMPORTANT:
> Perform a sensor regeneration as soon as the meter display shows "—" (four bars) to prevent the loss of sample.

Dosimeter Reference Chart 431-X

Expected concentration, related to sample volume and meter response

= Indicates the optimum meter response for that concentration with the corresponding volume

Volume of Air in mg/m³	431-X Meter Responce				
0.5	HL	HL	HL	HL	HL
0.1	0.275	0.549	HL	HL	HL
0.05	0.137	0.274	0.549	HL	HL
0.025	0.069	0.137	0.274	HL	HL
0.005	0.014	0.027	0.055	0.823	HL
0.001	0.003	0.005	0.011	0.165	0.329
	240	480	960	14,400	28,800

(.001 micrograms/m³ = 1 nanogram/m³)

= Indicates the optimum meter response for that concentration with the corresponding volume

Volume of air in micro-grams/m³	431-X meter response						
0.5	0.247	0.494	0.823	HL	HL	HL	HL
0.05	0.025	0.049	0.082	0.274	0.823	HL	HL
0.01	0.005	0.010	0.016	0.055	0.165	0.329	0.494
0.005	0.002	0.005	0.008	0.027	0.082	0.165	0.247
0.0005	0.000	0.000	0.001	0.003	0.008	0.016	0.025
	43,200	86,400	144,000	480,000	1,440,000	2,880,000	4,320,000

Relationship of flow rate and time to total sample volume

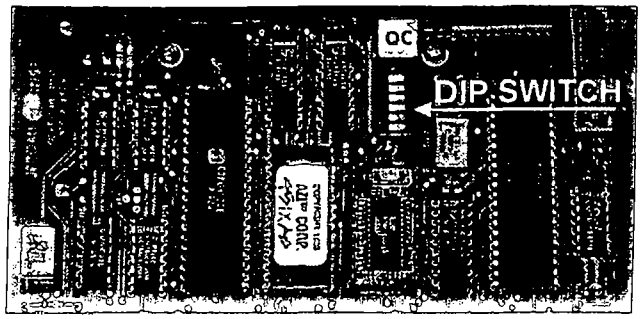
Flow Rate in cc/minute	Total volume collected in cc							
1000	60,000	120,000	240,000	480,000	720,000	1,440,000	2,880,000	4,320,000
100	6,000	12,000	24,000	48,000	72,000	144,000	288,000	432,000
60	3,600	7,200	14,400	28,800	43,200	86,400	172,800	259,200
20	1,200	2,400	4,800	9,600	14,400	28,800	57,600	86,400
10	600	1,200	2,400	4,800	7,200	14,400	28,800	43,200
2	120	240	480	960	1,440	2,880	5,760	8,640
	1	2	4	8	12	24	48	72
	Hours of Sample Collection							

Use this formula for calculating the concentration of mercury in air:

$$\frac{\text{Meter Responce X 87.5}}{\text{Flow Rate of Sampling Pump X Time}} = \text{Concentration}\,(mg/m^3)$$

14 APPENDIX C - INTERNAL DIP SWITCH SETTINGS

Main circuit board RED DIP switches (SW2)



This is the red DIP switch box located at the top, center of the instrument's main circuit board.

The 431-X provides regulated film heat at both 50 Hz and 60 Hz line frequencies. This also provides two ranges of preset but unregulated film heat (100-120/200-240 volt and 110-130/220-260 volt ranges). The two ranges are available to reduce the effects of chronic low or high line voltage.

Note: The ranges are doubled when the AC line selector switch is set to the 220V position. The DIP switch positions 1 and 6 must be properly set.

DIP 1	DIP 6	Function
OFF	OFF	60 Hz regulated film heat (100-130/200-260VAC)
OFF	ON	50 Hz regulated film heat (102-130/205-260 VAC)
ON	OFF	50/60 Hz preset film heat (110-130/220-260 VAC)
ON	ON	50/60 Hz preset film heat (100-120/200-240 VAC)

Regulated film heat should normally be used (DIP 1 OFF) except in the few cases where extremely dirty line voltage conditions may exist. These conditions might be found where large motors are being controlled or other situations may exist where the voltage may vary outside the 100-130 VAC range with regularity. In those cases the two preset heat ranges will allow some degree of satisfactory operation.

