



EPA Region X
START

Superfund Technical Assessment and Response Team

*Upper Columbia River/Lake Roosevelt
Expanded Site Inspection
Sampling and Quality Assurance Plan
TDD: 01-02-0001-A*

*EPA Contract: 68-S0-01-02
May 2001*

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Roy F. Weston, Inc. • 190 Queen Anne Avenue North • Seattle, WA 98109-4926

SAMPLING AND QUALITY ASSURANCE PLAN

Upper Columbia River/Lake Roosevelt Expanded Site Inspection
Northeast Washington

TDD: 01-02-0001-A

Prepared By:

ROY F. WESTON, INC.
190 Queen Anne Avenue North, Suite 200
Seattle, WA 98109-4926

Contract No: 68-S0-01-02

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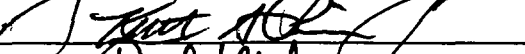

APPROVALS			
TITLE	NAME	SIGNATURE	DATE
EPA Task Monitor	Mark Ader		5/10/01
EPA QA Officer	Ginna Grepo-Grove		5/10/01
START Project Manager	Keith Pine		5/9/01
START QA Officer	Paul Swift		05/09/01

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SQAP DISTRIBUTION LIST

Name	Organization	Title	Contact Information
Mark Ader	U S Environmental Protection Agency Seattle, WA	Site Assessment Manager	Phone (206) 553-1808 e-mail ader mark@epamail epa gov
Monica Tonel	U S Environmental Protection Agency Seattle, WA	Site Assessment Manager	Phone (206) 553-0323 e-mail tonel monica@epamail epa gov
Ginna Grepo-Grove	U S Environmental Protection Agency Seattle, WA	Chemist, Quality Assurance and Data Unit, OEA	Phone (206) 553-1632 e-mail grepo-grove gina@epamail epa gov
Matthew Gubitosa	U S Environmental Protection Agency Seattle, WA	Geographic Information Systems Analyst	Phone (206) 553-4059 e-mail gubitosa matt@epamail epa gov
Susan FitzGerald	Roy F Weston, Inc Seattle, WA	START Site Leader	Phone (206) 521-7654 e-mail fitzgers@mail rfweston com
Keith Pine	Roy F Weston, Inc Seattle, WA	START Project Manager	Phone (206) 521-7681 e-mail pinek@mail rfweston com
Leatta Dahlhoff	Ecology and Environment, Inc Seattle, Washington	START Project Manager	Phone (206) 624-9537 e-mail ldahlhoff@ene com
Patti Stone	Colville Confederated Tribes	Water Quality Coordinator, Office of Environmental Trust	Phone (509) 634-2415 e-mail stonewq@televar com
Dr F E. Kirschner	AESE, Inc (under contract to the Spokane Tribe)	Senior Scientist and CEO	Phone (509) 924-0184 e-mail fredk@icehouse net

LIST OF ACRONYMS

<u>Acronym</u>	<u>Definition</u>
ASL	Above Sea Level
ASTM	American Society for Testing and Materials
B	Detected concentration below the contract required detection limit but above the instrument detection limit
bgs	below ground surface
CCT	Colville Confederated Tribes
cfs	cubic feet per second
CLP	Contract Laboratory Program
CLPAS	Contract Laboratory Program Analytical Service
Critical	Required to achieve project objectives or limits on decision errors
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
°C	degrees Celsius
DGPS	Differential Global Positioning System
Dup.	Duplicate
EDD	electronic data deliverables
EPA	United States Environmental Protection Agency
E&E	Ecology and Environment, Inc.
ESAT	Environmental Services Assistance Team
ESI	Expanded Site Inspection
FASP	Field Analytical Services Program
GIS	Geographic Information System

LIST OF ACRONYMS (Continued)

<u>Acronym</u>	<u>Definition</u>
GPS	Global Positioning System
H	high bias
HRS	Hazard Ranking System
HAZWOPER	Hazardous Waste Operations and Emergency Response
K	unknown bias
kg/day	kilograms per day
L	low bias / Liter
MCL	Maximum Contaminant Level
MEL	Manchester Environmental Laboratory
mg/kg	milligrams per kilogram
MHW	Mean High Water
MS/MSD	matrix spike/matrix spike duplicate
MTCA	Model Toxics Control Act
NA	Not Applicable
Noncritical	For informational purposes only or needed to provide background information
NPS	National Park Service
PA	Preliminary Assessment
PAHs	polynuclear aromatic hydrocarbons
PCDD	polychlorinated dibenzo- <i>p</i> -dioxins
PCDF	polychlorinated dibenzofurans
PCBs	polychlorinated biphenyls
PCP	pentachlorophenol

LIST OF ACRONYMS (Continued)

<u>Acronym</u>	<u>Definition</u>
PM	Project Manager
PPE	probable point of entry
Q	Detected concentration below the contract required quantitation limit but above the method quantitation limit
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QMP	Quality Management Plan
RM	River Mile(s)
RSCC	Regional Sample Control Coordinator
SAP	Sampling and Analysis Plan
SD	Sediment
SDWA	Safe Drinking Water Act
SOPs	standard operating procedures
SP	Seep
SQAP	Sampling and Quality Assurance Plan
SOP	standard operating procedures
SS	Soil
START	Superfund Technical Assessment and Response Team
SVOCs	semivolatile organic compounds
TAL	Target Analyte List
TBD	to be determined

LIST OF ACRONYMS (Continued)

<u>Acronym</u>	<u>Definition</u>
TCDD	tetrachlorodibenzo-p-dioxin
TCDF	tetrachlorodibenzofuran
TDD	Technical Direction Document
TM	Task Monitor
TOC	Total Organic Carbon
USGS	United States Geologic Survey
VOCs	volatile organic compounds
WESTON	Roy F. Weston, Inc.
WRATS	Water Rights Application Tracking System
WDOH	Washington Department of Health
XRF	X-Ray Fluorescence analyzer

SECTION 1

PROJECT MANAGEMENT

1.1 PROJECT/TASK ORGANIZATION

This section outlines the individuals directly involved with the Upper Columbia River/Lake Roosevelt project and their specific responsibilities. Lines of communication are shown in the Project Organization Chart (Figure 1-1).

1.1.1 Purpose

Pursuant to EPA START Contract No. 68-S0-01-02 and Technical Direction Document (TDD) No. 01-02-0001-A, WESTON will perform an Expanded Site Inspection (ESI) at the Upper Columbia River/Lake Roosevelt site, located in northeast Washington. The Upper Columbia River/Lake Roosevelt ESI is a multi-organization sampling effort conducted by United States Environmental Protection Agency (EPA) Region X Office, in cooperation with the U.S. Parks Service. This ESI will be conducted through a cooperative effort involving both START contractors, Roy F. Weston, Inc. (WESTON®) and Ecology and Environment, Inc. (E&E). WESTON will conduct sampling activities along the Upper Columbia River and Lake Roosevelt, at selected locations on the Pend Oreille River downstream of Metaline, Washington. WESTON will also collect samples from points in the watersheds and near the mouths of selected Upper Columbia and Pend Oreille River tributaries. E&E personnel will investigate potential releases from numerous mines in the tributary watersheds in the study area. This Sampling and Quality Assurance Plan (SQAP) describes the proposed sampling and quality assurance procedures associated with the investigations that will be conducted by WESTON.

This document is a combined Sampling and Analysis Plan (SAP) and site-specific Quality Assurance Project Plan (QAPP) for field sampling activities. The combined SAP/QAPP, hereafter called the SQAP, includes a brief site 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 to be combined with information presented in WESTON's Quality Management Plan (QMP) for EPA Region 10 START. Copies of the QMP and the site-specific Health and Safety Plan (prepared for the ESI) are available in WESTON's office located at 190 Queen Anne Avenue North, Suite 200, Seattle, Washington 98109-4926. Standards contained in the SQAP and QMP will be used to ensure the validity of data generated by WESTON for this project.

1.1.2 Roles and Responsibilities

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

1.1.2.1 EPA Region 10 Task Monitor (TM)

EPA TM Mark Ader is responsible for providing technical direction and EPA oversight to WESTON's sampling activities along the Upper Columbia River and Lake Roosevelt, at selected locations on the Pend Oreille River downstream of Metaline, Washington, and at points in the watersheds and near the mouths of selected Upper Columbia and Pend Oreille River tributaries, which is tentatively scheduled for May and June, 2001. Mr. Ader is also responsible for project coordination and decision-making, reviewing and approving the site-specific SQAP and subsequent revisions in terms of project scope, objectives, and schedules, ensuring site-specific SQAP implementation, and serving as the primary point of contact for general project problems.

1.1.2.2 EPA Region 10 TM

EPA TM Monica Tonel is responsible for providing technical direction and EPA oversight to E&E's investigation of potential releases from numerous mines in the tributary watersheds in the Upper Columbia River/Lake Roosevelt area scheduled for June, 2001.

1.1.2.3 EPA Region 10 Quality Assurance (QA) Officer

The EPA Region 10 QA officer is Ms. Ginna Grepo-Grove. Ms. Grepo-Grove is responsible for reviewing and approving the site-specific SQAP and revisions in terms of quality assurance aspects, and may also conduct assessments of field activities.

1.1.2.4 EPA Region 10 Regional Sample Control Coordinator (RSCC)

The EPA Region 10 Sample Control Coordinator is Laura Castrilli. Ms. Castrilli is responsible for coordinating sample analyses performed through the EPA Contract Laboratory Program (CLP) and/or the EPA Region 10 Manchester Environmental Laboratory (MEL) and will provide sample identification numbers.

1.1.2.5 WESTON START Project Manager (PM)

The WESTON START PM is Mr. Keith Pine. Mr. Pine has the responsibility for the overall performance of the START team. He will review and approve the site-specific SQAP. He makes the ultimate decisions for the implementation of START projects and ensures that the implementation of the project is performed in accordance with the specifications of the SQAP and WESTON's QMP and SOPs. Mr. Pine has overall responsibility for maintaining project budget and schedule. In the absence of the START PM, the START Site Leader will assume the PM's responsibilities.

1.1.2.6 WESTON START Site Leader

The WESTON START Site Leader is Ms. Susan FitzGerald. The WESTON START Site Leader reports directly to the WESTON START PM. Ms. FitzGerald is the primary contact point with the EPA TM, will provide overall coordination of field work, and oversees the preparation of the

site-specific SQAP. She will ensure that the final approved version of the site-specific SQAP is implemented correctly and will record any deviations from it. Ms. FitzGerald will receive the CLP/Region 10 laboratory information from the RSCC and is the primary START point of contact for technical problems. She will be responsible for the execution of decisions and courses of action deemed appropriate by the EPA TM.

1.1.2.7 WESTON START QA Officer

The WESTON START QA Officer is Dr. Paul Swift. Dr. Swift is responsible for reviewing and approving the site-specific SQAP and conducting in-house audits of field operations. Dr. Swift is also responsible for auditing and reviewing the field activities and final deliverables, and proposing corrective action, if necessary, for nonconformities. All WESTON field audits will be preapproved by the Task Monitor.

1.1.2.8 WESTON START Program Manager and EPA Project Officer

The WESTON START Program Manager, Mr. Frank Monahan, and the EPA Project Officer, Ms. Sharon Nickels, are responsible for coordinating resources requested by the TM for this project and for the overall execution of the START program.

1.1.2.9 EPA Investigation and Engineering Unit

The EPA Investigation and Engineering Unit will provide sampling vessels and vessel operators for sample collection activities. Designated vessel operators include Mr. David Terpening and Mr. Doc Thompson.

1.1.2.10 United States Forest Service (USFS)

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

1.1.2.11 ESAT FASP Team

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

1.1.2.12 Manchester Environmental Laboratory (MEL) or Designated CLP Laboratory

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

1.1.2.13 Commercial Analytical Laboratory

A designated laboratory will perform analyses not supported by MEL or CLP.

1.2 PROBLEM DEFINITION/BACKGROUND

Information pertaining to site background (Section 1.2.1), site operations and source characteristics (Section 1.2.2), and site characterization (Section 1.2.3) is presented in the subsections below.

1.2.1 Site Background

Information presented in this subsection is based on a review of site background information and interviews with property owners and representatives from various regulatory agencies.

1.2.1.1 Site Location

Information pertaining to site location is presented below.

Site Name:	Upper Columbia River/Lake Roosevelt
CERCLIS ID No.:	WASFN1002171
Location:	Northeast Washington
Counties:	Ferry, Stevens, Pend Oreille
Site Contact(s):	Mr. Dave Lyngholm Area Manager United States Department of the Interior (USDoI) Bureau of Reclamation P.O. Box 620 Grand Coulee, Washington 99133-0620 (509) 633-9501 Ms. Patti Stone Water Quality Coordinator Office of Environmental Trust Confederated Tribes of the Colville Reservation P.O. Box 150 Nespelem, Washington 99155 (509) 634-2415 Mr. Randy Connolly Superfund Coordinator Spokane Tribe – DNR P.O. Box 480 Wellpinit, Washington 99040

1.2.1.2 Site Description

The Columbia River originates in southeastern British Columbia and enters the United States at River Mile (RM) 745, northeast of Northport, Washington. The river flows generally south then west through Washington, and along the boundary between Washington and Oregon states before emptying into the Pacific Ocean. The Pend Oreille River is one of the major tributaries to the Columbia River. It joins the Columbia at Waneta, British Columbia, roughly one-half mile north of the U.S.-Canada border. The primary area of interest for this investigation includes roughly 80 river miles along the Columbia River, from Inchelium, Washington to Trail, British Columbia, and from the confluence of the Pend Oreille River in British Columbia, roughly 30 miles upstream to Metaline, Washington (Figure 1-2).

Four dams control flow conditions along the rivers within the study area. The largest of the four is Grand Coulee Dam, located on the Columbia River near RM 597. The impounded waters of the Columbia River upstream of the Grand Coulee Dam form Franklin D. Roosevelt Lake, more commonly known as Lake Roosevelt. The Columbia River provides approximately 90 percent of the inflow to Lake Roosevelt; the remainder is contributed by four other sizable rivers (the Kettle, Colville, Spokane, and Sanpoil Rivers) and the numerous tributaries that flow directly into the lake. In general, lake conditions extend from Grand Coulee Dam northward to the vicinity of Onion Creek; north of the Onion Creek area, riverine conditions dominate along the Columbia River.

The full pool elevation of Lake Roosevelt is 1,290 feet above sea level (ASL), which is usually achieved in July and held steady through December. Each winter, the water level is drawn down to accommodate spring runoff from the tributary watersheds. Maximum drawdown typically occurs near the end of April, at which time the lake level is below 1,237 feet ASL (E&E 2000). Minimum pool elevation is 1,208 feet ASL. Depths along the river generally decrease as one travels upstream. Maximum depths near Inchelium are on the order of 200 feet below MHW, while at Onion Creek, maximum depths are approximately 70 feet below MHW. North of Onion Creek to the international border, maximum depths decrease to between 30 to 40 feet below MHW. The MHW datum also changes along the river; north of Deep Creek (roughly 1.5 miles north of Northport), the datum gradually changes from 1290 to 1292.6 at the international border (Northwest Map Service, 1995).

In addition to the Grand Coulee Dam on the Columbia River, three dams exist within the study area on the Pend Oreille River. Boundary Dam, operated by Seattle City Light, is located approximately one mile south of the U.S./Canada border. Seven-Mile Dam, operated by BC Hydro, is located seven miles downstream from the border in Canada. Waneta Dam, owned by Cominco Ltd., is located roughly five miles further downstream, about 0.5 mile above the confluence of the Pend Oreille with the Columbia River.

1.2.2 Site Operations and Source Characteristics

Types of operations and sources that have potentially contributed to the impacts associated with the Upper Columbia River/Lake Roosevelt area have included mining and milling, smelting, wood products production (i.e., pulp mills), and the operation of municipal sewage treatment plants. These operations are discussed in the subsections below.

1.2.2.1 Mining and Milling

Mining and milling operations for the production of metal ores and other resources (dolomite, silica, and clay) developed at numerous sites along both U.S. and Canadian tributaries to the Upper Columbia River in the late 1800s (E&E 2000; USGS 1994a). Metals mining in the Columbia River watershed produced primarily zinc and lead, and to a somewhat lesser extent copper, gold, silver, tin, tungsten, and molybdenum (USGS 1994a).

Typically, ores were concentrated at mills located near each mine before being transported to smelters for further refining. Ore processing methods resulted in the production of large quantities of wastes containing arsenic, cadmium, cyanide, lead, mercury, copper, and zinc (E&E 2000). The Northport area was mined primarily for lead and zinc. Low-grade zinc and lead ore concentration methods involved crushing and pulverizing the ore, classifying the material using a stream of water. The classified ore material was then concentrated through a floatation process that involved adding reagents to the fine-grained ore material, then skimming off the resulting concentrated metals. Reagents used in floatation processes included alcohols, soda ash, lime, pine oil, cresylic acid, eucalyptus oils, coal tar (wood creosotes), xanthates, thiocarbonilide, copper sulfate, sodium cyanide, and sodium silicate. The cleaned, concentrated metals were then dried on vacuum filters and shipped to smelters for refinement (E&E 2000).

The Republic mining district on the Sanpoil River was producing gold and silver by 1896. Gold recovery was most commonly carried out using a cyanide leaching process. Ore pulp from the classifier was either passed over a sheet of mercury, which removed the gold by amalgamation, or the pulp was concentrated using floatation. In each case, tailings from the primary process underwent cyanidation to further enhance recovery of the gold. Cyanide wastes resulting from this process were handled through any combination of dilution, acid treatment, ponding, and alkaline-chlorination (E&E 2000).

Areas of northern Idaho were also undergoing extensive mining operations at this same time, which affected sediment and water quality in the Coeur d'Alene watershed, including Lake Coeur d'Alene and the Spokane River. The Washington Department of Health is investigating the Dawn Mining Company Mill Ponds site in Ford, Washington, and the EPA is investigating the Midnight Mine site in Wellpinit, Washington, to determine the extent to which uranium mining and milling operations associated with these facilities have affected tributaries to the Spokane River Arm. The Spokane River discharges to Lake Roosevelt at approximately RM 639 (E&E 2000).

Numerous mine operations were also developed within the Pend Oreille River watershed. The largest mines on the Pend Oreille within the ESI study area are the Grandview Mine and Pend Oreille Mine, located near Metaline, WA.

Mining activities in the area today are relatively low, especially for metals ore production (USGS 1994a).

1.2.2.2 Smelters

Following initial concentration processes, ores were transported to Smelter facilities for further refinement. Two primary smelters in the Upper Columbia watershed include the former LeRoi smelter at Northport, Washington (15 RM south of the U.S./Canada border), and the Cominco smelter at Trail, British Columbia (roughly 11 RM north of the border) (E&E 2000).

The LeRoi smelter began refining gold and copper ores in 1896; by 1908 it was processing 500 tons of ore per day; the facility closed in 1909, but reopened in 1914 to process lead ore. The smelter finally closed in 1921. Various companies have since used the property for lumber operations. Large quantities of iron and slag waste were generated during the years of gold and copper smelting at the facility (E&E 2000).

Limited soil sampling was conducted during an EPA site inspection (ESI) in 1993. Nine of the 16 metals analyzed were determined to be present in one or both tailings piles at the site at significantly elevated concentrations.

The Washington State Department of Ecology (Ecology) performed sampling of the soil and slag piles at the former LeRoi smelter in 1997. Preliminary results indicated metals concentrations present at two to three orders of magnitude above background levels. Plans to conduct a groundwater investigation at the site were put on hold due to a lack of available resources (E&E 2000).

The Cominco smelter has been in operation since the turn of the twentieth century, and is still the world's largest integrated lead-zinc smelter and refiner (E&E 2000). The Cominco facility includes a fertilizer plant located adjacent to the smelter that utilizes residual sulfur generated at the metallurgical plant.

Effluent from the Cominco facility has historically been discharged to the Columbia River through five outfalls: one from the fertilizer production operation, one for slag discharge, and three submerged outfalls from the metallurgical plant. The slag appears primarily as a black sand-sized material, but also contains approximately 0.5 percent fines (USGS 1994a). The volume and contaminant levels of Cominco's effluent have been regulated by a provincial government permit. The effluent permit was revised in 1996 to meet goals for water quality and effluent toxicity, based on studies conducted by the Canadian government and Cominco itself. Metals concentrations in effluent from the metallurgical plant from 1980 to 1996 included concentrations as high as 18 kilograms per day (kg/d) of arsenic, 62 kg/d of cadmium, 200 kg/d of lead, 4 kg/d of mercury, and 7,400 kg/d of zinc (Cominco 1997). In 1994 Cominco discharged

an average of approximately 800,000 pounds (300 metric tons) of slag per day to the Columbia River, which was composed of approximately 2.5 percent zinc, 0.5 percent copper, and less than 0.1 percent lead (E&E 2000).

Cominco began a modernization program in 1979 that continued through the 1980s and 1990s. Modernization projects included efforts to control spills and dust, building a new lead smelter, installing air emissions controls, eliminating discharge of slag, replacing the phosphate fertilizer production with ammonium sulphate operation, and reducing effluent discharges. The flumes that were used to discharge slag to the river were removed in 1995; instead, the slag is now being shipped to cement plants to be incorporated into the manufacture of Portland cement (E&E 2000).

Stoney Creek Landfill is an inactive landfill and arsenic storage site associated with Cominco. It was identified as a significant contributor of contaminants to the Columbia River. Surface water in Stoney Creek currently exceeds provincial water quality objectives (Stone 2001). Remedial activities at this site are ongoing; no information on the status of the site was identified in the file documents reviewed during the PA (E&E 2000).

Cominco also historically discharged sulfides as air emissions through its 409-foot brick stack (up to 10,000 tons per month in 1925). The pollutants from the stack migrated south and were entrapped in the river valley in north Stevens County. Citizens of Northport complained of health effects stemming from the emissions. The case was brought before an international court, and in 1931 the International Joint Commission recommended that the Canadian government stop polluting the atmosphere (E&E 2000). Though the operation of the new smelter has reduced air emissions from the smelter, local citizens and representatives of the Colville Confederated Tribes (CCT) are concerned that present emissions may contain smaller particulates that may pose increased health threats (E&E 2000). Additionally, the Trail area has been designated a Contaminated Site by Environment Canada. Cominco is performing an Ecological Risk Assessment in relation to Trail Operations; the results of this assessment will be integrated with the findings of a human health risk assessment carried out by the Trail Lead Task Force (E&E 2000).