Switch Number	Normal Position	Action
2	ON	Nanograms mode
3	ON	Displays relative (not true)voltage during regen (0-255)
4	OFF	Display L-O-H when "zero" button pressed
	ON	Display 00-99 when "zero" button pressed
5	ON	Locks into 0-10mg/m^3 range (survey mode)

15 APPENDIX D - OPTION BOARD

15.1 Dip Switch Settings

This is the six position DIP switch located on the Option Board, the small board mounted near the center of the main board.

Summary of blue DIP switch functions:

Switch	Function
1	Regeneration enable and time
2	Regeneration enable and time
3	Auto sample enable
4	Auto sample time
5	Auto sample time
6	DC power mode enabled (when ON and regeneration is started, this closes the relay on the data logger interface board to switch the inverter ON.

Timed Sensor Regeneration (Timed regeneration attempted one hour after start, then at interval.)

Switch #1	Switch #2	Regeneration Interval
OFF	OFF	OFF
ON	OFF	6 Hours
OFF	ON	24 Hours
ON	ON	72 Hours

Auto Sampling (Without JCI or data logger attached)

Switch #3	Switch #4	Switch #5	Sampling Frequency
ON	ON	ON	No automatic sampling
OFF	ON	ON	5 minutes
OFF	OFF	ON	15 minutes
OFF	ON	OFF	30 minutes
OFF	OFF	OFF	1 hour

NOTE: Switch instrument power off before changing DIP switch settings.

15.2 Instrument Zeroing

- The Jerome 431-X has essentially three zeros:
  - ➢ The instrument automatically rezeroes between samples and each sample is a unique reading. To take a sample, simply press the SAMPLE button.
  - ➢ The zero on the membrane switch is used to re-establish a baseline between the reference and sensor gold film after a sensor regeneration. This zero is manually adjusted by pressing the ZERO button and turning the potentiometer on the top of the instrument until the display reads 0. Adjust only after sensor regeneration; it is normal for H to be displayed after sampling.
  - ➢ The 431-X option board provides an auto zero feature that is invisible to the user. In some cases, the instrument does not resume sampling after a regeneration. At that time .L.L.L appears on the display when the ZERO button is pressed and the error message "manual bridge adjust needed" will be added to the notes column of the JCI text file if the JCI software is used. If this problem persists, it may be necessary to re-set the auto zero.

- When necessary to re-adjust the auto zero point:

  - ➢ Turn the instrument off.
  - ➢ Note original DIP switch settings.
  - ➢ Turn DIP switch 4 on red DIP box to ON.
  - ➢ Set the switches on the option board's blue DIP box to 1,2,6 OFF; 3,4,5 ON.
  - ➢ Turn the instrument ON.
  - ➢ Switch option board DIP #1 OFF and ON three times, leaving it ON.
  - ➢ While pressing the ZERO button, turn the potentiometer on the option board until the numbers increase (maximum of 20). Note the display will flicker one digit.
  - ➢ Return all switches to original position.

NOTE: The higher the auto zero number, the lower the capacity of the sensor and the more sensor regenerations are needed.

15.3 Automatic Regeneration

- The auto-regeneration should take place at the pre-configured time, with these exceptions:

- The instrument will always attempt a regeneration one hour after the SAMPLE button is pressed. The auto-regeneration will take place at the specified interval after that initial hour (for example, at hour 7, 13, 19, etc, if programmed for 6 hour auto-regeneration).

- The instrument may NOT always perform the regeneration. The circuit forces the instrument towards 100% saturation to initiate the regeneration. If the sensor has not seen much mercury, the instrument will not auto-saturate. In this case, a regeneration will not take place. The instrument will always regenerate whenever the sensor is saturated. There should be no significant loss in sensitivity when a sensor auto-regeneration does not occur for 2-3 days.

- Test this feature by initiating an auto-regeneration by turning the instrument ON and switching the daughter board's blue DIP switch #2 OFF and ON. If the line cord is plugged in, do not interrupt this regeneration cycle.