Several other significant smelters historically operated in the Upper Columbia watershed including several in British Columbia: two in the Greenwood area and one at Grand Forks.

1.2.2.3 Pulp Mill

The Celgar Pulp Company (Celgar) is located about 30 RM upstream of the international border, 2 RM downstream of the Hugh Keenleyside Dam near Castlegar, British Columbia. Celgar began operations in 1961. Until 1993, the mill's bleaching processes were primarily chlorine-based. During the bleaching process, chlorine reacts with compounds in the wood (including pentachlorophenol [PCP] if it had been used as a wood preservative), which results in the formation of compounds such as polychlorinated dibenzo-*p*-dioxins, polychlorinated

dibenzofurans, and other organics (USGS 1994a; E&E 2000). Celgar discharges effluent containing organic compounds to the Columbia River (USGS 1994a).

Wood debris and pulp fibers in effluent discharged into aquatic environments may form fiber mats that settle to the bottom and accumulate. These mats may contain chemicals resulting from pulp production and bleaching processes that persist in the environment long after the mats themselves have decomposed. Persistent chemicals associated with fiber mats have included heavy metals, tetrachlorodibenzo-p-dioxins (TCDDs), and polynuclear aromatic hydrocarbons (PAHs) (E&E 2000).

Celgar initialized expansion and modernization projects in 1991. The mill began using chlorine dioxide instead of chlorine in its bleaching process, and began secondary treatment of the plant effluent. Substantial reductions in TCDD and tetrachlorodibenzofuran (TCDF) concentrations in the effluent were reported by 1993 (E&E 2000). In addition, modifications to the mill process resulted in reduction of the amount of dioxin and furan precursor compounds (USGS 1994a).

The fiber mat located adjacent to the mill outfall was noted to have decreased in size as a result of the changes in the mill processes, and the remaining mat is now composed of a mix of wood debris (from ongoing logbooming) and flyash silt (E&E 2000). The PA report notes that the decrease in the size of the fiber mat at the mill may be due to downstream flushing in addition to natural degradation. Spongy materials observed at Northport Beaches have raised the concern of the CCT, who believe further investigations are needed to determine if they originate from the Celgar fiber mat (E&E 2000).

1.2.2.4 Municipal Sewage Treatment Plants and Other Potential Sources

Several sources discharge treated wastewater to the Columbia River or its tributaries. The Columbia River receives wastes from municipal treatment plants at Castlegar and Trail, British Columbia; similarly, wastes from Spokane are discharged to the Spokane River, and wastes from Colville and Chewelah are discharged to the Colville River. A large release of raw sewage to the Columbia River occurred in 1991 when the sewage collection system in Trail, BC became overloaded; the CCT has expressed concern that other undocumented releases have occurred during other overload events (E&E 2000).

Other potential sources of contaminants to the Upper Columbia River watershed include heavy metals from the Northwest Alloys Magnesium Plant on the Colville River, cyanide and fluoride groundwater contamination from the Kaiser aluminum plants in Spokane, runoff from process piles in Chewelah, and effluent discharge containing manganese and other wastes from the Avista electric generation plant at Kettle Falls (E&E 2000).

1.2.3 Site Characterization

This section summarizes previous site investigations (Section 1.2.3.1), discusses migration/exposure pathways and targets (Section 1.2.3.2), and describes areas of potential contamination (Section 1.2.3.3).

1.2.3.1 Previous Site Investigations

Many investigations have been conducted by both environmental agencies and interest groups to address a wide range of concerns regarding the conditions of the Upper Columbia River Basin and Lake Roosevelt.

In 1950, the Washington Pollution Control Commission, in cooperation with the United States Public Health Service and the British Columbia Department of Health and Welfare, sponsored a preliminary survey of the sources of mill and mining waste pollution in the Upper Columbia River Basin and Lake Roosevelt. The operations and waste handling methods of 11 mining operations, including mines and flotation and cyanidation mills, were examined and waste disposal alternatives to discharging to waterways were presented for some of the mine sites (E&E 2000).

In 1984, Ecology collected fish tissue samples from the Columbia River near Northport, and from nine other Washington rivers, and analyzed for metals concentrations. The highest concentrations of lead and cadmium were found in samples from Northport. The mean lead concentration in fillet samples from Northport was 6.4 micrograms per gram ($\mu\text{g/g}$), approximately 90 percent of the United States Food and Drug Administration's unofficial guideline of 7.0 $\mu\text{g/g}$ (E&E 2000).

In 1992, the USGS and the LRWQC, in cooperation with CCT, conducted a sediment quality assessment of Lake Roosevelt and the upper reaches of the Columbia River to the U.S./Canada border. The investigation included analyses of trace elements and organic compounds in sediments, bed sediment toxicity, and benthic community structure. Data were also collected by the Columbia River Integrated Environmental Monitoring Program, a coalition of Canadian environmental agencies and industries that studies and monitors the Columbia River in Canada. The investigation collected samples from the major, and selected minor, tributaries to the Columbia. Five background samples were collected from locations in the Kootenay River and Lower Arrow Lake, upstream of the Cominco and Celgar sites, and roughly 35 to 45 miles upstream from the border. The analytical results indicated that concentrations of arsenic, cadmium, copper, lead, mercury, and zinc in samples collected from the Columbia River and Lake Roosevelt were elevated relative to background concentrations. The investigators also compared the sediment concentrations to benchmarks developed by the Ontario provincial government, in particular, Ontario's "severe effects level", the concentrations at which pronounced impacts to benthic organisms may be expected to occur. Trace element concentrations (copper, lead, or zinc) exceeded Ontario's "severe effects level" at 28 locations in the Northport reach of the Columbia River and Lake Roosevelt. However, because the total trace element analysis methods used in the study yield higher concentrations than the total recoverable methods on which the Ontario standards are based, a direct comparison was not possible, and the actual number of locations with severe-effects concentrations were determined likely to be less than 28. The USGS assumed that total trace element concentrations would not exceed total recoverable concentrations by more than 100 percent, and based on this, calculated that the Ontario "severe effects level" was exceeded at 18 sample locations (E&E 2000).

The USGS determined that trace element concentrations were higher in suspended sediments than bed sediments, but that concentrations in unfiltered water samples were relatively low, and did not exceed water quality criteria. The USGS concluded that these findings reflected both the small concentrations of suspended sediment and the large dilution capacity of the river (E&E 2000).

The 1992 USGS investigation found that benthic invertebrate communities in the erosional habitats of the Columbia River resembled those commonly associated with contaminated or habitat-degraded areas. Communities displayed relatively low abundance and diversity, and were dominated by stress-tolerant organisms. Although communities in the depositional habitats were more difficult to assess due to the naturally low diversity of benthic organisms in reservoirs, USGS concluded that it was likely these communities had also been affected by the elevated concentrations of sediment-bound trace elements (E&E 2000).

Concentrations of PCBs, TCDDs, and TCDFs in the Columbia River began to be a concern in the 1980s following the discovery of elevated levels of TCDFs in lake whitefish that were collected just downstream of the Celgar facility in Castlegar, British Columbia. As a result, Health and Welfare Canada issued a health advisory in May 1989, recommending that people limit their consumption of lake whitefish. From 1990 to 1998, USGS and Ecology conducted investigations to determine concentrations of PCBs, TCDDs, and TCDFs in Lake Roosevelt fish tissue, and to evaluate the risks associated with human consumption of those fish.

In 1990, 1992, and 1993, Ecology carried out monitoring programs of TCDDs and TCDFs in suspended sediment and fish tissue to determine concentration trends. Ecology concluded that the primary historical source of furans and dioxins in Lake Roosevelt is the Celgar pulp mill. Ecology found that concentrations of these contaminants had decreased generally with time over their study period, and credited changes in pulp production and wastewater treatment methods used at the mill. A total maximum daily limit (TMDL) for 2,3,7,8-TCDD of 2.3 milligrams per day was established for the Columbia River at the U.S./Canada boundary, which limits the combined discharge of this contaminant from all upstream sources (E&E 2000).

In 1998, the USGS examined contaminant levels in rainbow trout within the study area and compared them to levels found in 1994. They concluded that most of the contaminants identified as potential threats to human health had decreased, with the exception of PCBs (determined by the presence of Aroclor 1254), which did not appear to have significantly changed. Concentrations were higher in fish from the upper reach of the river than in those from the lower reach; therefore, the two areas were evaluated separately. The median concentration of Aroclor 1254 in wild rainbow trout in the upper reach was 24 µg/kg in 1998, compared to 21.6 µg/kg in 1994; however, the USGS determined no significant statistical difference between the two years because of high variability. A significant difference was found among three trout groups in the lower reach. Multiple comparison tests found no significant difference between the 1994 mixed rainbow trout (median 15.6 ppb) and the 1998 wild rainbow trout (median 15.5 ppb), but the concentration of Aroclor 1254 in 1998 net pen rainbow trout (median 5.7 ppb) was significantly lower (E&E 2000).

The Ecology studies in 1992 and 1993 also included analyses of metals in suspended particles samples collected by continuous-flow centrifuge at Northport. Since there are no standards for metals in suspended particles, Ecology used Ontario benchmarks for bottom sediments for comparison purposes. Ecology determined that aside from the arsenic concentrations found in the 1993 particulate sample, the concentrations of arsenic, cadmium, copper, lead, mercury, and zinc all exceeded Ontario's "severe effects levels" for trace metals in bed sediments (E&E 2000).

In 1994, the USGS, in cooperation with the CCT, analyzed fish tissue to determine concentrations of mercury and other metals in three fish species (walleye, smallmouth bass, and rainbow trout) in the Upper Columbia River. Though samples of each of the fish species contained detectable concentrations of mercury, the highest levels were found in the walleye samples, at concentrations ranging from 0.11 mg/kg to 0.44 mg/kg. Walleye are predatory fish; therefore, their potential to bioaccumulate mercury is higher. Mercury concentrations were also found to correlate well with fish age. While the Federal Food and Drug Administration (FDA) standard of 1.0 ppm was not exceeded, the USGS and the Washington Department of Health (WDOH) issued a fact sheet summarizing the study and advising the public to limit consumption of Lake Roosevelt walleye (E&E 2000).

In August, 1999, representatives of the CCT submitted a petition for assessment of release to the EPA, requesting Region X to conduct a Preliminary Assessment of the hazards to public health and the environment in the area of the Upper Columbia River Basin from the Canadian Border, southward through Lake Roosevelt, to the Grand Coulee Dam, encompassing the water, river- and lake-beds, and banks (Short Cressman and Burgess PLLC 1999). The Preliminary Assessment was completed in 2000, and concluded that further investigation of the area under CERCLA was warranted (E&E 2000).

1.2.3.2 Migration/Exposure Pathways and Targets

This section discusses the surface water migration pathway and potential targets within the site's range of influence. The groundwater migration, soil exposure, and air migration pathways are not being assessed as part of this ESI sampling effort.

1.2.3.2.1 Surface Water Migration Pathway

Columbia River flow rates at the international boundary averages 101,000 cubic feet per second (cfs), and 103,340 cfs at Northport. The river's upland drainage area covers an estimated 85,000 square miles. The two-year, 24-hour rainfall event for the region averages approximately 1.5 inches (E&E 2000).

Surface water is used for domestic purposes, irrigation, stock watering, fire protection, power generation, and commercial purposes. The municipal water supply for Grand Coulee is drawn from a surface water intake on Lake Roosevelt. A population of 1,103 is served through 394 residential connections. Water is sampled and analyzed quarterly to comply with Federal public water supply regulations. No exceedances of MCLs were reported between 1995 and 2000 (E&E

2000). Ecology's Water Rights Application Tracking System (WRATS) lists permits for surface water intakes along the upper Columbia River from the Grand Coulee Dam to the international boundary. Thirty-seven permits are on record listed as single or general domestic, and seven are listed as multiple domestic. Assuming each single domestic intake serves two people, and each multiple domestic intake serves 20, the total number of people served by surface water intakes located within between the Grand Coulee Dam and the international boundary is 1,317. This number should be interpreted as a conservative estimate since other listed surface water intake applications did not specify the source of the surface water and were not included in the estimate; the actual number of surface water intakes along the Columbia River may be higher (E&E 2000).

Fishing occurs on the Upper Columbia River for both recreation and subsistence. CCT states that the natural and cultural resources of the Upper Columbia River Basin are of central importance to the Colville Tribe's subsistence and culture (Stone, 2001). The Bonneville Power Administration has funded the construction and operation of two hatcheries on the watershed. A creel (fish catch) survey for the Columbia River upstream of Grand Coulee Dam conducted in 1993 included data collected from tribal campgrounds, NPS boat launches, and river access points. In total, 824,615 fish were reported harvested, equaling over 1.1 million pounds. Rainbow trout and walleye accounted for the highest number of fish and the majority of weight (E&E 2000).

Sensitive environments within the surface water TDL at the site include 5.34 linear miles of wetland frontage. Two federally listed threatened species, the bull trout and the bald eagle, are known to exist. As mentioned previously, the Lake Roosevelt National Recreation Area is a unit of the national park system (E&E 2000).

Tribally sensitive areas of central importance to the CCT include areas used for cultural, ceremonial, traditional, subsistence, and economic purposes. In 1891, the Colville Tribes ceded the North Half of the Reservation to the United States, which includes a portion of the Columbia River, with expressly reserved hunting and fishing rights. The Tribes continue to rely heavily on the anadromous fisheries that exist down-river from Grand Coulee Dam. The Tribes have also become more reliant on the resident fishery and water resources above the dams for subsistence, recreation, and economic development, as they continue efforts to create jobs and improve the quality of life for Tribal members and the broader community (E&E 2000).

1.2.3.3 Areas of Potential Contamination

Based on a review of background information and discussions with site representatives, sediments of the Upper Columbia River and Lower Pend Oreille River have been identified for inspection under the Upper Columbia River/Lake Roosevelt ESI. Site Investigation sediment sampling will be conducted in areas considered potential contamination sources and in areas that may have been contaminated through the migration of hazardous substances.

Numerous potential sources of contamination such as mine, mill, and smelter sites, are located within the Columbia River watershed. Creeks draining through or near the mine sites may be

impacted by potential contaminant sources, and transport contaminants to the river. Lake Roosevelt, the Upper Columbia River, and the Pend Oreille River are recreation areas where sport and subsistence fishing/harvesting occurs. Potential contaminants of concern in sediments are Target Analyte List (TAL) metals including arsenic, cadmium, copper, lead, mercury, and zinc, and PCBs.

The municipal water supply for the City of Grand Coulee is drawn from Lake Roosevelt at an intake located near Grand Coulee Dam. Surface water samples will be collected from the vicinity of the municipal system intake and system Safe Drinking Water Act (SDWA) sampling points. Since this is a drinking water supply, potential contaminants of concern are volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), TAL metals and Pesticides/PCBs.

1.3 PROJECT/TASK DESCRIPTION AND SCHEDULE

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

1.3.1 Project Description

This section defines the objectives and scope for performing the ESI activities at the Upper Columbia River/Lake Roosevelt site. The main goals for the ESI activities are as follows:

- Collect and analyze samples to characterize the potential sources discussed in Section 1.2.3.3,
- Determine off-site migration of contaminants,
- Provide the EPA with adequate information to determine whether the site is eligible for placement on the National Priorities List, and
- Document a threat or potential threat to public health or the environment posed by the site.

1.3.2 Schedule

Sampling for the Upper Columbia River/Lake Roosevelt ESI will be completed in two stages. The first stage will include sampling Lake Roosevelt and Columbia River sites, as well as the tributaries north of Marcus Island without mine and/or mill sites. The first stage is scheduled to begin in mid-May to take advantage of relatively low water levels in the lake. Water levels are expected to be at approximately 1231' MSL on 14 May, and to rise approximately 1 foot per day until full pool elevation is reached at the end of June (Sprankle 2001). The second stage will include sampling on tributaries with mine and mill sites; the mines and mills will be investigated by E&E while WESTON collects tributary background and mouth samples. The proposed schedules for the field work presented below is 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, such as inclement

weather, difficulties in accessing a sampling site, 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. The proposed schedule for the first stage of fieldwork is shown in Table 1-1.

The proposed schedule for the second stage of fieldwork is shown in Table 1-2.

1.4 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The project data quality objectives are to provide valid data of known and documented quality to characterize sources, determine off-site migration of contaminants, determine whether the site is eligible for placement on the National Priorities List, and document threat(s) or potential threat(s) to public health or the environment posed by the site. The DQO process applied to this project followed that described in the EPA document, *Guidance for the Data Quality Objectives Process* (EPA 1994a).

1.4.1 DQO Data Categories

All samples collected under this QAPP 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 Objective Process for Superfund* (EPA 540/G-93/71).

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. Table 2-3 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 B and in the Laboratory Statement of Work (SOW) in Appendix D.

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 QAPP 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 FSP 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 the site, 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:

$$RPD = (100) \times \frac{(S_1 - S_2)}{(S_1 + S_2)/2}$$

where:

S_1 = normal sample value
 S_2 = 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 or 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, 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 required for this project include the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) class (and annual refreshers). Health and safety procedures for WESTON personnel are addressed in the WESTON site-specific health and safety plan. As previously discussed (Section 1.2), this document is maintained in WESTON's Seattle office. 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.

The operation of the portable x-ray fluorescence analyzer (XRF) to be used in field screening for this project will be operated by START personnel who have been specifically trained in its proper use and maintenance. WESTON will follow the EPA SOP for operation of the XRF unit, which is currently in preparation.

1.6 DOCUMENTATION AND RECORDS

This document is to be combined with information presented in WESTON's *Quality Management Plan* (QMP; WESTON 2001) for Region 10 START. This information is covered by the Standard Operating Procedures (SOPs) found in Appendix A, the Supplemental Forms found in Appendix B, the Commercial and CLP Laboratory Statements of Work, and the commercial laboratory Quality Assurance Manual, which has been previously reviewed by WESTON. A copy of the START QAPP is available in WESTON's Seattle office. Standards contained in the SOPs and the QMP will be used to ensure the validity of data generated by WESTON for this project.

Following the completion of fieldwork and the receipt of analytical data, information will be transferred to E&E, as directed by the EPA Task Monitor. Original project file materials including reports, analytical data packages, correspondence, chain-of-custody documentation, logbooks, corrective action forms, referenced materials, and photographs will be provided to the EPA TM at the close of the project.

Mine Sites SI
EPA Task Monitor
Monica Tonel
(206) 553-0323

Sediment SI*
EPA Task Monitor
Mark Ader
(206) 553-1808

EPA QA Officer
Ginna Grepo-Grove
(206) 553-1632

WESTON START
Project Manager
Keith Pine
(206) 521-7681

WESTON QA
Officer
Paul Swift
(206) 521-7625

EPA RSCC
Laura Castrilli
(206) 553-4323

E & E START
Project Manager
Leatta Dahlhoff
(206) 624-9537

ESAT FASP Team

WESTON START
Site Leader
Susan FitzGerald
(206) 521-7654

EPA Boat Operators
Dave Terpening
(206) 553-6905
Doc Thompson

E & E Field Team

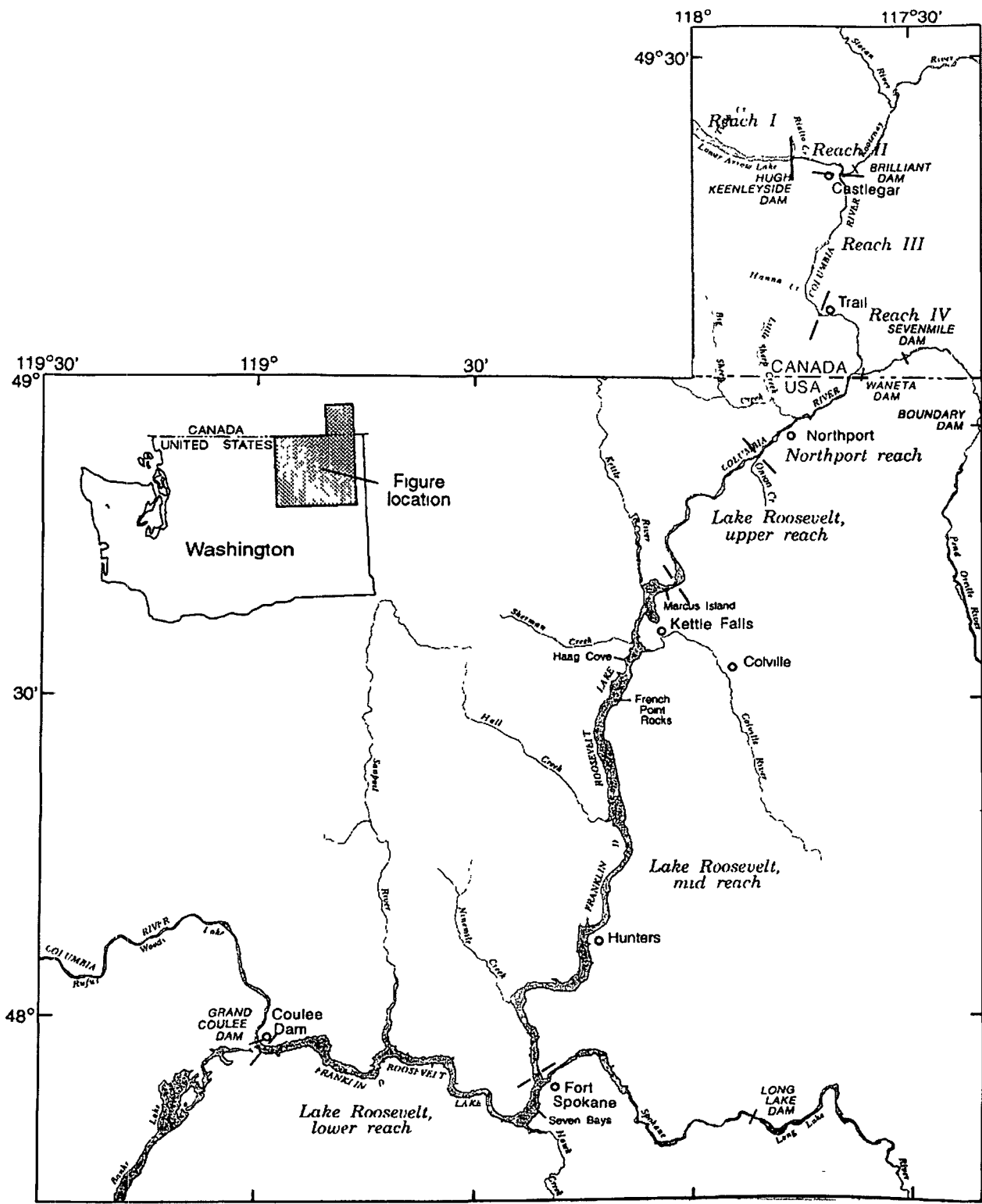
WESTON Field
Team
To Be Determined

CLP Laboratory
To Be Determined

* Approving Authority

Project Organization Chart
Upper Columbia River/Lake Roosevelt ESI
Washington





Source. Map adapted from USGS, 1994a



Site Vicinity Map Upper Columbia River/Lake Roosevelt SI



Figure
1-2

Table 1-1—Proposed Field Work Schedule (Stage One)

Activity	Start	Complete
Mobilize to Site	4/16/2001	5/13/2001
Sample Collection Activities	5/14/2001	6/14/2001
Laboratory Receipt of Samples	5/15/2001	6/15/2001
Demobilize from Site	6/15/2001	6/19/2001
Laboratory Analysis	5/15/2001	7/15/2001
Receipt of Data from Laboratory	6/13/2001	7/27/2001
Data Validation	7/30/2001	8/17/2001
Writing of Project Report	8/20/2001	9/21/2001
Target Project Completion Date	Not Applicable	9/21/2001

Table 1-2—Proposed Field Work Schedule (Stage Two)

Activity	Start	Complete
Mobilize to Site	6/4/2001	6/18/2001
Sample Collection Activities	6/18/2001	6/30/2001
Laboratory Receipt of Samples	6/19/2001	7/2/2001
Demobilize from Site	6/30/2001	7/3/2001
Laboratory Analysis	6/20/2001	7/30/2001
Receipt of Data from Laboratory	7/18/2001	8/13/2001
Data Validation	8/13/2001	8/24/2001
Writing of Project Report	8/20/2001	9/21/2001
Target Project Completion Date	Not Applicable	9/21/2001

SECTION 2

MEASUREMENT/DATA ACQUISITION

2.1 SAMPLING PROCESS DESIGN

This ESI is to be conducted through a cooperative effort involving both WESTON and E&E. WESTON will conduct sediment sampling to evaluate potential impacts to bed sediment along the Upper Columbia River and Lake Roosevelt, and at selected locations of the Pend Oreille River downstream of Metaline, Washington. To assess impacts to the river from tributaries, WESTON will conduct sediment sampling on selected tributaries in the project area. WESTON will also collect water samples from the City of Grand Coulee municipal water intake located in Lake Roosevelt at Grand Coulee Dam and from the system's usual SDWA sampling point. E&E personnel will be investigating potential releases from numerous mine sites in the tributary watersheds in the study area. This SQAP describes the proposed sampling and quality assurance procedures associated with those aspects of the investigation to be conducted by WESTON.

This section will describe sample locations (Section 2.1.1); the global positioning system (Section 2.1.2); logistics (Section 2.1.3); cooler return (Section 2.1.4); coordination with Federal, State, and local authorities (Section 2.1.5); and the proposed schedule (Section 2.1.6).

2.1.1 Sample Locations and Analyses

Sample locations have been selected to achieve the objectives discussed in Section 1.3.1.

Table 2-1 provides sample information regarding the sampling design and whether the measurement is considered critical or non-critical. Table 2-2 presents the anticipated number and types of samples, analytical methods, specific requirements for sample container size and type, sample preservation and holding times, and special handling requirements for samples expected to be collected at the site. Table 2-3 summarizes the number of QA/quality control (QC) samples to be submitted according to the method requirements. A summary of the sampling plan is presented in Table 2-4.

Proposed sample locations are illustrated on Plates 1 through 12. Specific sampling locations will be determined at the time of sampling based on observations made in the field. Conditions potentially contributing to deviations from the anticipated sampling locations include new observations or information obtained in the field that warrant altering the sampling approach, difficulty in reaching a desired sampling depth or achieving sufficient sample volume due to adverse 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 (Appendix B). Every attempt will be made to collect representative samples with the equipment being used.

A summary of sampling locations and rationale is provided below.

2.1 1.1 Upper Columbia River/Lake Roosevelt Sediments

The results of previous studies indicate that the greatest concentrations of sediment contaminants in the Upper Columbia River in the United States were generally found in the northern portions of the river and Lake Roosevelt. An estimated 55 samples of surface sediment are to be collected from the river banks between the international border (RM 745) and the vicinity of Inchelium (RM 675), a distance of approximately 70 RM. Sediment samples will be collected from the Columbia River in Canada at the direction of the EPA TM. All sediments will be collected from depositional environments located below the mean high water (MHW) elevation, and from depths ranging from the surface to up to eight inches (0.67-foot) bgs. Water levels during the sampling period are expected to be approximately 50 feet below full pool elevation; therefore, many of the sediment sampling locations are expected to be subaerial. The elevation of sampling locations will be estimated using visual observation of apparent mean high water shoreline level indicators or by the use of an altimeter. Exact sample locations and depths will be determined during the ESI field effort. Additionally, at the request of the CCT, samples from a subsurface interval of 1.5 ft to 2 ft bgs will be collected from two selected locations using a hand auger. One auger sample will be collected near the base of the North Dam boat ramp near Grand Coulee Dam, and the other auger sample will be collected near the south end of Marcus Island.

All sediment samples from the Upper Columbia River and Lake Roosevelt will be submitted to an off-site fixed laboratory for TAL metals (CLPAS ILMO4.1 and/or EPA SW-846 Methods 6010/6020/7000 Series), PCBs and Pesticides (Pesticides/PCBs (CLPAS) OLM04.2), and TOC (EPA SW-846 9060M). Trace metals and hydrophobic organic contaminants such as PCBs tend to naturally accumulate in fine-grained sediment deposits in streams (USGS 1994b); therefore, sediment sampling will be biased toward fine-grained sediment at most sample locations, except in the northern reaches of the Columbia River where slag deposits may occur as fine black sand. All potential exposed sediment sample locations will be screened *in situ* for metals concentrations using an XRF prior to selecting the sampling location in the field. A more detailed discussion of the sampling methodology is presented in Section 2.2.1.

2.1.1.2 Lake Roosevelt Surface Water

The municipal water supply for the City of Grand Coulee is drawn from Lake Roosevelt at an intake located near Grand Coulee Dam. It is estimated that two surface water samples will be collected, one from the vicinity of the municipal system intake and one at the system's usual SDWA compliance sampling point. The surface water samples will be submitted to an off-site fixed laboratory for analysis of VOCs (CLPAS, OLM04.2), SVOCs (CLPAS, OLM04.2), TAL metals (CLPAS, ILMO4.1) and Pesticides/PCBs (CLPAS, OLM04.2).

2.1.1.3 Pend Oreille River Sediments

The Pend Oreille is a major tributary to the Upper Columbia River. Like that of the Columbia, the Pend Oreille River watershed has a history of intensive mining and associated activities. Two major sites, the Pend Oreille Mine and the Grandview Mine, are located along the banks of the Pend Oreille just downstream of Metaline, WA. Three dams largely control sediment transport along the Pend Oreille from Metaline to its confluence with the Columbia River. Approximately four surface sediment samples will be collected at depositional points within the Pend Oreille River as detailed below.

- One sample will be collected from accumulated sediments in the reservoir immediately upstream of Waneta Dam.
- One sample will be collected from accumulated sediments in the reservoir immediately upstream of Seven Mile Dam
- One sample will be collected from accumulated sediments in the reservoir immediately upstream of Boundary Dam in the United States.
- One sample will be collected from the river at a location near the Grandview mine near Metaline Falls, Washington.

All sediments will be collected from depositional environments located below the MHW elevation, as estimated based on apparent mean shoreline level indicators or by an altimeter, and from depths ranging from the surface to eight inches (0.67-foot) bgs. Exact sample locations depths will be determined during the ESI field effort.

All sediment samples from the Pend Oreille River will be submitted to an off-site fixed laboratory for TAL metals (CLPAS ILMO4.1 and/or EPA SW-846 Methods 6010/6020/7000 Series), PCBs and Pesticides (Pesticides/PCBs (CLPAS) OLM04.2), and TOC (EPA SW-846 9060M). Trace metals and hydrophobic organic contaminants such as PCBs tend to naturally accumulate in fine-grained sediment deposits in streams (USGS 1994b); therefore, sediment sampling will be biased toward fine-grained sediment where it exists.

2.1.1.4 Upper Columbia/Lake Roosevelt and Pend Oreille River Tributary Sediments

Using USGS 7.5-minute topographic maps, a total of one-hundred-eighteen (110) tributaries draining into the Upper Columbia River and Lake Roosevelt were identified between the international border and the vicinity of Inchelium. The sediment in the 69 tributaries located from Marcus Island south to the vicinity of Inchelium will not be sampled during this field effort, but may be sampled at a later date. Of the remaining tributaries, approximately 11 have been identified as having had significant mining or milling operations within their watersheds; these tributaries will be sampled during the second stage of this sampling effort at the same time E&E is investigating mine and mill sites in the area. During stage one, surface sediment samples will be collected from the approximately 30 tributaries located along the river north of Marcus Island

to the international border that have not had significant mining or milling operations within their watersheds

Using USGS 7.5-minute topographic maps, a total of ten tributaries to the Pend Oreille River were identified in the United States between Metaline, Washington and the international border. Approximately five of these have been identified as having had significant mining or milling operations within their watersheds; these tributaries will be sampled during the second stage of this sampling effort (see Section 2.1.1.6 Mine/Mill Tributary Sediments). During stage one, surface sediment samples will be collected from the approximately 5 tributaries located along the river north of Metaline that have not had significant mining or milling operations within their watersheds.

Tributary sediment samples will be used to evaluate the potential contribution of the tributaries to contaminant levels in the river. The tributary samples will be collected from locations above the influence of the river as indicated by the MHW level elevation in the area. The elevation of sampling locations will be estimated using visual observation of apparent mean high water shoreline level indicators or by the use of an altimeter. All potential exposed sediment sample locations will be screened *in situ* for metals concentrations using an XRF prior to selecting the sampling location in the field. All sediments will be collected from depositional environments at depths ranging from the surface to eight inches (0.67-foot) bgs. Exact sample locations and depths will be determined during the ESI field effort.

All tributary sediments will be submitted to an off-site fixed laboratory for TAL metals (CLPAS ILMO4.1 and/or EPA SW-846 Methods 6010/6020/7000 Series).

2.1.1.5 River and Lake Background and Attribution Sediments

The Hazard Ranking System (HRS) requires the determination of background contaminant levels in order to identify instances of observed contamination within the study area using chemical analyses. The HRS dictates that background samples should be as similar as possible to the potential release samples, except for potential influence from sources. Similarity will be determined using sample and location information recorded at each sampling point. Background samples and release samples should be collected from similar water bodies (EPA 1994); therefore, it is anticipated that the background samples for Lake Roosevelt sediment will be collected from Lower Arrow Lake, upstream of the Hugh Keenleyside Dam, and background samples for the Upper Columbia River sediment will be collected from the Columbia River just downstream of Hugh Keenleyside Dam (upstream of the pulp mill at Castlegar). The Kootenay River is a major tributary to the Columbia River. To characterize sediment contributions from the Kootenay, attribution samples will be collected from the Kootenay River system at locations downstream of Brilliant Dam, and from locations along the Slocan River, a large tributary that joins the Kootenay roughly six miles upstream of Brilliant Dam. The sediment sample locations from the Kootenay will be selected to match the grainsize classes represented by the Columbia River sediment samples. Attribution samples for the Pend Oreille River sediment will be collected upstream of Metaline, Washington. An estimated 4 to 16 background samples will be

collected, including approximately 2 to 8 for the Lake Roosevelt sediment samples, and 2 to 8 for the Columbia River sediment samples. An estimated 6 to 24 attribution samples will be collected including approximately 4 to 16 attribution samples from the Kootenay/Slocan Rivers and 2 to 8 for the Pend Oreille River. The exact locations and number of the background and attribution samples will be determined in the field, based on descriptions of river/lake samples (e.g., sediment grain size, organic content).

All background and attribution samples will be submitted to an off-site fixed laboratory for TAL metals (CLPAS ILMO4.1 and/or EPA SW-846 Methods 6010/6020/7000 Series), PCBs and Pesticides (Pesticides/PCBs (CLPAS) OLM04.2), and TOC (EPA SW-846 9060M). The results of the analyses of the background samples will be used to establish contaminant levels indicative of an observed release within the study area. Attribution sample analytical results will indicate contaminant levels upstream of the study area.

2.1.1.6 Mine/Mill Tributary Sediments

As part of the ESI, during stage two of this field effort, E&E will investigate potential releases at mine sites in the Columbia and Pend Oreille watersheds. Approximately 11 tributaries to the Upper Columbia River, and approximately nine tributaries to the Pend Oreille (including Cedar Creek, Church Creek, and the Salmo River in British Columbia), contain mine/mill sites within their watersheds in the United States. E&E will conduct sampling at the mine/mill sites including the PPE(s). In watersheds identified by E&E as having had a potential release of contaminants to the surface water pathway, WESTON personnel will collect two mine/mill tributary sediment samples; one at a location upgradient of all mine/mill sites in the watershed, and another just upgradient of the stream's point of confluence with the receiving river (as indicated by MHW level of the river). Analytical data from each upgradient and confluence stream sediment sample will apply only to the particular watershed from which it was collected. Based on an estimate of the number of tributaries on which mine/mill sites exist, tributary upgradient and tributary mouth sediment samples will potentially be required on approximately 11 tributaries to the Upper Columbia River, and approximately 9 tributaries to the Pend Oreille, for an estimated total of 20 tributary upgradient samples and 20 tributary mouth samples.

All mine/mill tributary sediments will be submitted to an off-site fixed laboratory for TAL metals (CLPAS ILMO4.1 and/or EPA SW-846 Methods 6010/6020/7000 Series), PCBs and Pesticides (Pesticides/PCBs (CLPAS) OLM04.2), and TOC (EPA SW-846 9060M). All exposed potential tributary sediment sample locations will be screened for metals concentrations using an XRF prior to selecting the sampling location in the field.

2.1.1.7 Lake Roosevelt Background Surface Water

Background concentrations for Lake Roosevelt surface water will be determined through analysis of one surface water sample collected from Lower Arrow Lake in B.C., behind the Hugh Keenleyside Dam.

2.1.2 Global Positioning System

Global positioning system (GPS) readings from a Trimble GeoExplorer will be used to record the coordinates of every sample location. The data will be stored in location-specific individual files, the names of which will be noted on the field sampling records. WESTON field personnel have been instructed in the use of the GeoExplorer units by Matt Gubitosa of EPA. Differential corrections (DGPS) of the GPS data to improve location accuracy will be performed by Mr. Gubitosa through post-processing following the field effort. DGPS coordinates will be provided in the final ESI report as an appendix.

2.1.3 Logistics

The sampling points in the project area will be accessed by boat or by land. Boats used in the field sampling in the United States will be provided by the EPA Region 10's Investigations and Engineering Unit. The US Parks Service has also agreed to provide backup vessels, and to serve as an emergency contact, if needed. Boats used in the field sampling in Canada will be provided by a private contractor. Written or verbal access consent will be obtained from property owners prior to conducting ESI field sampling on private properties.

Sample aliquots collected for fixed laboratory analysis will be delivered to the EPA Region 10 laboratory or an alternative laboratory as directed by the EPA. All samples will be shipped by commercial airlines. Sample control and shipping are discussed in Section 2.3.

2.1.4 Cooler Return

For laboratories other than the EPA Region 10 laboratory, WESTON will provide a completed air bill accompanied by a plastic envelope with adhesive back and an address label in the chain-of-custody bags taped to the lid inside coolers so the laboratory can return the coolers to WESTON. The air bill 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-S0-01-02." This notation will enable the laboratories to return the sample coolers to WESTON's warehouse. The air bills 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 WESTON for cooler return in this manner is not required.

2.1.5 Coordination with Federal, and Local Authorities

The START will keep the TM informed of sampling progress and issues that may affect the schedule or outcome of the ESI, problems encountered, unusual contacts with the public or the media, and obtain guidance from the EPA regarding project activities when required. Additionally, the START will notify the EPA RSCC with changes to the sampling schedule for MEL and/or CLP analyses and provide shipping information on every sample shipment within 24

hours of shipment or before noon on Friday for Saturday delivery. All samples will be shipped to the laboratory within 48 hours of sample collection except when circumstances prevent delivery to the shipping company within business hours.

WESTON will notify property owners/operators, and tribal representatives prior to the initiation of field activities.

2.2 SAMPLING METHODS' REQUIREMENTS

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

2.2.1 Sampling Methodologies

Sediment and surface water samples will be collected using methodologies described in Appendix A.1 Sediment Sampling SOP No. RFW/R10-003, and A.2 Surface Water Sampling SOP No. RFW/R10-004. The START Site Leader and EPA TM will be responsible for ensuring that appropriate sample collection procedures are followed and will take appropriate actions to correct the deficiencies. All samples collected will be maintained under chain-of-custody and will be stored and shipped in iced coolers.

All sediment samples for this ESI will be collected as distinct grab samples, except in instances where low volume recovery in sampling equipment necessitates compositing individual grab volumes from a sample location, which will be noted on the field sampling record.

2.2.1.1 River and Lake Sediment Sampling

Sediment samples will be collected from river and lake environments along the Upper Columbia and Pend Oreille Rivers. Along the Upper Columbia River and Lake Roosevelt, metals concentrations at each potential exposed sediment sampling area will be screened using a portable x-ray fluorescence (XRF) analyzer prior to sample collection. Approximately three locations within each area will be screened *in situ*, and the area showing the highest concentrations will be sampled. XRF screening will not be conducted on sediment along the Pend Oreille River, as daily fluctuating water levels reported along the river upstream of Boundary Dam suggest the moisture content in surface sediment in this area is likely to make it unsuitable for XRF screening. The surface sediment will be collected using dedicated stainless steel spoons, where feasible, to depths that will be determined in the field. If necessary, subaqueous sediments will be sampled using a van Veen or ponar-type grab sampler deployed from a boat. Two selected locations will be sampled at a subsurface interval of 1.5-ft to 2-ft bgs using a hand auger. XRF screening will not be conducted on subaqueous or subsurface sediment.

As stated previously, sampling will be generally biased toward fine-grained sediments. Sediment will be collected from the minimum surface area required to gather a sufficient sample volume.

Except at the two hand-augered locations, sediment sample depths are not expected to exceed eight inches (0.67 ft) below the surface. A description of the site location and conditions will be recorded at the time of sampling. The sampled sediment will be placed in a dedicated stainless steel bowl, and examined by a trained geoscientist for physical description. The geoscientist will examine the sediment using a hand lens, and sediment descriptions including grain size proportions (e.g., estimated percent clay, silt, sand, and gravel), estimated percentages of slag and organic content, sample color, odor, etc. will be recorded on the field sampling record (Appendix B). Each sample will be photographed, then homogenized and placed into pre-labeled sample containers.

The HRS guidance document (EPA 1992) states that sediment types be similar in background and release samples, and “visual documentation of sediment type similarity is generally sufficient.” In addition to the surface sediment field sampling record, a matrix of sample data will be maintained to aid in the selection of background sample locations. The matrix will classify each sample based into one of four categories according to the estimated percentage of fine grained sediment (silt and clay) or “fines” in each sample:

1. < 25% fines
2. Greater than 25% but less than 50% fines
3. Greater than 50% but less than 75 % fines
4. >75% fines

2.2.1.2 Tributary Sediment Sampling

Sediment samples will be collected from areas of exposed sediment using dedicated stainless steel spoons, where feasible. If necessary, subaqueous sediments will be sampled using an Ekman-type grab sampler or hand auger. The surface sediment will be collected using dedicated stainless steel spoons, to depths that will be determined in the field. Metals concentrations at each potential exposed sediment sampling area will be screened using a portable XRF analyzer prior to sample collection. As with river sampling, tributary sampling will be generally biased toward fine-grained sediments. Sample depths are not expected to exceed eight inches (0.67 ft) below the surface. Sediment will be collected from the minimum surface area required to gather a sufficient volume for the sample. A description of the site location and conditions will be recorded at the time of sampling. The sampled sediment will be placed in a dedicated stainless steel bowl, and a physical description of the sample will be recorded by a trained geoscientist, as described above. The sediment will then be photographed, homogenized, and placed into pre-labeled sample containers.

2.2.1.3 Surface Water Sampling

Two water samples will be collected, one from Lake Roosevelt in the vicinity of the Grand Coulee municipal water intake, and one at the system SDWA sampling point. The lake surface water sample will be collected either by hand dipping the sample container into the water, if possible, or by lowering an appropriate sampling device (e.g., Kemmerer bottle) to the desired depth, then decanting the collected water into a pre-labeled sample container. The drinking water

sample will be collected by direct sampling from the system's sampling port. At each sampling point, the sample volume for VOC analysis will be collected first, followed by the sample for SVOC analysis, then the sample volumes for the remaining analytes.

2.2.2 Sampling Equipment Decontamination

Every effort will be made to minimize the need for decontamination of sampling equipment through to the use of dedicated sampling equipment (e.g., bowls, spoons); however, the use of non-dedicated sampling equipment (e.g., dredge samplers) may be required in some locations. In these cases, the non-dedicated sampling equipment will be decontaminated prior to each use to avoid sample cross-contamination.

The following procedure (as listed in Appendix A) is to be used for all nondedicated 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 or river water.
3. Rinse thoroughly with analyte free water.
4. Use a solvent rinse (isopropanol) if the sample will be analyzed for organics and high concentrations are anticipated.
5. Air-dry the equipment, if possible, away from potential sources of contamination (e.g., splashes).

2.2.3 Investigation-Derived Wastes

WESTON will make every effort to minimize the generation of investigation-derived wastes (IDW) that cannot be disposed of as solid waste. All extra sediment volume collected for a sample will remain at the sampling location. Disposable personal protective clothing and sampling equipment generated during field activities will be torn or cut to avoid reuse, double-bagged in plastic garbage bags, labeled, and disposed of at an approved solid waste disposal facility. Decontamination water containing solvents will be considered a hazardous waste. To minimize generation of hazardous waste decontamination water, solvents will be used sparingly and separated from non-hazardous decontamination water (e.g., containing only Liquinox). All hazardous IDW will be handled and disposed of in an EPA-approved manner.