15.4 DC Power Mode Enable

15.5 Instruments with the 431-X option board modification can be used with any +12 VDC source for continuous operation, if the AZI DC-AC Power Inverter Kit, part number Y031-0902, is installed.

15.6 The instrument requires 115 volts AC for regeneration. To preserve the life of the DC power source, the DC-AC inverter will switch on automatically for the regeneration only. The external switch on the inverter should always be OFF to preserve battery life during normal sampling.

15.7 When the instrument starts a regeneration and when DIP Switch #6 is ON, the instrument sends a signal to close the relay on the 431-X data logger interface board mounted between the data logger and the instrument. This switches the inverter ON using the inverter's internal switch.

NOTE: When this mode is enabled, the instrument does NOT check for 115 VAC for the regeneration. If there is no AC power to the instrument, and a regeneration is initiated, the instrument will flash .H.H.H (rather than .P.P.P), however the sensor will not heat, nor will the sensor clean.

16 WARRANTY

Seller warrants to buyer that products delivered pursuant to this Agreement shall, at the time of delivery, and for a period of one (1) year thereafter (the Internal Battery Pack, where applicable, is warranteed for a period of ninety [90] days only), be free from defects in material or workmanship and shall conform to seller's specifications or such other specifications as seller has agreed to in writing. Seller's obligations with respect to claims under this warranty shall be limited, at seller's option, either to the replacement of defective or non-conforming product or to an appropriate credit for the purchase price thereof subject to the provisions of seller's Warranty Policy as amended from time to time, said Policy being incorporated herein by reference.

Return products under warranty claims will be shipped to seller's plant by buyer at buyer's expense and shall be accompanied by a statement of the reason for the return and an approved Return Material Authorization Number issued by seller. Buyer remains responsible for payment for products not accepted for warranty adjustment and freight and handling costs associated therewith.

Notwithstanding the foregoing, no warranty shall be enforceable in the event that product has been subjected to environmental or stress testing by buyer or any third party without written approval of seller prior to such testing. Further, no warranty shall be enforceable if the alleged defect is found to have occurred as a result of misuse, neglect, improper installation, repair, alteration, accident, or improper return handling procedure by buyer.

Discontinued product is warrantied only for a credit or replacement at seller's option.

THE EXPRESS WARRANTIES GRANTED ABOVE SHALL EXTEND DIRECTLY TO BUYER AND NOT TO BUYER'S CUSTOMERS, AGENTS, OR REPRESENTATIVES AND, EXCEPT FOR WARRANTY OF TITLE, IS IN LIEU OF ALL OTHER WARRANTIES, WHETHER EXPRESSED OR IMPLIED, INCLUDING ANY IMPLIED WARRANTIES OF FITNESS FOR A PARTICULAR PURPOSE AND MERCHANTABILITY, SUCH OTHER WARRANTIES BEING SPECIFICALLY DISCLAIMED BY SELLER. IN NO EVENT SHALL EITHER PARTY'S LIABILITY FOR ANY BREACH OR ALLEGED BREACH OF THIS AGREEMENT EXCEED THE TOTAL EXTENDED PRICE OR PRICES SHOWN ON UNFILLED ORDERS, NOR SHALL EITHER PARTY BE LIABLE FOR ANY SPECIAL, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM BREACH OR ALLEGED BREACH.

Notwithstanding the foregoing, if any product covered by order(s) placed hereunder is designated as "developmental" "prototype" or "experimental," no warranty whatsoever except a warranty of title to component materials, will be applicable thereto and buyer shall indemnify seller for any claims for liability asserted seller in connection therewith.

Medical Applications: Seller's products are not designed for use in medical appliances, devices or systems where malfunction of buyer's product can result in personal injury. Buyer's customers using or selling buyer's products for use in medical applications do so at their own risk and agree to fully indemnify buyer.

The foregoing state the entire liability of seller in connection with products supplied hereunder.

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Arizona Instrument LLC
Jerome 431-X Mercury Vapor Analyzer Operation Manual
Part Number SS-086
Revision D
September, 2000

If you have any questions regarding the operation of this instrument, please call our toll free number from the United States and Canada 800-528-7411. Internationally, call (602) 470-1414 or fax (480) 804-0656.

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