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

This section describes sample identification and chain-of-custody procedures that will be used for the Upper Columbia River/Lake Roosevelt ESI field activities. The purpose of these procedures is to ensure that the quality of samples is maintained during collection, transportation,

storage, and analysis. All chain-of-custody requirements comply with WESTON's SOPs for sample handling. All sample control and chain-of-custody procedures will follow the *User's Guide to the Contract Laboratory Program* (EPA 1991).

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

- Sample identification numbers,
- Sample tags or labels,
- Custody seals,
- Chain-of-custody and traffic report records,
- Field logbooks,
- Sample collection forms,
- Analytical request forms, and
- Analytical records.

During the field effort, the site manager or delegate is 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),
- Chain-of-custody record numbers,
- Bottle lot numbers, and
- Air bill 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 Regional Sample Coordination Center. Each sample label will be affixed to the jar and covered with Mylar 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 numbers, all samples will be assigned a unique identification code based on a consistent sample designation scheme that will be used internally. The sample designation scheme is designed to suit the needs of the field staff, data management, and data users, and will not be provided to the laboratory. The sample identification code is described below and summarized in Table 2-5.

All sample identification codes will consist of three components separated by a dash. These components are station code, consecutive sample number, and media code. The sample designation scheme is as follows:

Station Code	Sample Number	Media Code
SS	nnn	MM

The three components are described in the following sections.

Station Code

The station code component is a four-character code that identifies each sampling station. The station code component has two parts: a two-letter station designation XX indicating the area where the sample was collected. The station codes designated for this project are as follows:

- CR — Upper Columbia River/Lake Roosevelt
- TR — Tributary
- PO — Pend Oreille River
- UT — Upgradient Tributary
- BK — Background
- AT — Attribution
- RS — Rinsate
- TB — Trip Blank

Sample Number

The station code is followed by a three-digit “nnn” sequential numbering scheme (e.g., 001, 002, etc.).

Media Code

The media code is a two-character code that defines the media type of the sample. The media codes designated for this project are as follows:

- SD — Sediment
- SW — Surface water

The internal sample ID will not be entered on laboratory chain-of-custody forms; only EPA sample numbers will be used to submit samples for analysis. WESTON will maintain a cross-reference of the proposed sample identification codes and EPA sample numbers.

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 chain-of-custody

purposes, all QA/QC samples will be subject to the same custodial procedures and documentation as site samples.

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

- Sample number,
- Sample location number,
- Date and time of collection,
- Analysis required, 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 the seals are disturbed. Sample shipping containers (e.g., coolers, drums, and cardboard boxes, etc., 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 seals to ensure that seals are not broken accidentally during shipment. Upon receipt 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 chain-of-custody record and analytical traffic report forms will be completed as described in the *User's Guide to the Contract Laboratory Program* (EPA 1991). The chain-of-custody record and analytical traffic reports will be completed fully at least in duplicate by the field technician designated by the site manager as responsible for sample shipment to the appropriate laboratory. Information specified on the chain-of-custody record will contain the same level of detail found in the site 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 samples to the transporter, with the date and time of transfer noted and signature of the designated sample custodian at the receiving facility.

If samples require rapid laboratory turnaround, the person completing the chain-of-custody 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., air bill 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 air bill or delivery note, constitute a complete custody record. It is the site 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 Daily Logs and Data Forms*

Field 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. If corrections to log entries are necessary, these corrections 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 will be noted in the project or task log concerning photographs:

- Date, time, and location where photograph was taken;
- Photographer;
- Description of photograph taken;
- Reasons why photograph was taken;
- Sequential number of the photograph and the film roll number;
- Direction.

2.3.2 **Custody Procedures**

The primary objective of chain-of-custody 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 chain-of-custody rules.
- The Site Leader will record sample data in the field logbook.
- The Site Leader will determine whether proper custody procedures were followed during the fieldwork and will decide if 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 two copies of the chain-of-custody records. When transferring samples, the individuals relinquishing and receiving them must sign, date, and note the time on the chain-of-custody record. This record will document sample custody transfer.
- Samples will be dispatched to the laboratory for analysis with separate chain-of-custody records accompanying each shipment. Shipping containers will be sealed with custody seals for shipment to the laboratory. The chain-of-custody 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 chain-of-custody record before placement in the shipping container.
- All shipments will be accompanied by chain-of-custody records identifying their contents. The original record will accompany the shipment. The other copies will be distributed appropriately to the site team leader and site manager.
- 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 chain-of-custody agrees with samples received. The commercial laboratory will follow its internal chain-of-custody procedures as stated in the laboratory QA Manual.

2.4 ANALYTICAL METHODS REQUIREMENTS

This section discusses the analytical strategy (Section 2.4.1) and the 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 the laboratory 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 site 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 Port Orchard, Washington, or an alternative laboratory designated by the EPA, and the START-subcontracted commercial laboratory. EPA and/or CLP laboratory analyses will take place under the standard three-week turnaround time period, with validation by the EPA QA office for these analyses taking place under the standard two-week turnaround time period. Hardcopy results from the EPA and/or CLP laboratories will be delivered to the EPA upon completion of each sample delivery group. Electronic results from the EPA and/or CLP laboratories will be delivered to the EPA upon project completion. START-subcontracted laboratory analyses will take place under the standard four-week turnaround time period with validation by START chemists for these analyses taking place under the standard two-week turnaround time period. Hardcopy and electronic data results from the subcontracted commercial laboratory will be delivered to the START upon completion of each sample delivery group. Table 2-3 summarizes laboratory instrumentation and methods to be used for the ESI.

All instruments used during fixed laboratory sample analyses will be operated and calibrated in accordance with the analytical methods specified in the SQAP. Maintenance of field and laboratory equipment and instruments will be performed in accordance with the manufacturer's specifications. All of the analyses shall adhere to the technical and QC specifications outlined in the SQAP and the analytical methods.

In the cases where laboratory results exceed QC acceptance criteria, reextraction and/or reanalysis will occur as indicated in the applicable analytical method. Commercial laboratory

results (preliminary data) will be generally be available within 2 weeks of sample receipt. The respective laboratory analysts will be responsible to ensure that appropriate sample analysis procedures are followed and take appropriate actions to ensure deficiency correction. A copy of the Statement of Work for the commercial laboratory analyses, including copies of the applicable analytical methods, will be included in Appendix D upon completion and will be provided to the EPA prior to sample collection.

2.5 QUALITY CONTROL REQUIREMENTS

QC checks for sample collection will be accomplished by a combination of Chain-of-Custody protocols and laboratory quality assurance as prescribed in the sampling or analytical methods. No QC samples (i.e., double blind performance evaluation samples) are planned for this activity outside of normal laboratory QC criteria outlined in the analytical methods. These QC samples include rinsate and method blanks, matrix and surrogate spikes, and calibration check samples; results from these samples will be compared to QC requirements listed in Section 4.1.2.

With the exception of the XRF screening analyses, all of the other analyses that will be performed for this project will produce definitive data. Data quality indicator targets for this project are specified in section 1.4—Data Quality Objectives and are summarized in Table 2-3 of this SQAP. Bias on estimated flagged data shall be determined by the validation process. The laboratories' DQOs for completeness and the field team's ability to meet the DQO for representativeness are set at 90%. Precision and accuracy requirements are outlined in Table 2-3.

2.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

The field equipment used during this project includes the GPS unit and a field-portable x-ray fluorescence spectrometer. Testing, inspection, and maintenance of these instruments will be performed in accordance with the manufacturer's recommendations and/or the SOPs listed in Section 2.2.4. Spare parts for the field equipment will be available from the manufacturer generally within 48 hours of ordering.

All field instrumentation and equipment used for 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 WESTON 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 manufacturer's 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 site field logbook.

In addition to preventive maintenance procedures, daily calibration checks will be performed at least once daily before use and recorded in the respective logbooks. Additional calibration checks will be performed as required. All logbooks will become part of either the permanent site 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 manufacturer's guidelines and recommendations, as well as criteria set forth in the applicable analytical methodology references and/or in accordance with the laboratory's Quality Assurance Project Plan and SOPs.

For the field instrumentation (GPS unit, XRF unit, altimeter), the calibration will be performed in accordance with the manufacturer's recommendations and the SOPs listed in Section 2.2.4.

2.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

This information is covered by the SOPs and the START QMP (WESTON 2001). Standards contained in these documents will be used to ensure the validity of data generated by WESTON for this project. Sample jars are pre-cleaned by the manufacturer; certification documenting this is enclosed with each box of jars. The START will include this documentation as part of the site file. Non-dedicated equipment is demonstrated to be uncontaminated by the use of rinsate blanks.

2.9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

No data will be used from other sources.

2.10 DATA MANAGEMENT

This document is to be combined with information presented in WESTON's QMP for Region 10 START. Copies of the START QMP are available in WESTON's Seattle office. Standards contained in the START QMP will be used to ensure the validity of data generated by WESTON for this project. Data validation will be performed as listed in Section 4.1.2. Data tracking, storage, and retrieval are tracked through the TDD "blue sheet", which records where the paper

and electronic data are located. All paper data is stored in locked file cabinets; access to these files is restricted to key START personnel. Electronic data is archived by TDD.

START will use Access software and the following procedures to compile and manage two major but related types of data: GIS (or locational) data and chemical analytical data.

GIS data are collected as required by a given project. When GIS data are collected, the following fields are captured as appropriate:

- Station Identifier
- Station Alternate or Previous Ids
- Station Type Description
- Station Coordinates
- Station Horizontal Datum (if applicable)
- Method for determining Station Location
- Station Coordinate Units
- Station Elevation
- Station Vertical Datum (if applicable)
- Method for determining Station Elevation

and appropriate Station Attributes such as:

- Well Depth
- Well Screening Interval
- Depth of Water
- Sediment or Soil Sampling Interval.

The major data items captured to create a complete chemical analytical dataset are as follow:

- Station Identifier
- Sample Identifier
- Sample Description (Primary, Field, Duplicate, Replicate, etc.)
- Sample Date
- Full name of analytical parameter, observation or compound analyzed
- CAS number when available or appropriate
- Analytical result value
- Data validation qualifier
- Units
- Analytical Method
- Sample Media
- Sample Media modifier

2.10.1 Analytical Data Loading

The process of entering sample analytical data into a data management system is called analytical data loading. There are two primary strategies for loading analytical data: automated loading of Electronic Data Deliveries (EDDs), and direct hand entry from paper results. In general, EDDs are created by analytical laboratories and delivered with hard-copy (paper) results as a standard part of sample analysis.

Data management procedures will accommodate manual data validation, or for loading data on data sets that have been validated before they are delivered to WESTON. In the case of WESTON-validated data, no automated tools are needed to effect data validation changes, and an audit will lead to hard-copy laboratory results with validation changes noted and signed by qualified staff.

2.10.2 Data Pre-Processing

Before data entry, a hard-copy form of the data will be generated if not provided. Large volumes of source data are divided into manageable data entry groups. Each data entry group is supplied with a standard coversheet and assigned a sequential, unique data entry group ID. The data entry group ID serves as the basis for filing all data groups, and as the means of quickly tracking any datapoint to the data entry group from which it came.

2.10.3 Primary Dataset Development

The Environmental Data Management System (EDMS) will be used to capture and build a complete sample analysis dataset that matches source materials *exactly*. This dataset is complete when all inventoried sample analysis information has been received and entered, or if missing, has been properly accounted for. This complete dataset is called the *primary dataset*.

As it is the function of the primary dataset to represent source materials exactly, and is not appropriate for analysis, the primary dataset is maintained without modification. All post-processing is performed on copies of the primary dataset using a post-processing module. A copy of the primary dataset made for data analysis purposes is called an *analytical dataset*.

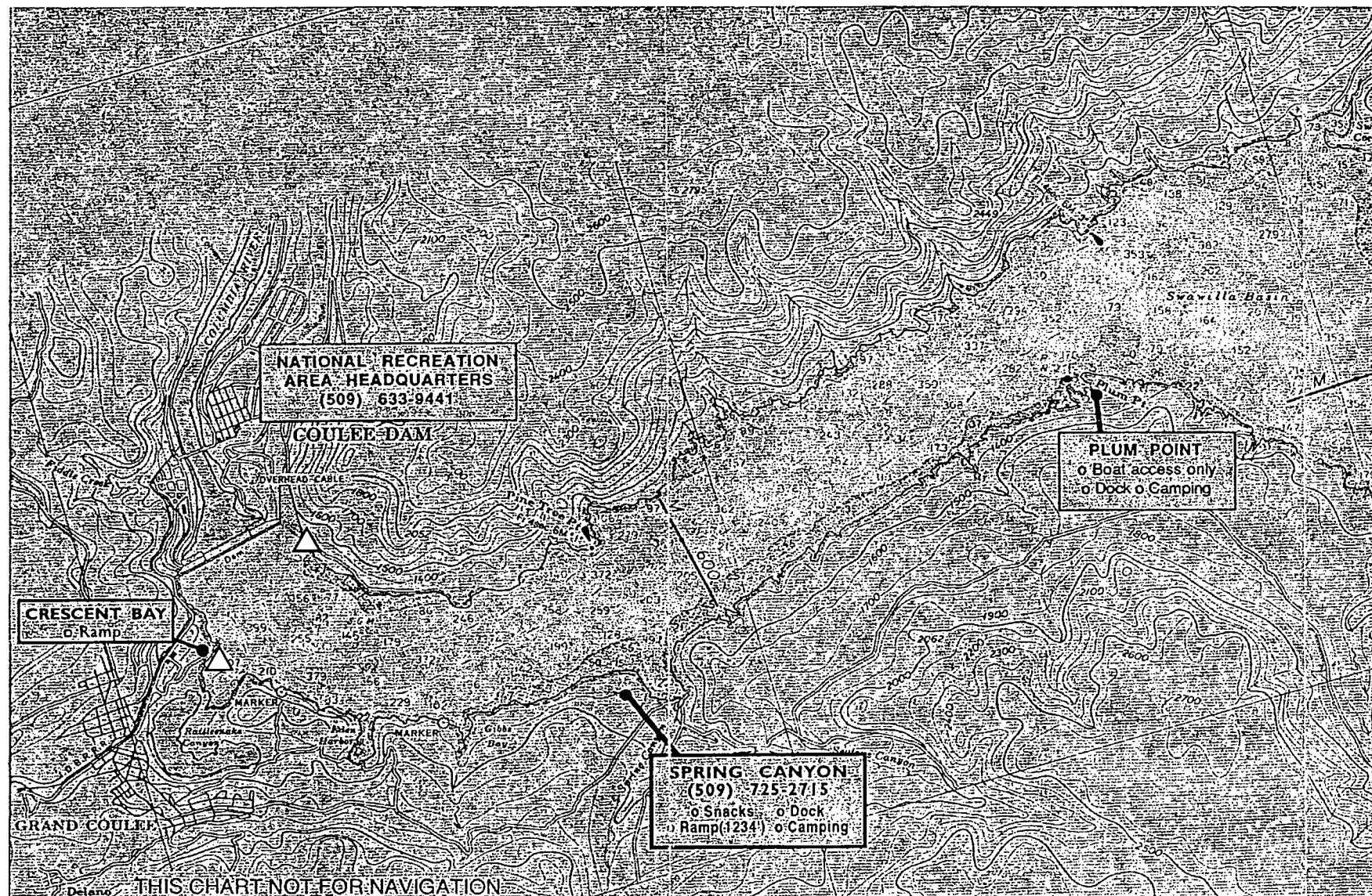
2.10.4 Data Post Processing

Several post-processing procedures are available, the major steps are to:

- Standardize reporting units
- Reassign analytical groups as necessary
- Detect and manage co-located datapoints
- Create compound totals (e.g., LPAHs, HPAHs)
- Total organic carbon normalization
- Perform data comparisons to reference values.

2.10.5 Analytical Dataset Development

The analytical dataset is complete once all appropriate post-processing steps are completed. The completed analytical dataset is used for all data analysis and for exporting to external analysis software such as GIS.



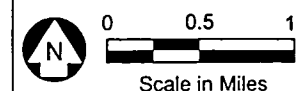
Notes:

- 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
- 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

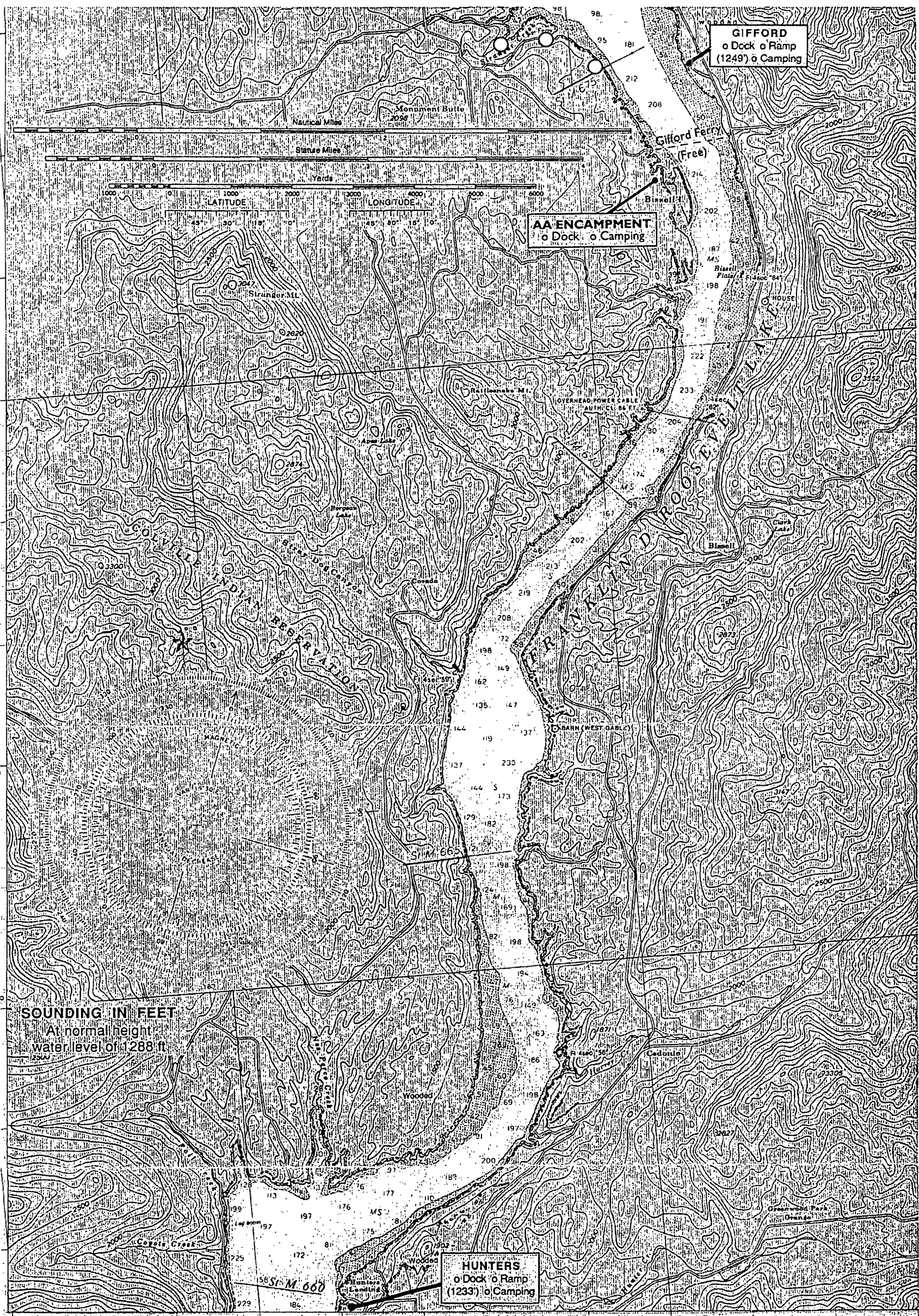
Source: Department of Mines and Resources, Surveys and Mapping Branch (1982, 1985, 1988)

LEGEND

△ Proposed River/Lake Sediment Sample Location



Proposed River/Lake and Tributary Mouth Sediment Sample Locations
Upper Columbia River/Lake Roosevelt SI



Notes:

- 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
- 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

Source: Franklin D. Roosevelt Lake, WA, Northern Part, Northwest Map Service 1995.

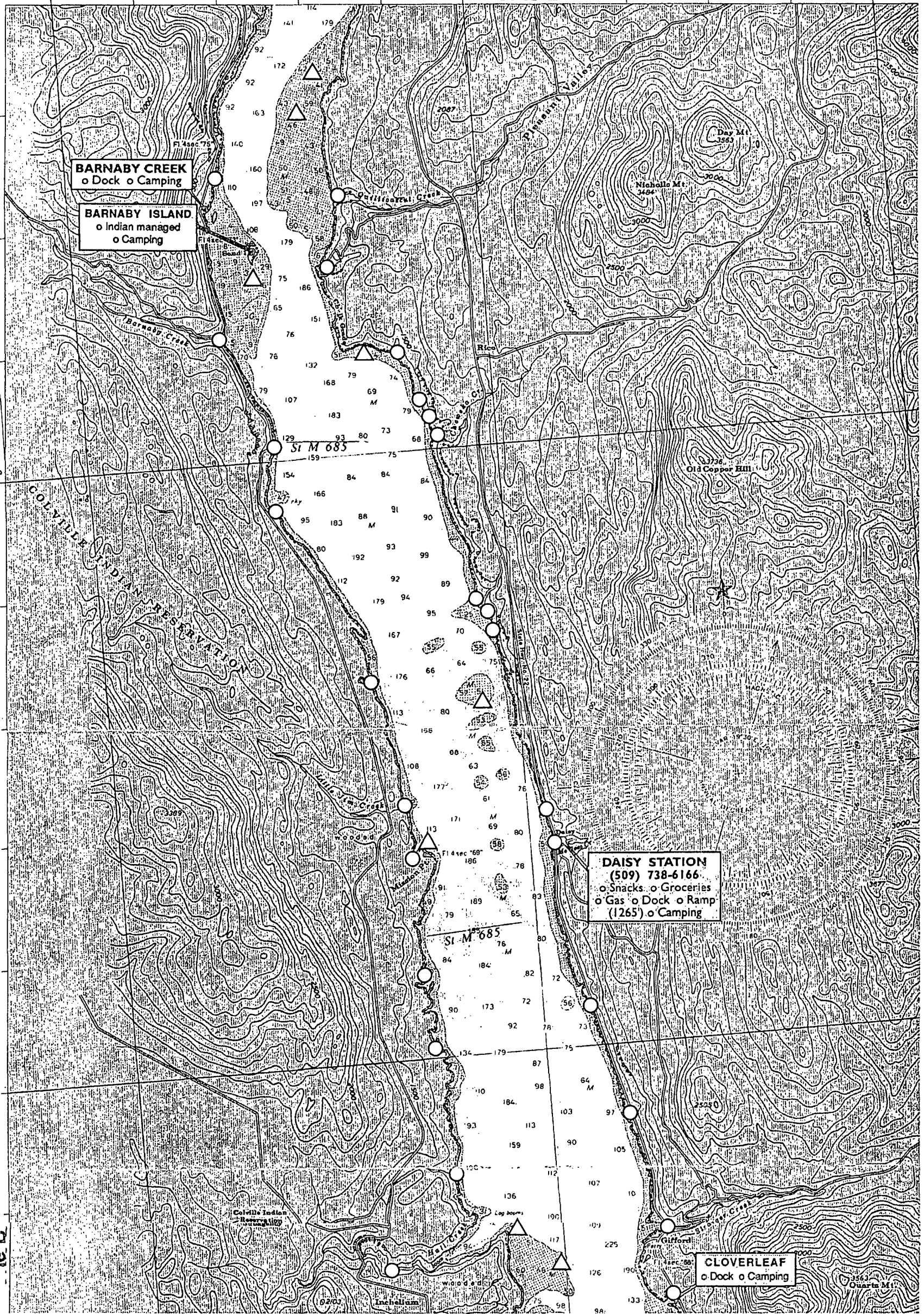
LEGEND

- Tributary Mouth to be Sampled at a Later Date



WESTON
MANAGERS DESIGNERS/CONSULTANTS

Proposed River/Lake and Tributary Mouth Sediment Sample Locations Upper Columbia River/Lake Roosevelt SI

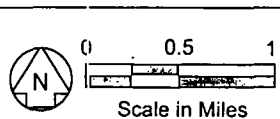


- Notes:
- 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
 - 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

Source: Franklin D. Roosevelt Lake, WA, Northern Part, Northwest Map Service 1995.

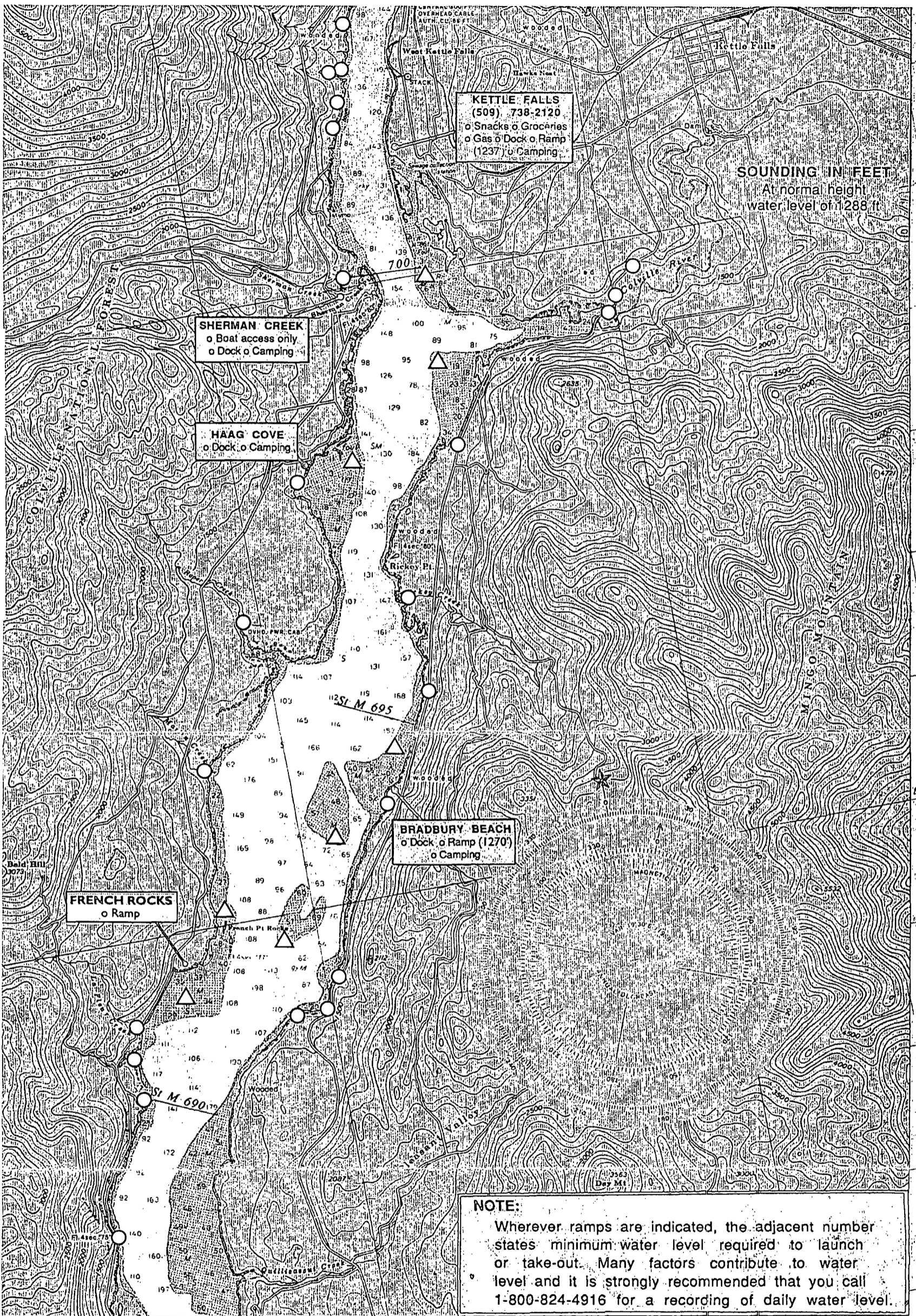
LEGEND

- △ Proposed River/Lake Sediment Sample Location
- Tributary Mouth to be Sampled at a Later Date



**Proposed River/Lake and Tributary Mouth Sediment Sample Locations
Upper Columbia River/Lake Roosevelt SI**



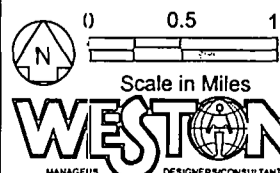


- Notes:
- 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
 - 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

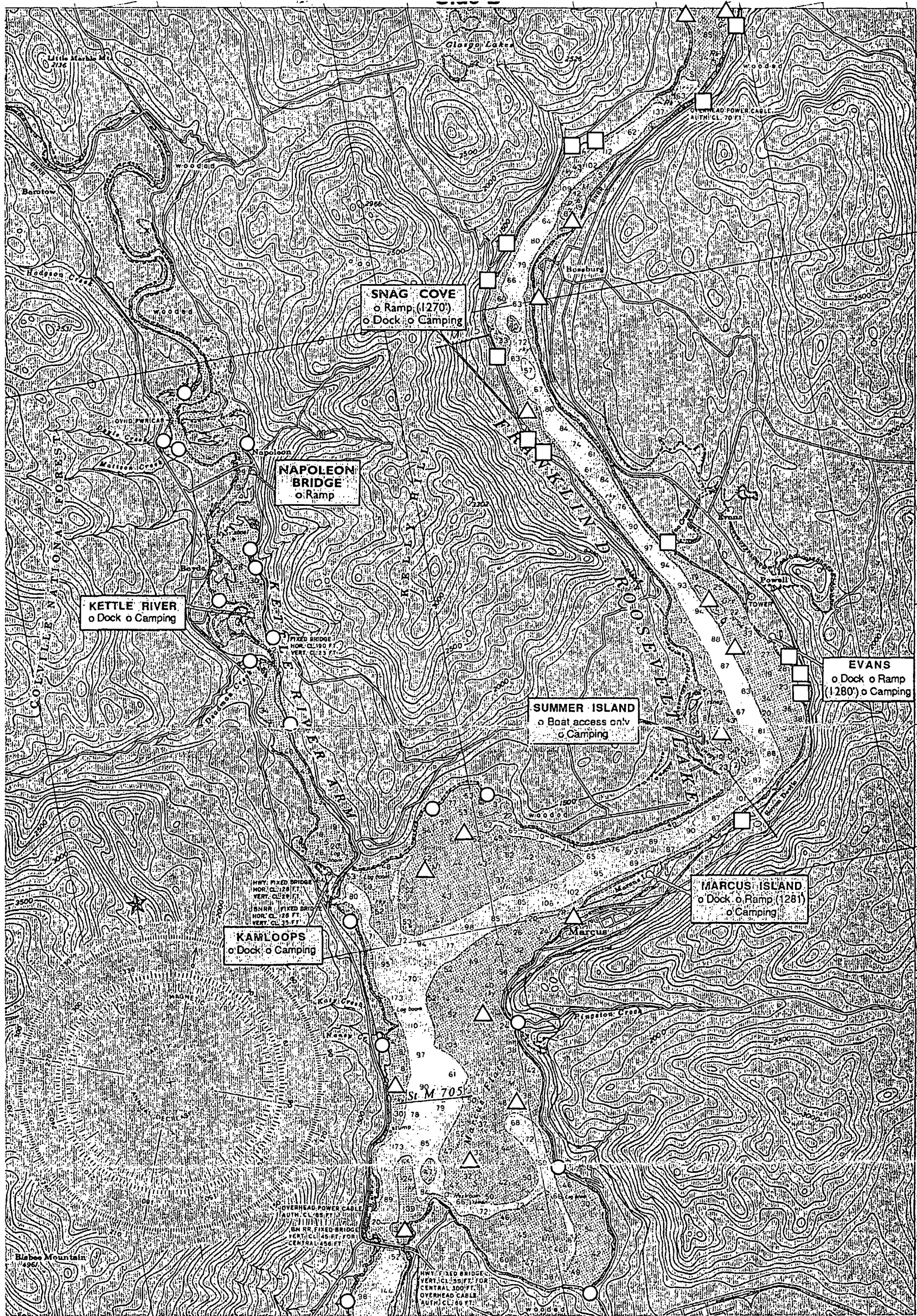
Source: Franklin D. Roosevelt Lake, WA, Northern Part, Northwest Map Service 1995.

LEGEND

- △ Proposed River/Lake Sediment Sample Location
- Tributary Mouth to be Sampled at a Later Date



**Proposed River/Lake and Tributary Mouth Sediment Sample Locations
Upper Columbia River/Lake Roosevelt SI**

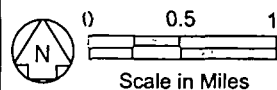


Notes:
 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

Source: Franklin D. Roosevelt Lake, WA, Northern Part, Northwest Map Service 1995.

LEGEND

- △ Proposed River/Lake Sediment Sample Location
- Proposed Tributary Mouth Sediment Sample Location
- Tributary Mouth to be Sampled at a Later Date



Scale in Miles
WESTON
 MANAGERIAL DESIGNERS/CONSULTANTS

**Proposed River/Lake and Tributary Mouth Sediment Sample Locations
 Upper Columbia River/Lake Roosevelt Sl**





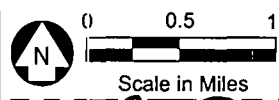
Notes:

- 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
- 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

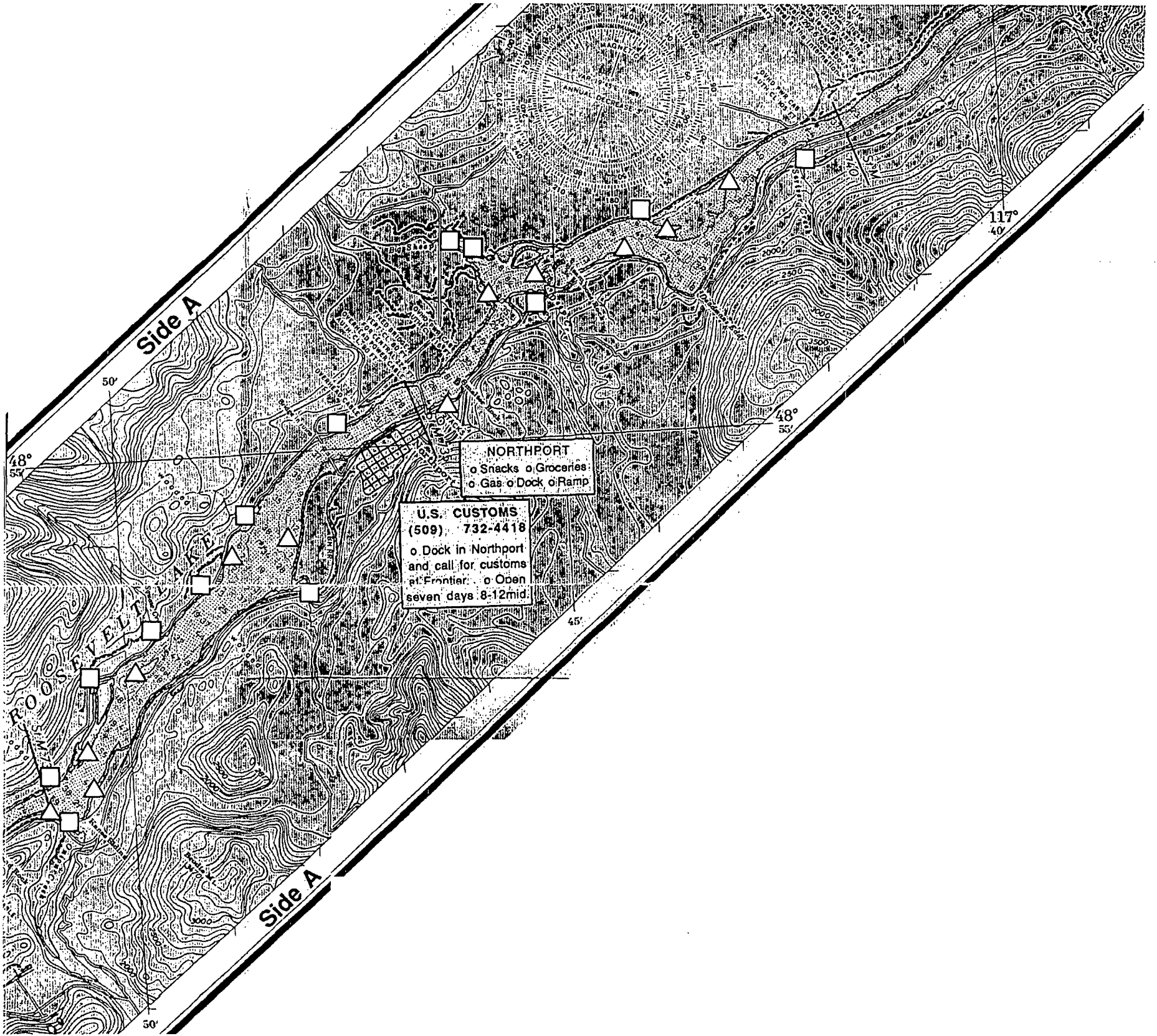
Source: Franklin D. Roosevelt Lake, WA, Northern Part, Northwest Map Service 1995.

LEGEND

-  Proposed River/Lake Sediment Sample Location
-  Proposed Tributary Mouth Sediment Sample Location

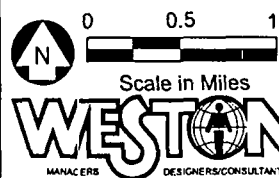


Proposed River/Lake and Tributary Mouth Sediment Sample Locations Upper Columbia River/Lake Roosevelt SI

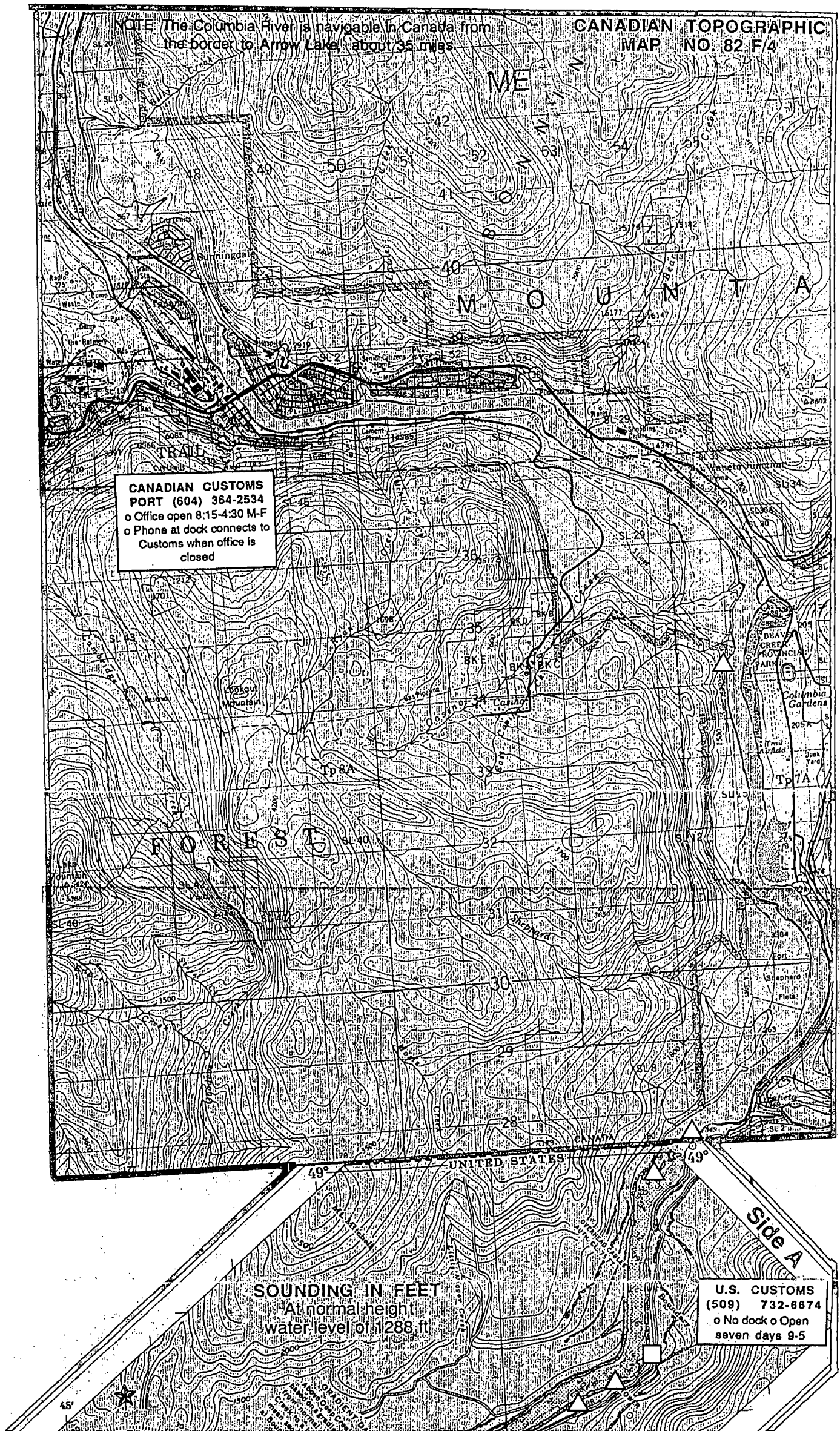


Notes:
 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

Source: Franklin D. Roosevelt Lake, WA, Northern Part, Northwest Map Service 1995.



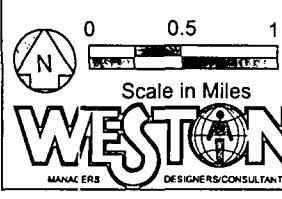
Proposed River/Lake and Tributary Mouth Sediment Sample Locations Upper Columbia River/Lake Roosevelt SI



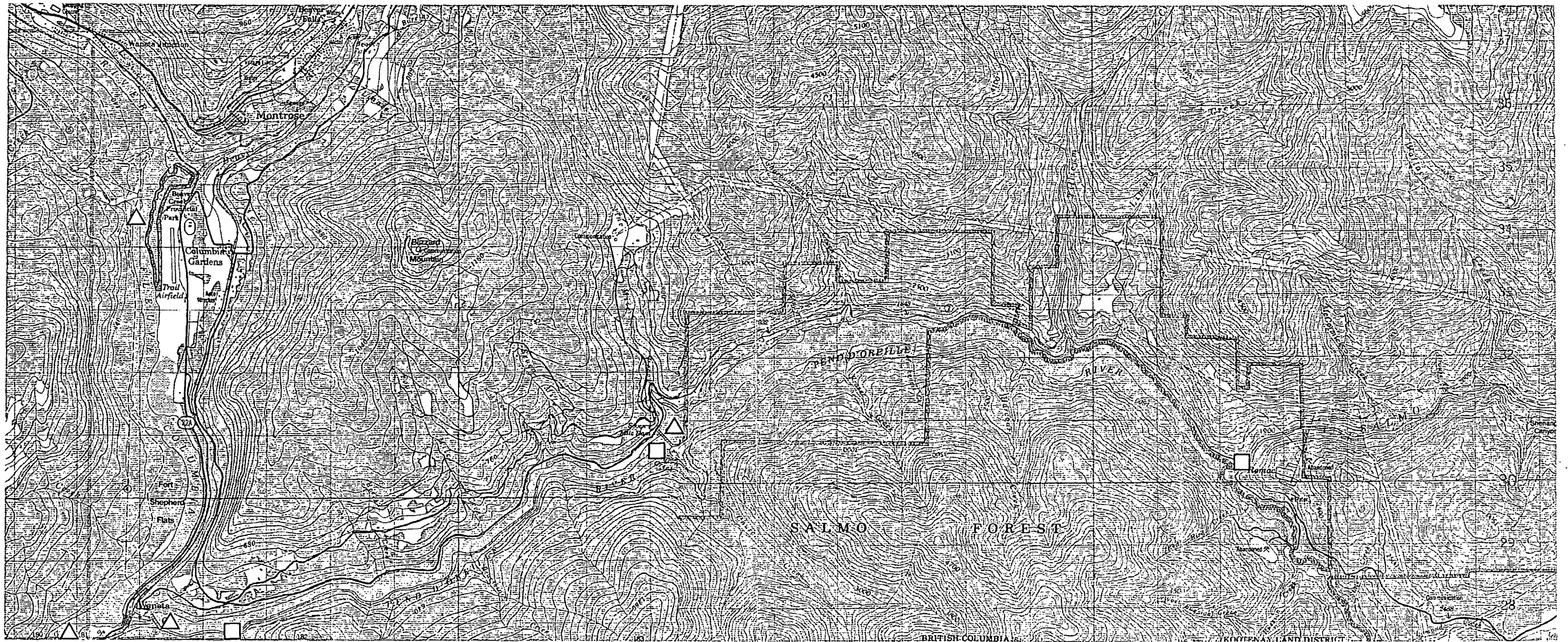
- Notes:
- 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
 - 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

Source: Franklin D. Roosevelt Lake, WA, Northern Part, Northwest Map Service 1995.

LEGEND	
△	Proposed River/Lake Sediment Sample Location
□	Proposed Tributary Mouth Sediment Sample Location



Proposed River/Lake and Tributary Mouth Sediment Sample Locations Upper Columbia River/Lake Roosevelt Sl





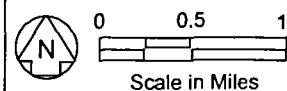
Notes:

- 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
- 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

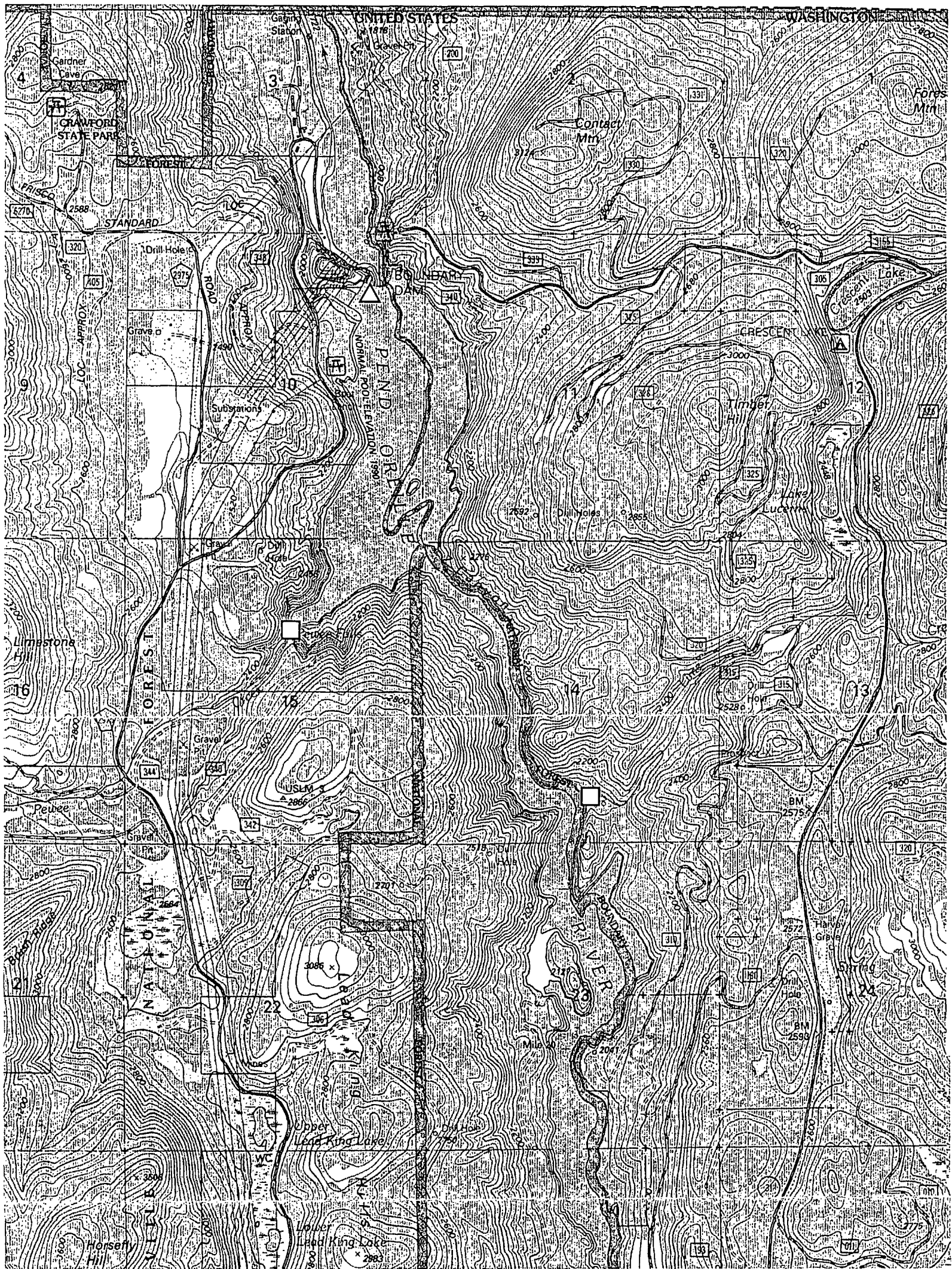
Source: Department of Mines and Resources, Surveys and Mapping Branch (1982, 1985, 1988)

LEGEND

-  Proposed River/Lake Sediment Sample Location
-  Proposed Tributary Mouth Sediment Sample Location



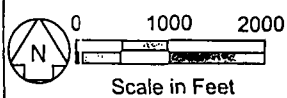
**Proposed River/Lake and Tributary Mouth Sediment Sample Locations
Upper Columbia River/Lake Roosevelt SI**



- Notes:
- 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
 - 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

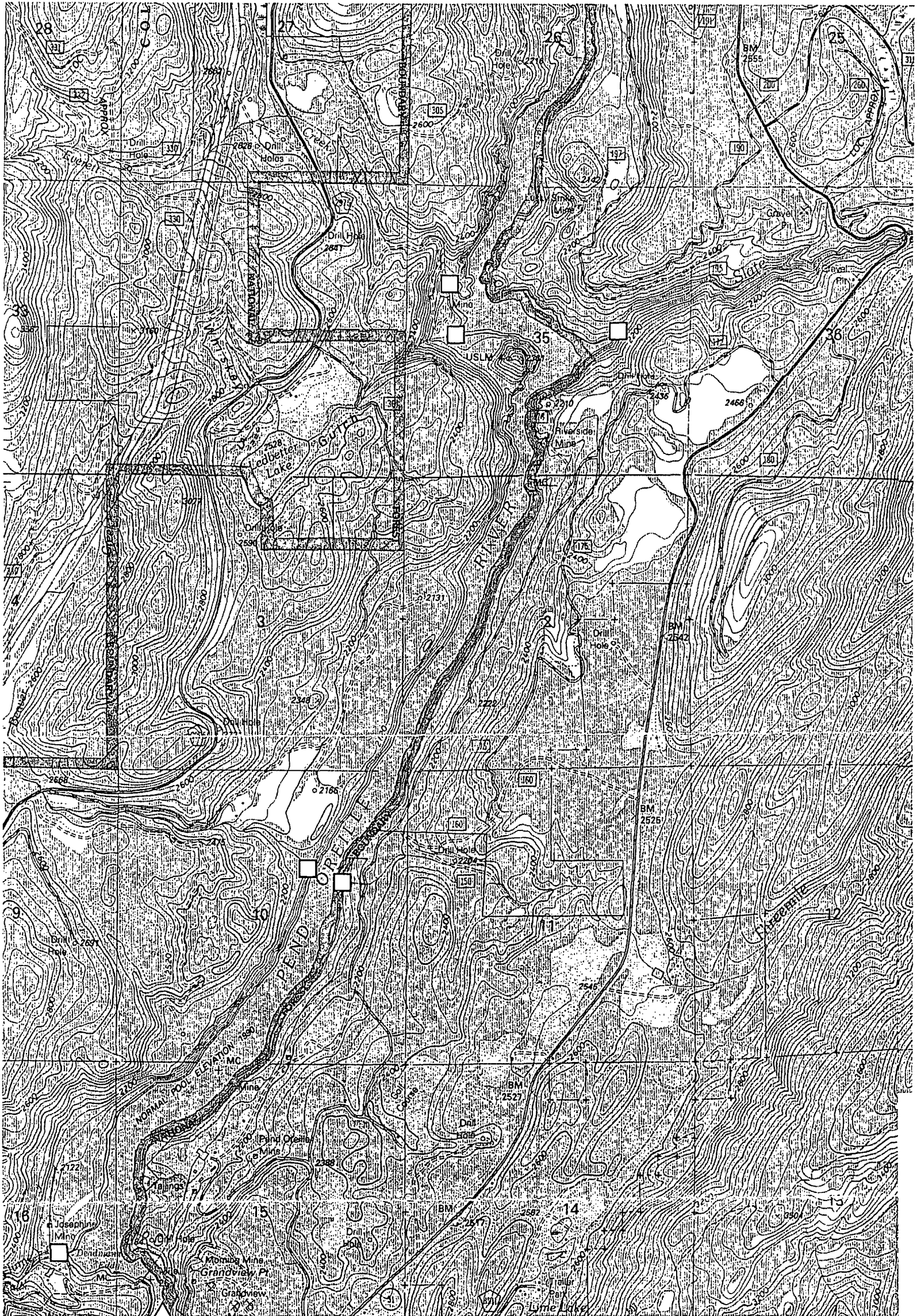
Source: USGS 7.5' Quadrangle of Boundary Dam, WA (1992)

LEGEND	
△	Proposed River/Lake Sediment Sample Location
□	Proposed Tributary Mouth Sediment Sample Location



Proposed River/Lake and Tributary Mouth Sediment Sample Locations Upper Columbia River/Lake Roosevelt SI





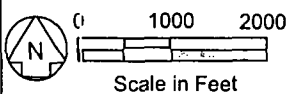
Notes:

- 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
- 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

Source: USGS 7.5' Quadrangle of Boundary Dam, WA (1992)

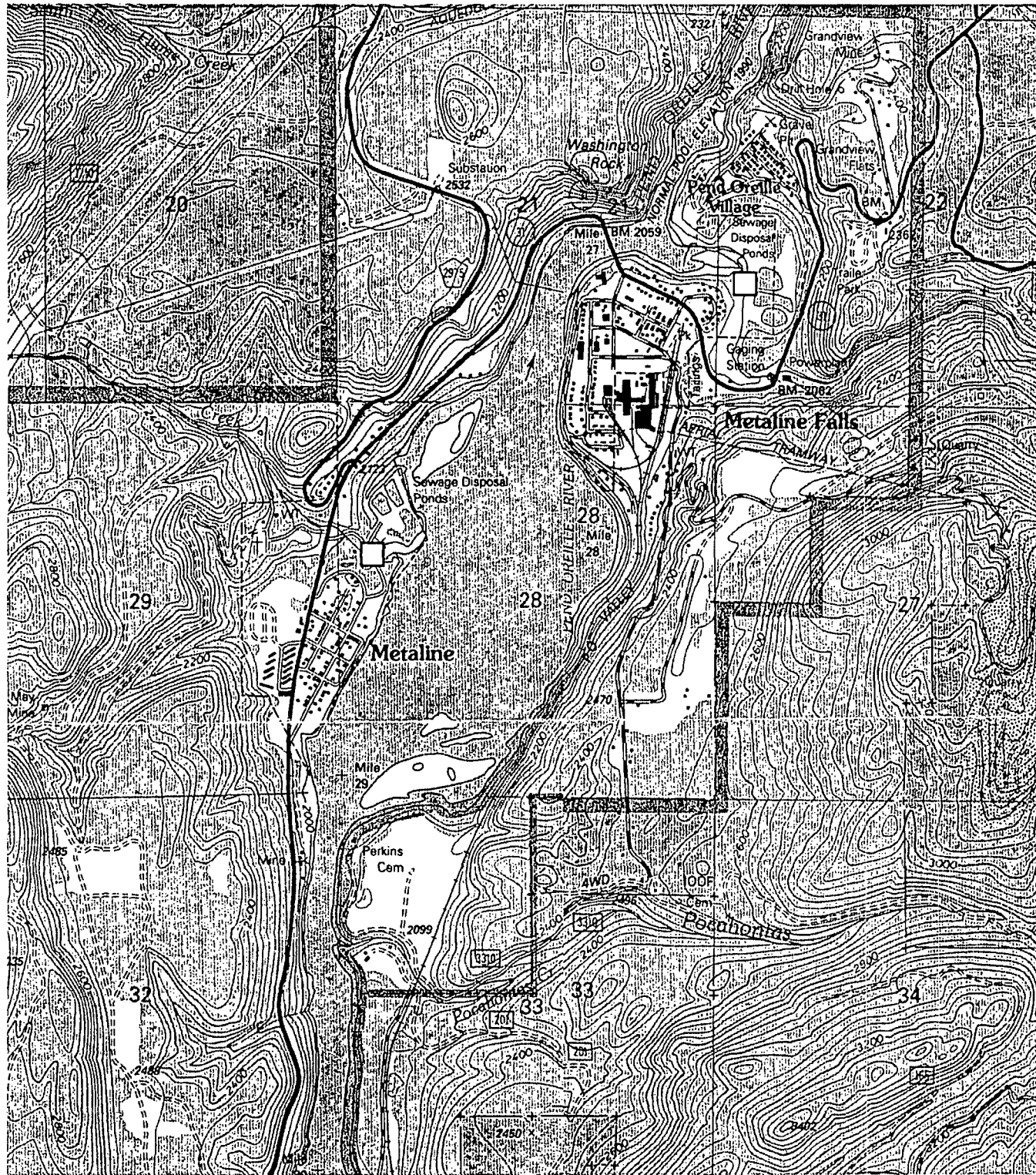
LEGEND

- △ Proposed River/Lake Sediment Sample Location
- Proposed Tributary Mouth Sediment Sample Location



**Proposed River/Lake and Tributary Mouth Sediment Sample Locations
Upper Columbia River/Lake Roosevelt SI**



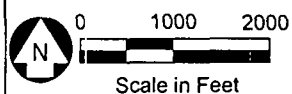


Notes:

- 1) The locations of background, attribution, and tributary upgradient samples will be determined during field effort.
- 2) Sample locations may be altered or added in the field at the direction of the EPA TM.

Source: USGS 7.5' Quadrangle of Metaline, WA and Metaline Falls, WA (1992)

LEGEND	
□	Proposed Tributary Mouth Sediment Sample Location



Proposed River/Lake and Tributary Mouth Sediment Sample Locations Upper Columbia River/Lake Roosevelt SI



**Table 2-1—Sample Information Summary
Upper Columbia River/Lake Roosevelt Expanded Site Investigation
Lake Roosevelt, Washington**

Project Sampling Schedule ^a	Parameter/Limits	Design Rationale	Sampling Design Assumptions	Sample Selection Procedures	Measurement Classification (Critical/Noncritical)	Nonstandard Method Validation
Soil/Sediment Samples	Cu, Pb, Zn by XRF/semi-quantitative	Screening, hot spot identification	Point of release/deposition	Screening for selection of lab samples	Noncritical	Follow Method/manufacturer's manual
Soil/Sediment Samples	TAL Metals/CRDL PCBs/CRQL TOC/0 1%	Gather data to determine HRS ranking	Contaminants were released to the soil	Samples will be collected from potentially contaminated areas* and/or stained soil	Critical	PCBs will be validated following laboratory- and method-specific criteria
Surface Water Sample	TAL Metals/CRDL PCBs/CRQL VOCs SVOCs Cl-Pesticides	Determine if contaminants have impacted surface water at municipal intake point	Contaminants impacted surface water at municipal intake point	One sample will be collected area adjacent to intake	Critical	PCBs will be validated following laboratory- and method-specific criteria

^a - All samples will be collected during the field event

* - As indicated from past investigations at the site and from on-site observations.

Key

CRDL = Contract Required Detection Limit

CRQL = Contract Required Quantitation Limit

Critical = Required to achieve project objectives or limits on decision errors

NA = Not Applicable

Noncritical = For informational purposes only or needed to provide background information.

**Table 2-2—Sample Analyses Summary
Upper Columbia River/Lake Roosevelt Expanded Site Inspection
Lake Roosevelt, Washington**

Matrix	Location	Analytical Parameters and Method	Sample Preservation	Technical Holding Time ^a	Sample Container(s)
Soil/ Sediment	Bed Sediment	TAL metals (CLPAS) ILMO4 1	Cool to 4°C ± 2°C	180 days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with polyethylene-lined lid
		Pesticides/PCBs (CLPAS) OLMO4 2	Cool to 4°C ± 2°C	Extract within 14 days of collection, analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		TOC EPA SW-846 9060M	Cool to 4°C ± 2°C	28 days from collection	One 8-oz wide-mouth glass jar with Teflon-lined lid
Water	Grand Coulee Municipal Drinking Water System	TAL metals (CLPAS) ILMO4 1	Cool to 4°C ± 2°C, HNO ₃ to pH ≤ 2	180 days from collection (28 days for mercury)	One 1-liter polyethylene bottle with polyethylene-lined lid
		Pesticides/PCBs (CLPAS) OLM04 2	Cool to 4°C ± 2°C	Extract within 7 days of collection, analyze within 40 days of extraction	Two 1-liter amber glass jars with Teflon- lined lids
		VOCs (CLPAS) OLM04 2	Cool to 4°C ± 2°C, HCl to pH ≤ 2	14 days from collection	Two 40-ml jars with Teflon-lined septa
		SVOCs (CLPAS) OLM04 2	Cool to 4°C ± 2°C	Extract within 7 days of collection, analyze within 40 days of extraction	Two 1-liter amber glass jars with Teflon- lined lids
QC Water Samples	Rinsate Blank	TAL metals (CLPAS) ILMO4 1	Cool to 4°C ± 2°C, HNO ₃ to pH ≤ 2	180 days from collection (28 days for mercury)	One 1-liter polyethylene bottle with polyethylene-lined lid
		Pesticides/PCBs (CLPAS) OLM04 2	Cool to 4°C ± 2°C	Extract within 7 days of collection, analyze within 40 days of extraction	Two 1-liter amber glass jars with Teflon- lined lids
		VOCs (CLPAS) OLM04 2	Cool to 4°C ± 2°C, HCl to pH ≤ 2	14 days from collection	Two 40-ml jars with Teflon-lined septa
		SVOCs (CLPAS) OLM04 2	Cool to 4°C ± 2°C	Extract within 7 days of collection, analyze within 40 days of extraction	Two 1-liter amber glass jars with Teflon- lined lids
	Trip Blanks	VOCs (CLPAS) OLM04 2	Cool to 4°C ± 2°C, HCl to pH ≤ 2	14 days from collection	Two 40-ml jars with Teflon-lined septa

^a Technical holding times have been established only for water matrices. Water technical holding times were applied to sediment, soil, and product samples where applicable, in some cases, recommended sediment/soil holding times are listed.

Key

C = Celsius

CLPAS = Contract Laboratory Program Analytical Service

HNO₃ = Nitric Acid

L = Liter

ml = Milliliter.

oz = Ounce

PCBs = Polychlorinated Biphenyls

TAL = Target Analyte List

TOC = Total Organic Carbon

VOCs = Volatile Organic Compounds.

**Table 2-3—QA/QC Analytical Summary and Fixed Laboratory Analytical Methods
Upper Columbia River/Lake Roosevelt Expanded Site Inspection
Lake Roosevelt, 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 ^e	Precision and Accuracy
					Organic MS/MSD ^b	Inorganic MS/MSD ^b	Rinsate Blanks ^c	Trip Blanks ^d		
Field Analysis	Soil/ Sediment	Pb, Cu, Zn by XRF EPA 6200	Per Method	NA	NA	NA	NA	NA	NA	Per Method
EPA Region 10 or CLP Laboratory	Soil/ Sediment	TAL Metals/ (CLPAS) ILM04 1	AA & ICP/ CRDL	186 / 186	NA	9 / 0	15 / 15	NA	210 / 201	75% - 125% +/- 35%
		Pesticides/PCBs (CLPAS) OLM04 2	GCS & ECD/ CRQL	149 / 149	8 / 0	NA	15 / 30	NA	164 / 179	OLM04 2/ OLM04 2
Commercial Laboratory	Soil/ Sediment	TOC EPA SW-846 9060M	1 mg/kg	149 / 149	8 / 0	NA	NA	NA	157 / 149	Per Method
EPA Region 10 or CLP Laboratory	Water	Pesticides/PCBs (CLPAS) OLM04 2	GCS & ECD/ CRQL	4 / 4	1 / 2	NA	2/4	NA	7 / 10	OLM04 2/ OLM04 2
		VOCs	GCS & MD/ CRQL	4/8	1 / 2	NA	2/4	2	7 / 16	OLM04 2/ OLM04 2
		SVOCs	GCS & MD/ CRQL	4/8	1 / 2	NA	2/4	NA	7 / 14	OLM04 2/ OLM04 2
		TAL Metals/ (CLPAS) ILM04 1	AA & ICP/ CRDL	4 / 4	NA	1 / 1	2/4	NA	7 / 9	75% - 125% +/- 35%

^a Total number of field samples is estimated

^b No extra volume is required for soil/sediment or product samples, for water samples, triple volume is required for organic analyses, and double volume is required for inorganic analyses
Sample numbers are based on 1 matrix spike/matrix spike duplicate (MS/MSD) per 20 samples per matrix

^c The total number of rinsate samples could vary depending on the total number of samples collected. 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

^d The total number of trip blanks could vary depending on the total number of sample shipments. This number is based on the estimated number of shipping containers. Note that trip blanks consist of water aliquots for both soil and water field samples

^e Total analyses and containers includes both field and QA/QC aliquots to be submitted for fixed laboratory analysis. Note that trip blanks and 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 Service

CRDL = Contract-Required Detection Limit

CRQL = Contract Required Quantitation Limit

ECD = Electron Capture Detection

GCS = Gas Chromatographic Separation

ICP = Inductively Coupled Plasma

MD = Mass Spectrometric Detection

MS/MSD = Matrix Spike/Matrix Spike Duplicate

NA = Not Applicable

PCBs = Polychlorinated Biphenyls

Pesticides = Chlorinated Pesticides

QA = Quality Assurance

QC = Quality Control

SVOCs = Semivolatile Organic Compounds

TAL = Target Analyte List

TOC = Total Organic Carbon

VOCs = Volatile Organic Compounds

Table 2-4—Sampling Plan Summary

Type	Location	Estimated # Samples	Rationale
Columbia River Sediment Samples	U S Upper Columbia River and Lake Roosevelt	57	Grab samples at depositional points Screen with XRF (sample count includes two auger sampling points)
	U S Upper Columbia River Tributaries	41	Samples in tributaries near mouths, including ephemeral tributaries No XRF screening
	Canadian Columbia River	2	Sample in river above confluence with Pend Oreille
	Canadian Columbia River Tributary	1	Stoney Creek
	Canadian Pend Oreille River	2	One sample upstream of Wanita Dam and one sample upstream of Seven Mile Dam
	Canadian Pend Oreille River Tributaries	3	Samples near mouths of Cedar Creek, Church Creek, and Salmo River
	Pend Oreille River to Metaline	2	One sample just upstream of Boundary Dam, one sample upstream of Pend Oreille Mine and downstream of Grand View Mine, one sample by Metaline
	U S Tributaries to the Pend Oreille River	10	Samples in tributaries near mouths
Upland Tributary Sediment Samples	U S Upper Columbia River Tributary Samples Upstream of Mines	11	11 Tributaries have mines
	Canadian Pend Oreille River Tributary Samples Upstream of Mines	3	Samples upstream in Cedar Creek, Church Creek, and Salmo River
	U S Pend Oreille River Samples Upstream of Mines	5	5 Tributaries have mines
Background Sediment Samples	Arrow Lake	8	Background samples at locations and with grain sizes corresponding to release samples
	Columbia River after Keenleyside	8	Background samples at locations and with grain sizes corresponding to release samples
	Kootenay River	8	Attribution
	Slocan River	8	Attribution
	Pend Oreille River Background samples above Metaline	8	Background samples at locations and with grain sizes corresponding to release samples
Surface Water Samples	Grand Coulee Municipal intake and system effluent sample	2	Sample lake water entering municipal system, and drinking water supplied to City
	Surface water background	1	Background sample in a similar setting as water intake sample
QC Samples	Sediment Field Duplicate Samples	9	One field duplicate per 20 samples per matrix and analyte
	Surface Water Field Duplicate Samples	1	One field duplicate per 20 samples per matrix and analyte
	Non-dedicated sediment sampling equipment nnsates	15	One per day of estimated use
	Non-dedicated surface water sampling equipment nnsates	2	One per day of use
	VOC Trp Blanks	2	One per cooler containing samples for VOC analysis

Total # Sediment Release/Background/Attribution Samples: 177
Total # Surface Water Release/Background Samples: 3
Total # Field QC Samples: 29
Total samples: 209

**Table 2-5—Sample Coding
Upper Columbia River/Lake Roosevelt SI**

Digits	Descriptions	Code Example
1,2	Station Code	CR (Upper Columbia River/Lake Roosevelt)
		TR (Upper Columbia River/Lake Roosevelt Tributary Mouth)
		PO (Pend Oreille River)
		UT (Tributary Upland)
		BK (Background)
		AT (Attribution)
		RS (Rinsate QA)
		TB (Trip Blank QA)
3, 4, 5	Consecutive Sample Number	001 (First Sample of Source Code)
6, 7	Matrix Code	SD (Sediment)
		SW (Surface Water)
		WT (Water)

SECTION 3

ASSESSMENT/OVERSIGHT

3.1 ASSESSMENTS AND RESPONSE ACTIONS

The EPA QA manager 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 site audit. The actions taken may also involve the EPA Project Officer, Contracting Officer, and/or QA Officer. Once the response actions have been implemented, the EPA QA manager 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 may be conducted in accordance with the WESTON START *Quality Management Plan*. No audits are planned for the Upper Columbia River/Lake Roosevelt ESI.

If major deviations from the QA requirements of the project and the CLP SOW were observed in the data validation process, the EPA QA Officer will contact the laboratory to correct the problem. If the laboratory will not be responsive to the request, the QA Officer will inform the CLP Regional Project Officer and the Task Monitor of the situation. A brief narrative will be written explaining the contract deviations and recommendations will be given based on the quality of the submitted data. Reduce payment and/or re-analysis at the laboratory's expense shall be pursued by the Regional CLP PO. Re-sampling and subsequent re-analysis will be decided by the TM.

Additional sampling for corrective actions and/or any addendum to this SQAP shall be documented using the Corrective Action Form and the Sample Alteration Form (Appendix B).

3.2 REPORTS TO MANAGEMENT

Debriefing of the EPA TM occurs by the START PM on a daily basis. Laboratory deliverables will be as specified in the CLP Organic and Inorganic Statements of Work (OLMO4.2 and ILMO4.1, respectively) for CLP and/or MEL data, as specified in the laboratory subcontract bid specification package for commercial laboratory analyses. Once the project is complete and the resulting data is obtained, the START Site Leader will assist in preparation of a sediment sampling summary report. The report will include a summary of the activities performed during the field effort and the resulting data (along with any statements concerning data quality). The report will be forwarded to E&E for inclusion in the final ESI report to be prepared by E&E.

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

SECTION 4

DATA VALIDATION AND USABILITY

4.1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

The data 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 chain-of-custody; and field instrument calibration, results, and/or performance check documentation (if required by the method). The QA review will also examine adherence to the procedures as described in the cited SOPs and the specified analytical methods in the SQAP.

4.1.1 Data Validation

Analytical data generated through the CLP contract will be validated by Region 10 QA Office or its designee, data generated by MEL will be validated by MEL or its designee and data generated through subcontract laboratories by WESTON will be validated by WESTON. The data validations will be performed on a regular two-week turn around time that 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:

- EPA Contract Laboratory Program (CLP) National Functional Guidelines for Inorganic Data Review (1994).
- EPA CLP National Functional Guidelines for Inorganic Data Review (1999).

Validation deliverables will include a QA memo discussing QA conformance and deviations issues that 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 memo. 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 the 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 values is an estimate 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.

4.2 DATA VERIFICATION

Data validation deliverables will be submitted to WESTON for verification. Using the QA memo, the Forms I with validation qualifiers and the electronic data deliverables (EDDs) from the laboratories, WESTON will ensure that consistency exists among three documents. WESTON 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 will be used as a tool to determine whether these targets were met. Also, using the compiled data, WESTON and TM will determine variability and soundness of the data and the data gaps that will need to be filled to meet the objectives of the project. Preliminary ranking of the site will also be determined if additional sampling is needed for the site.

SECTION 5

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APPENDIX A
STANDARD OPERATING PROCEDURES

SOP No.: RFW/R10-003

Document Control Number: 12644-001-002-AABH

**STANDARD OPERATING PROCEDURE
FOR
SEDIMENT SAMPLING**

Prepared by:

Roy F. Weston, Inc. (WESTON®)
Region X
Superfund Technical Assessment and Response Team (START)
190 Queen Anne Avenue North, Suite 200
Seattle, WA 98109

April 2001

EPA Contract No. 68-S0-01-02

APPROVALS



Prepared by:	_____	Date:	_____
	Environmental Scientist		
Reviewed by:	 _____	Date:	<u>05/09/01</u>
	Site Assessment Leader		
Approved by:	 _____	Date:	<u>05/09/01</u>
	Quality Assurance Officer		

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1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures used by Region X Roy F. Weston, Inc. (WESTON®), Superfund Technical Assessment and Response Team (START) personnel in collecting representative sediment samples. Purposes for the collection of sediment samples by START personnel and their subsequent analysis will be detailed in site-specific Sampling and Quality Assurance Plans (SQAPs), and may include determining whether concentrations of hazardous substances in sediment samples exceed established action levels, confirming or identifying hazardous substances that may have impacted the environment, determining if contaminants are migrating off site, or if the concentrations of hazardous substances may present a risk to public health, welfare, or the environment.

The methodologies discussed in this procedure are applicable to the sampling of sediment in both flowing and standing water. They are generic in nature and may be modified in whole or part to meet the handling and analytical requirements of the contaminants of concern, as well as the constraints presented by the sampling area. However, if modifications occur, they shall be documented in the site logbook or report summarizing field activities.

For the purposes of this procedure, sediments are defined as detrital material located in either static (as in lakes, ponds, wetlands, or other impoundments) or flowing (streams) water bodies. Selection of an appropriate sediment sampling technique is contingent upon the depth of water at the sampling location and the physical characteristics of the sediment to be sampled.

2.0 Summary of Method

Sediment samples may be collected using a variety of methods and equipment depending on the depth of the water body layer, the portion of the sediment profile required (surface versus subsurface), the type of sample required (disturbed versus undisturbed), and the sediment type. If surface water samples are to be collected at the same sampling location as the sediment sample, the surface water sample is to be collected first. START has prepared a separate SOP, entitled *Standard Operating Procedure For Surface Water Sampling*. This SOP should be reviewed prior to collecting surface water samples. Sediments collected from a flowing water body (such as stream) should be collected beginning at the most downstream sampling location, and progressing to the most upstream sampling location. This procedure is followed to collect a representative sediment sample at the sampling location and to avoid sediment cross-contamination caused when sediments are disturbed during the sampling process.

Sediment is collected from a water body either directly using a hand-held device (such as a shovel, trowel, or auger) or indirectly using a remotely activated device (such as an Ekman or Ponar dredge). Following collection, the sediment sample is placed into a decontaminated container such as a stainless steel bowl, homogenized, and placed into appropriate sample containers. Sediment samples should be homogenized only after the portion for volatile organic compound (VOC) analysis has been collected, in order to avoid the loss of VOC contaminants during the homogenization process.

3.0 Acronym List

cc	-	Cubic centimeters
CLP	-	Contract Laboratory Program
COC	-	Chain-of-Custody
DAS	-	Delivery of Analytical Services
DOT	-	Department of Transportation
DQOs	-	Data Quality Objectives
EPA	-	U.S. Environmental Protection Agency
GPS	-	Global Positioning System
HASP	-	Health and Safety Plan
IATA	-	International Air Transport Association
MS/MSD	-	Matrix Spike/Matrix Spike Duplicate
OSHA	-	Occupational Safety and Health Administration
PE	-	Performance Evaluation
pest/PCB	-	Pesticide/Polychlorinated Biphenyl
PFO	-	Personal Flotation Device
PPE	-	Personal Protective Equipment
ppm	-	Parts per million
PRP	-	Potentially Responsible Party
QAPP	-	Quality Assurance Project Plan
QA/QC	-	Quality Assurance/Quality Control
RFP	-	Request for Proposal
SDG	-	Sample Delivery Group
SOP	-	Standard Operating Procedure
SQAP	-	Sampling and Quality Assurance Plan
START	-	Superfund Technical Assessment and Response Team
SVOC	-	Semivolatile Organic Compound
TDD	-	Technical Direction Document
TWP	-	Task Work Plan
VOA	-	Volatile Organic Analysis
VOC	-	Volatile Organic Compound
WESTON	-	Roy F. Weston, Inc.

4.0 Health and Safety Warnings

START personnel performing work on hazardous waste sites will follow Occupational Safety and Health Administration (OSHA), U.S. Environmental Protection Agency (EPA), and WESTON/START specific health and safety procedures and protocols. START personnel conducting on-site and off-site sediment sampling activities will also be performing tasks in accordance with EPA-approved SQAPs, and Operating Practices prepared and approved by START.

In order to ensure the safety of personnel during sampling activities, the buddy system, periodic air monitoring, and caution will be used throughout field activities. To minimize risks due to chemical

exposure, dermal and respiratory protection may be required if air monitoring equipment indicates that the environment is unsafe. Field activities will follow the site health and safety plan (HASP), which further addresses the safety considerations of the property. Hazards identified in or around the site may include physical hazards (slips, trips, and falls). Additional potential physical hazards exist in association with sediment sampling, and may include drowning or exposure due to hypothermia.

When collecting sediment samples from water bodies containing known or suspected hazardous substances, adequate precautions must be taken to ensure the sampler's safety. The START team member collecting the sample should not get too close to the edge of the water where bank failure may cause him or her to lose their balance. The use of personal protective equipment (PPE) and engineering controls, such as safety nets, shall be specified in the site HASP, and shall be based on site-specific conditions. Appropriate PPE may include lifelines or personal flotation devices (PFDs). If sampling from a vessel is required, appropriate protective measures may be required, and shall be detailed in the site HASP.

For any field assignments involving the collection of subsurface samples, excavation, or any other type of intrusive activities, it is a legal requirement to call the appropriate utility clearance center before beginning any intrusive activities on site. The Utilities Underground Location Center telephone number for Washington and Oregon is 1-800-424-5555; the frequent user ID# is 9772. The Utilities Underground Location Center requires two (2) business days advance notification for activities in Washington, and 48 hours advance notification for activities in Oregon. The Alaska Locate Call Center phone number is 1-800-478-3121; advance notification time requirements in Alaska vary with the location. Idaho public utilities require two (2) business day advance notification and are covered by four different numbers as follows:

- Bonner, Boundary, Latah, and Clearwater counties - Utilities Underground Location Center: 1-800-424-5555
- Kootenai County - Password Inc.: 1-800-428-4950
- Shoshone and Benewah counties: 1-800-398-3285
- Nez Perce and Idaho counties, and all other counties to the south - Dig Line Utility Protection Service of Idaho: 1-800-478-3121

The samples collected at the site will be shipped to predesignated Delivery of Analytical Services (DAS) laboratories according to either Department of Transportation (DOT) Hazardous Materials Regulations or International Air Transport Association (IATA) Dangerous Goods Regulations, or hand-delivered to the laboratories. Samples will be transported in a manner that will maintain their integrity, as well as protect against detrimental effects from sample breakage or leakage. The Roy F. Weston, Inc. *Manual of Procedures for Shipping and Transporting Dangerous Goods* will be followed whenever samples are shipped.

Decontamination wastes will be collected and secured on site. Separate containers will be used for the aqueous wastes and for flammable, non-chlorinated solvents (methanol and hexane) wastes. Proper personal protection will be worn during decontamination procedures and will include gloves, eye protection, and splash-resistant protective clothing. Off-site disposal of decontamination wastes, purge water, and contaminated personal protective equipment (PPE) will be conducted through the Subcontract Agreement established by Region X START for disposal of investigation-derived wastes. Non-contaminated wastes will be tightly sealed, double-bagged, and disposed of appropriately.

5.0 Interferences

Sediment particle size and organic content are directly related to water velocity and flow characteristics of a body of water. Contaminants are more likely to be concentrated in fine-grained (silts and clays) sediments having a high organic content. In contrast, coarse-grained (sand, pebbles, and cobbles) sediments with low organic content do not typically concentrate pollutants. Therefore, the selection of a sampling location can greatly influence the analytical results.

Some interferences or potential problems associated with sediment 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. If this is not possible or practical, then decontamination of non-dedicated sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, inadequate homogenization of the samples, and improper sampling technique for VOC samples, which may result in variable, non-representative results.

For sample matrices such as sediments containing high percent moisture, START routinely provides the analytical laboratory with additional sample volume in order to meet EPA Region X QA analytical guidance that addresses the representativeness of sediment sample matrices. Laboratories must analyze, extract, digest, or distill an increased aliquot of sediment when the sample's percent moisture is equal to or less than 30% solid. Since percent solids are not known until after samples are received by the laboratory, START routinely collects larger sample volumes for each analytical parameter for sediment samples than might be collected for a low-moisture soil sample.

6.0 Personnel Qualifications

Training of START members will be provided to ensure that technical, operational, and quality requirements are understood. START personnel are trained in-house to conduct sediment sampling activities. Training includes reviewing this SOP and other applicable SOPs and/or guidance documents, health and safety training, "hands-on" experience conducting sampling activities with more experienced START personnel, and with instrument calibrations.

7.0 Apparatus and Materials

Equipment/materials for collecting sediment samples includes, but is not limited to the following:

- Site-specific SQAP.
- Standard Operating Procedure for Sediment Sampling.
- Safety equipment specified in the site Health and Safety Plan.
- Field map of site and/or plot plan.
- Logbook.
- Field data sheets (e.g. soil description sheets, etc.).
- Graph paper.
- Tape measure.
- Compass.
- Survey stakes, flags (white), or buoys and anchors.
- Camera and film.
- Continuous flight (screw) auger.
- Continuous flight auger extension rods.
- Spatula, scoops, or trowel.
- Bucket auger.
- T-handle.
- Post hole auger.
- Stainless steel homogenization bucket or bowl.
- Spade or shovel.
- Thin-walled auger.
- Ponar dredge.
- Ekman dredge.

- Sediment coring device (tubes, points, drive head, drop hammer, “eggshell” check valve devices, acetate cores).
- Certified clean sampling containers.
- Conductivity, turbidity, and temperature meter.
- pH paper.
- Decontamination fluids (supplies)/equipment (pump sprayers, brushes, etc.).
- Plastic sheeting (Visqueen).
- Plastic tubs.
- Sealable plastic bags.
- Labels.
- Chain-of-custody forms and seals.
- Coolers and ice.
- Vermiculite.
- Strapping tape.
- Nylon rope.

7.1 Reagents

Reagents are not typically used for the preservation of sediment samples. Preservation solutions for rinsate blanks may include: nitric acid, sodium hydroxide, and hydrochloric acid.

8.0 Method Calibration

This section is not applicable to this SOP.

9.0 Sample Collection

Under the Removal Program, sediment samples may be collected to determine if any hazardous substances are present on site at such concentrations that a removal action is warranted; to determine the extent of contamination; or to ensure that clean up levels are achieved during removal actions.

Under the Site Assessment Program, sediment samples are generally collected to determine if concentrations of hazardous substances document an observed release to the surface water pathway and can be used to document actual contamination to drinking water supplies (surface water intakes), fisheries (food chain), and/or sensitive environments (wetlands, clean water act-protected bodies, endangered and/or threatened species, etc.) under the Hazard Ranking System.

9.1 Presampling Preparation

Following EPA approval of the site-specific QAPPs, and prior to conducting sampling activities, a pre-sampling meeting will be held by START team members to discuss the proposed sampling strategy and site health and safety issues. Attendees of the pre-sampling meeting will include the Project Manager, Site Leader, samplers, and the START Health and Safety Officer. During the pre-sampling meeting, the Site Leader will discuss the site history, contaminants of concern, sampling methodology, individual responsibilities, sample shipment or delivery, health and safety issues, and lines of communication anticipated during the sampling event.

Prior to mobilizing to the site to conduct sampling activities, the START Site Leader will fill out an equipment/supply list and transmit the list to the WESTON Equipment Stores technician one week prior to the sampling event. Necessary sampling equipment, sample containers, PPE, and vehicles are therefore reserved. Analytical services are procured for sediment samples through subcontract DAS laboratories at least one week prior to the sampling event by START analytical staff members under the supervision of the START Lead Chemist.

9.2 Sediment Sample Collection

Prior to conducting any on-site activities, START members review and sign the site-specific HASP. The START field team will establish a command post upwind of suspected source areas, if possible. START members will perform calibration checks of air monitoring instruments and document background ambient air monitoring levels. The samplers will decontaminate the sampling equipment. Decontamination will be conducted in accordance with the HASP, applicable SOPs, and/or the Request for Proposal(s) (RFPs) for drilling or excavation services. Decontamination generally consists of an alconox and water wash followed by a distilled water rinse, followed by a de-ionized water final rinse, and air drying. Additionally, where high concentrations of specific substances are anticipated, chemicals such as isopropanol, methanol, hexane, and/or nitric acid may be used in the decontamination process. Equipment decontamination fluids and PPE generated during sampling activities will be containerized and disposed appropriately based on the results of laboratory analyses of samples collected.

9.2.1 Sample Collection Procedures for Scoops or Trowels

Collection of surface sediment from beneath shallow water can be accomplished with tools such as scoops or trowels. Surface material can be removed to the required depth; then these tools can be used to collect the sample.

This method can be used to collect consolidated sediments but is limited somewhat by the depth of the water. Accurate, representative sediment samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A stainless steel or plastic scoop, or trowel will suffice in most applications. Care should be exercised to avoid the use of devices plated with chrome or other materials. Plating is particularly common with garden trowels. The following section discusses collecting sediment samples for typical analytical parameters under the START Program using scoops or trowels.

VOC Sampling - The proper collection of a sample for VOC analysis requires minimal disturbance of the sample to minimize loss of volatile compounds from the sample. Sediment VOC samples will be collected first using a dedicated sampling spatula and placed directly into the appropriate size, prelabelled glass sample container. The samples will then be preserved by placing the sample container in a cooler with ice.

To increase the data reliability and reproducibility, it is desirable to manually homogenize the sediment sample. After the VOC fraction of sediment is collected, the remaining sediment sample will be placed into a stainless-steel bowl or other appropriate homogenization container, and mixed thoroughly to obtain a homogenous sample representative of the entire sampling interval. A dedicated sampling spatula will be used to collect the sample material and place it into the appropriate sample container (preferably a wide-mouth container). Dedicated spatulas are used to prevent the possibility of cross-contamination between sample stations as well as eliminating the need for any decontamination procedures. The container cap will then be secured and the sample will be preserved by immediately placing containers in a cooler with ice.

In cases where split samples will be collected, an adequate amount of sample volume must be homogenized to account for the extra samples. Split samples will be used in situations where confirmation samples will be submitted to an analytical laboratory to verify on-site screening results, or an aliquot is requested by the potentially responsible party (PRP). The following section discusses collecting sediment samples for typical parameters following homogenation of the sample.

Semivolatile Organic Compound (SVOC) Sampling - Sediment samples will be collected using a dedicated sampling spatula as described above and placed into the

appropriate size, prelabelled glass sample container. The samples will then be preserved by placing the sample container in a cooler with ice.

Pesticide/Polychlorinated Biphenyl (Pest/PCB) Sampling - Sediment samples will be collected using a dedicated sampling spatula as described above and placed into the appropriate size, prelabelled glass sample container. The samples will then be preserved by placing the sample container in a cooler with ice.

Metals Sampling - Sediment samples will be collected using a dedicated sampling spatula as described above and placed into the appropriate size, prelabelled sample container. The samples will then be preserved by placing the sample container in a cooler with ice.

Cyanide Sampling - Sediment samples will be collected using a dedicated sampling spatula as described above and placed into the appropriate size, prelabelled sample container. The samples will then be preserved by placing the sample container in a cooler with ice.

9.2.2 Sample Collection Procedures for Auger and Thin-wall Sampling

This system consists of an auger, a series of extensions, a "T" handle, and a thin-wall tube sampler. The auger is used to bore a hole to a desired sampling depth, and is then withdrawn. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin-wall tube sampler. The system is then lowered down the borehole, and driven into the sediment to the completion depth. The auger is then withdrawn and the core collected from the thin-wall tube sampler. This method can be used to collect consolidated sediments, but is somewhat limited by the depth of the water.

Several types of augers are available. These include: bucket, continuous flight (screw), and posthole augers. Bucket augers are better for direct sample recovery since they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights, which are usually at 5-foot intervals. Posthole augers have limited utility for sample collection, as they are designed more for their ability to cut through fibrous, rooted, swampy areas. The following procedure describes how to collect a sediment sample using an auger:

- Attach the auger bit to a drill extension rod, then attach the "T" handle to the drill extension rod.
- Clear the area to be sampled of any surface debris.

- Insert the assembled auger into the sediment and begin augering, periodically removing accumulated sediment from the auger bucket.
- After reaching the desired depth, the auger is slowly and carefully removed from the boring. When sampling directly from the auger, collect sample after the auger is removed from boring. Once removed from the bore hole, the top inch of the sample core is removed and discarded. Samples for VOC analysis will be collected first following the procedures described in Section 9.2.1 - Sample Collection Procedures for Scoops or Trowels. The remaining sample will then be placed into a stainless steel bowl (or disposable pan) and homogenized using a stainless steel (or disposable) scoop. Any extraneous material not considered to be relevant for analysis is removed from the sample during homogenization and any water present will be decanted. The sample is then placed into the appropriate size, prelabelled sample containers (preferably wide-mouthed) for each analysis (typically SVOC, pest/PCBs, metals, and cyanide) to be performed. The container cap(s) will then be secured and the samples will be preserved by immediately placing the containers in a cooler on ice.

The following procedure describes how to collect a sediment sample using an auger with a thin-wall tube sampler.

- After reaching the desired depth, the auger is slowly and carefully removed the from the boring.
- The auger tip is removed from the drill rod and replaced with a pre-cleaned thin-wall tube sampler and the proper cutting tip is installed.
- The tube sampler is carefully lowered down the borehole, and the tube sampler is gradually forced into the sediment. Care should be taken to avoid scraping the borehole sides.
- The auger is slowly and carefully removed from the boring and the thin-wall tube sampler is removed.
- The top of the core is discarded (approximately 1 inch), as this represents material collected by the tube sampler before penetration of the layer of concern.
- Samples for VOC analysis will be collected first following the procedures described in Section 9.2.1 - Sample Collection Procedures for Scoops or Trowels. The remaining sample will then be placed into a stainless steel bowl (or disposable pan) and homogenized using a stainless steel (or disposable) scoop. Any extraneous material not considered to be relevant for

analysis is removed from the sample during homogenization and any water present will be decanted. The sample is then placed into the appropriate size, prelabelled sample containers (preferably wide-mouthed) for each analysis (typically SVOC, pest/PCBs, metals, and cyanide) to be performed. The container cap(s) will then be secured and the samples will be preserved by immediately placing the containers in a cooler on ice.

9.2.3 Sample Collection Procedures With an Ekman Dredge

This technique consists of lowering a sampling device to the sediment by use of a rope, cable, or extended handle. The mechanism is triggered, and the device entraps sediment in spring-loaded jaws, or within lever-operated jaws. The following procedures are used for collecting sediment samples with an Ekman dredge:

- Thread a sturdy nylon or stainless steel cable through the bracket, or secure the extended handle to the bracket with machine bolts.
- Attach springs to both sides. Arrange the Ekman dredge sampler so that the jaws are in the open position and the trip cables are positioned over the release studs.
- Lower the sampler to a point just above the sediment surface.
- Drop the sampler sharply onto the sediment.
- Trigger the jaw release mechanism by lowering a messenger down the line, or by depressing the button on the upper end of the extended handle.
- Raise the sampler and slowly decant any free liquid through the top of the sampler. Be careful to retain fine sediments.
- Open the dredge and transfer the sediment into a stainless steel or plastic bucket. Continue to collect additional sediment until sufficient material has been secured.

Collect samples for VOC analysis first following the procedures described in Section 9.2.1 - Sample Collection Procedures for Scoops or Trowels. The remaining sample will then be placed into a stainless steel bowl (or disposable pan) and homogenized using a stainless steel (or disposable) scoop. Any extraneous material not considered to be relevant for analysis is removed from the sample during homogenization and any water present will be decanted. The sample is then placed into the appropriate size, prelabelled sample containers (preferably wide-mouthed) for each analysis (typically SVOC, pest/PCB, metals, and cyanide) to be performed. The container

cap(s) will then be secured and the samples will be preserved by immediately placing the containers in a cooler on ice.

9.2.4 Sample Collection Procedures With a Ponar Dredge

This technique consists of lowering a sampling device to the sediment by use of a rope, cable, or extended handle. A mechanism is triggered, and the device entraps sediment in spring-loaded jaws, or within lever-operated jaws. The following procedures are used for collecting sediment samples with a Ponar dredge.

- Attach a sturdy nylon or steel cable to the hook provided on top of the dredge.
- Arrange the Ponar dredge sampler in the open position, setting the trip bar so the sampler remains open when lifted from the top.
- Slowly lower the sampler to a point just above the sediment.
- Drop the sampler sharply into the sediment, then pull sharply up on the line, thus releasing the trip bar and closing the dredge.
- Raise the sampler to the surface and slowly decant any free liquid through the screens on top of the dredge. Be careful to retain fine sediments.
- Open the dredge and transfer the sediment to a stainless steel or plastic bucket. Continue to collect additional sediment until sufficient material has been gained.

Collect samples for VOC analysis first following the procedures described in Section 9.2.1 - Sample Collection Procedures for Scoops or Trowels. The remaining sample will then be placed into a stainless steel bowl (or disposable pan) and homogenized using a stainless steel (or disposable) scoop. Any extraneous material not considered to be relevant for analysis is removed from the sample during homogenization and any water present will be decanted. The sample is then placed into the appropriate size, prelabelled sample containers (preferably wide-mouthed) for each analysis (typically SVOC, pest/PCB, metals, and cyanide) to be performed. The container cap(s) will then be secured and the samples will be preserved by immediately placing the containers in a cooler on ice.

9.2.5 Sample Collection Procedures With a Sample Coring Device

The sample coring device consists of a coring device, handle, and acetate core. The following procedures are used for collecting sediment samples with a sample coring device.

- Assemble the coring device by inserting the acetate core into the sampling tube.
- Insert the "eggshell" check valve mechanisms into the tip of the sampling tube with the convex surface positioned inside the acetate core.
- Screw the handle onto the upper end of the sampling tube and add extension rods as needed.
- Place the sampler in a perpendicular position on the material to be sampled.
- This sampler may be used with either a drive hammer for firm consolidated sediments, or a "T" handle for soft sediments. If the "T" handle is used, place downward pressure on the device until the desired depth is reached. Rotate the sampler to shear off the core of the bottom and retrieve the device.
- Slide the acetate core out of the sampler tube. The acetate core may be capped at both ends. The sample may be used in this fashion or samples may be collected for various analytical parameters described in Section 9.2.1 - Sample Collection Procedures for Scoops or Trowels.

Collect samples for VOC analysis first following the procedures described in Section 9.2.1. The remaining sample will then be placed into a stainless steel bowl (or disposable pan) and homogenized using a stainless steel (or disposable) scoop. Any extraneous material not considered to be relevant for analysis is removed from the sample during homogenization and any water present will be decanted. The sample is then placed into the appropriate size, prelabelled sample containers (preferably wide-mouthed) for each analysis (typically SVOC, pest/PCB, metals, and cyanide) to be performed. The container cap(s) will then be secured and the samples will be preserved by immediately placing the containers in a cooler on ice.

If the drive hammer is selected, use the following procedures for collecting a sediment sample.

- Insert the tapered handle (drive head) of the drive hammer through the drive head.
- With left hand holding the tube, drive the sampler into the material to the desired depth. Do not drive the tube further than the tip of the hammer's guide.
- Record the length of the tube that penetrated the sample material, and the number of blows required to obtain this depth.

- Remove the drive hammer and fit the keyhole-like opening on the flat side of the hammer onto the drive head. In this position, the hammer serves as a handle for the sampler.
- Rotate the sampler at least two revolutions to shear off the sample at the bottom.
- Lower the sampler handle (hammer) until it just clears the two ear-like protrusions on the drive head, and rotate about 90°.
- Withdraw the sampler by pulling the handle (hammer) upwards and dislodging the hammer from the sampler.
- Slide the acetate core out of the sampler tube. The acetate core may be capped at both ends. The sample may be collected in this fashion or samples may be collected for various analytical parameters described in Section 9.2.1 - Sample Collection Procedures for Scoops or Trowels.

Collect samples for VOC analysis first following the procedures described in Section 9.2.1. The remaining sample will then be placed into a stainless steel bowl (or disposable pan) and homogenized using a stainless steel (or disposable) scoop. Any extraneous material not considered to be relevant for analysis is removed from the sample during homogenization and any water present will be decanted. The sample is then placed into the appropriate size, prelabelled sample containers (preferably wide-mouthed) for each analysis (typically SVOC, pest/PCB, metals, and cyanide) to be performed. The container cap(s) will then be secured and the samples will be preserved by immediately placing the containers in a cooler on ice.

10.0 Handling and Preservation

Samples are shipped to analytical laboratories expeditiously to ensure that holding times are not exceeded. A cooler temperature blank, consisting of a small container of tap water, is included in each cooler to monitor the temperature of samples upon receipt at the laboratory. Sediment samples are considered to be properly preserved if they are maintained within the 2 to 6 degree Celcius temperature range. Microbial activity is minimized at these low temperatures, thereby preventing bio-degradation of organic contaminants in the sediment matrix.

Trip blanks are used to assess the degree of contamination introduced into samples during sample handling, shipment, and analysis. Contamination may also be introduced from the use of poorly cleaned non-dedicated sampling equipment, chemical preservatives, sample bottles, or from improper shipping and handling. Samples are shipped to analytical laboratories expeditiously to ensure that holding times are not exceeded. One pair of trip blanks is included in each cooler containing VOC samples.

Chemicals that may be used during the decontamination process may include Alconox detergent, nitric acid, hexane, and isopropanol. Chemical preservatives used during sediment sampling activities are generally limited to those preservatives which are required for aqueous equipment rinsate blanks and trip blanks. Chemical preservatives commonly used to preserve equipment rinsate blanks include hydrochloric acid, nitric acid, and sodium hydroxide. START routinely purchases high-quality certified-clean sampling containers to prevent contamination from sample bottles. Sample jars are also sealed in plastic bags, and carefully placed in coolers lined with vermiculite packing material and ice packs to prevent cross-contamination due to breakage of samples during shipment.

11.0 Sample Preparation and Analysis

This section is not applicable to this SOP.

12.0 Troubleshooting

All field screening 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/operation, and they must be documented.

13.0 Data Acquisition, Calculations, and Data Reduction

13.1 Computer Hardware and Software

Sediment sample locations may be located using a Trimble Pro XRS or GeoExplorer Global Positioning System (GPS) receiver. Prior to conducting field work, Trimble Pathfinder Office 2.51 (Pathfinder software) may be used to generate a data dictionary to be used during collection of sample locations. The data dictionary is then transferred from the computer to the GPS datalogger. Upon returning from the field, GPS data are downloaded from the GPS datalogger to a computer using the Pathfinder software. GPS data undergo differential correction, with base station data obtained from a variety of community base stations depending upon geographic location to improve location accuracy. Pathfinder is used to export GPS data in a variety of formats [i.e. ArcView shapefile, ARC/INFO, dBASE, and AutoCAD export (DXF) files]. Typically, GPS data are exported into dBASE files, and then manipulated withing Microsoft Excel 2000 software. Data are then manipulated where they can be utilized in site diagrams to display sample locations.

13.2 Data Management and Records Management

Field observations made during the sampling event will be recorded in a site logbook and/or field data sheets, including description of sampling locations and any deviations from the site-specific QAPPs. Chain-of-custody will be maintained until samples are relinquished to a courier or to the laboratories assigned to perform the analyses. Photographs will be taken to document site conditions. The location and direction from which photographs are taken

will be noted in the field logbook, in accordance with the scope of work. Reports, site file memoranda, figures, tables, boring logs, etc. will be saved in site-specific Technical Direction Document (TDD) directories.

14.0 Quality Control and Quality Assurance Section

This section describes quality assurance/quality control (QA/QC) pertinent to sediment samples, and the types and uses of the QA/QC samples that are collected in the field. QA/QC samples are analyzed to provide information on the variability and usability of environmental sample results. They assist in identifying the origin of analytical discrepancies to help determine how the analytical results should be used. They are used mostly to validate analytical results.

A data quality review of the laboratory sample analyses will be conducted by U.S. EPA.

At a minimum, a Tier I data validation will be conducted, which involves determining data completeness through a data package inventory check. A Tier II data validation is generally completed for Site Assessment projects, and may be done at the specific request of the EPA On-Scene Coordinator for Removal projects. Tier II data validation includes completing a data package inventory, and qualifying the data based on deficiencies in QC sample results. Tier III data validation includes all the elements of a Tier II data validation, plus a check of calculations and evaluation of sample results through an evaluation of the raw data. Deliverables resulting from the data validation process include a data validation memorandum and data tables. Qualifiers are applied to sample results for data packages which go through a Tier II or Tier III data validation, and provide the data user with a sense of the usability and limitations of the data.

Field duplicates (replicates), matrix spike/matrix spike duplicate (MS/MSD), trip blanks, rinsate (equipment) blanks, and temperature blanks are discussed below. QA/QC results may suggest the need for modifying sample collection, preparation, handling, or analytical procedures if the resultant data do not meet quality assurance objectives.

14.1 Field Duplicates

Field duplicates are used to assess the degree of sample heterogeneity and the reproducibility of the sample collection procedure and the laboratory analysis. Field duplicates are typically collected for sediment samples which are submitted for field screening analysis or for site assessment scoring activities. One field duplicate is typically collected for each matrix type and sample parameter for every 20 stations for Site Assessment projects. The field duplicate is assigned an individual sample number.

14.2 Laboratory Matrix Spike, Matrix Spike Duplicate

Matrix spike (MS) and matrix spike duplicate (MSD) samples are used to monitor laboratory performance and determine how effectively the analytical method is able to recover target compounds that are spiked into the matrix of interest. MS/MSD samples are spiked by the

analyst with a known concentration of a target analytes to monitor recovery of the target compounds. It is necessary to provide extra volume of sample to the laboratory for spiking analyses. Generally, organic parameters (e.g., VOC, SVOC, and Pesticide/PCBs) require triplicate sample volume for MS/MSD analysis. Inorganic analyses typically require MS and laboratory duplicate (Dupl) analyses. Laboratory duplicate precision monitors the laboratory's ability to reproduce its results. Inorganic parameters (e.g., metals and cyanide) require duplicate sample volume. The required frequency for MS/MSD and MS/Dupl analysis is generally one per every 20 samples of each matrix for each requested analytical parameter.

For sediment samples, extra volume is collected, homogenized thoroughly, and sub-sampled by alternately scooping the homogenized sample into two or three separate bottles, as needed. The exception occurs when collecting additional sample volume for VOC MS/MSD analysis, which are collected prior to homogenization. The MS/MSD samples are assigned the same sample number.

14.3 Trip Blanks

Trip blanks are used to assess the degree of contamination introduced into samples during sample handling, shipment, storage, and analysis. Contamination may be introduced from the sample bottle, the preservatives used, cross-contamination from highly contaminated samples, or from poor shipping and handling procedures experienced both in the field and in the laboratory. Trip blanks are typically collected for VOC analysis. Trip blanks prepared from organic-free water will be collected prior to the sampling event. Each bottle of the trip blank sample will be preserved with one drop of 1:1 HCl per 20 ml of sample to achieve a pH of less than 2. Trip blanks are handled, transported, and analyzed in the same manner as the other samples collected for that analysis. One set of trip blanks is collected for VOC analysis for each cooler in which VOC samples are shipped.

14.4 Rinsate (Equipment) Blanks

Dedicated or decontaminated sampling equipment will be used at each sediment sample location to minimize cross-contamination. Sampling equipment must be decontaminated prior to the start of sampling activities as well as between sample locations, unless the sampling activity is dedicated to one sample location. Rinsate blanks are used to assess contamination (typically, cross-contamination) brought about by improper decontamination procedures between sampling stations. Rinsate blanks are not required for dedicated, disposable sampling implements. Examples of equipment requiring decontamination and rinsate blanks include augers, shovels, Ponar and Ekman Dredge samplers, and stainless steel scoops and bowls.

Rinsate blanks are obtained by pouring analyte-free water over decontaminated sampling equipment to test for residual contamination. The water is collected into the appropriate sample containers which are handled (e.g., preserved), shipped, and analyzed for the same

set of analytical parameters as the samples that were collected that day. Where non-dedicated sampling equipment is used, rinsate blanks must be collected at the rate of one per day per 20 stations for each parameter for which sediment samples are collected.

14.5 Temperature Blanks

Temperature blanks provide information of the preservation (temperature) of the samples during shipment to the laboratories. Temperature blanks are obtained by pouring tap water into a 40-ml glass vial and placing one temperature blank per cooler of samples.

15.0 Reference Section

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STANDARD OPERATING PROCEDURE
FOR
SURFACE WATER SAMPLING

Prepared by:

Roy F. Weston, Inc. (WESTON®)
Region X
Superfund Technical Assessment and Response Team (START)
190 Queen Anne Avenue North, Suite 200
Seattle, WA 98109

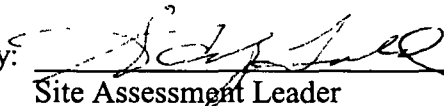
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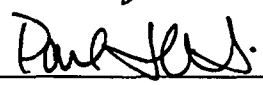
APPROVALS

Prepared by: _____
Environmental Scientist

Date: _____

Reviewed by: 
Site Assessment Leader

Date: 05/09/01

Approved by: 
Quality Assurance Officer

Date: 05/09/01

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1.0 Scope and Applicability

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures for Region X Roy F. Weston, Inc. (WESTON®), Superfund Technical Assessment and Response Team (START) personnel in collecting representative surface water samples. Purposes for the collection and analysis of surface water samples by START personnel and their subsequent analysis will be detailed in site-specific Sampling and Quality Assurance Plans (SQAPs), and may include determining whether concentrations of hazardous substances in surface water samples exceed established action levels, confirming or identifying hazardous substances that may have impacted the environment, determining if contaminants are migrating off site, or if the concentrations of hazardous substances may present a risk to public health, welfare, or the environment.

The methodologies discussed in this procedure are applicable to the sampling of representative liquid samples, both aqueous and non-aqueous, from streams, rivers, lakes, ponds, lagoons, and surface impoundments. This SOP also includes samples collected from depth, as well as samples collected from the surface. These procedures are generic in nature and may be modified in whole or part to meet the handling and analytical requirements of the contaminants of concern, as well as by the constraints presented by the sampling area. However, if modifications occur, they shall be documented in the site logbook or report summarizing field activities.

2.0 Summary of Method

Sampling methods and equipment used for collecting aqueous and non-aqueous liquids vary widely, depending upon sampling depth within the water column and the actual matrix of the sample to be collected. Sampling of liquids is generally accomplished through the use of one of the following samplers or techniques: Kemmerer bottle, Bacon Bomb sampler, dip sampler, or direct method.

3.0 Acronym List

°C	-	Degrees Celsius
cc	-	Cubic centimeters
CLP	-	Contract Laboratory Program
COC	-	Chain-of-Custody
DAS	-	Delivery of Analytical Services
Dupl	-	Duplicate
DOT	-	Department of Transportation
EPA	-	U.S. Environmental Protection Agency
GPS	-	Global Positioning System
HASP	-	Health and Safety Plan
HCl	-	Hydrochloric Acid
HNO ₃	-	Nitric Acid
IATA	-	International Air Transport Association
IDW	-	Investigation-derived wastes

mg	-	Milligram
ml	-	Milliliters
MS/MSD	-	Matrix Spike/Matrix Spike Duplicate
Na ₂ S ₂ O ₃	-	Sodium Thiosulfate
NaOH	-	Sodium Hydroxide
OSHA	-	Occupational Safety and Health Administration
PE	-	Performance Evaluation
pest/PCB	-	Pesticide/Polychlorinated Biphenyl
PFDs	-	Personal Flotation Devices
PPE	-	Personal Protective Equipment
ppm	-	Parts per million
PVC	-	Polyvinyl Chloride
QAPP	-	Quality Assurance Project Plan
QA/QC	-	Quality Assurance/Quality Control
RFP	-	Request for Proposal
SDG	-	Sample Delivery Group
SOP	-	Standard Operating Procedure
SQAP	-	Sampling and Quality Assurance Plan
START	-	Superfund Technical Assessment and Response Team
SVOC	-	Semivolatile Organic Compound
TDD	-	Technical Direction Document
VOA	-	Volatile Organic Analysis
VOC	-	Volatile Organic Compound
WESTON	-	Roy F. Weston, Inc.

4.0 Health and Safety Warnings

START personnel performing work on hazardous waste sites will follow Occupational Safety and Health Administration (OSHA), U.S. Environmental Protection Agency (EPA), and WESTON/START specific health and safety procedures and protocols. START personnel conducting on-site and off-site surface water sampling activities will also be performing tasks in accordance with EPA-approved SQAPs, and Operating Practices prepared and approved by START.

In order to ensure the safety of personnel during sampling activities, the buddy system, periodic air monitoring, and caution will be used throughout field activities. To minimize risks due to chemical exposure, dermal and respiratory protection may be required if air monitoring equipment indicates that the environment is unsafe. Field activities will follow the site health and safety plan (HASp), which further addresses the safety considerations of the property. Hazards identified in or around the site may include physical hazards (slips, trips, and falls). Additional potential physical hazards exist in association with surface water sampling, and may include drowning or exposure due to hypothermia.

When collecting surface water samples from water bodies containing known or suspected hazardous substances, adequate precautions must be taken to ensure the sampler's safety. The START team

member collecting the sample should not get too close to the edge of the water where bank failure may cause him or her to lose their balance. The use of personal protective equipment (PPE) and engineering controls, such as safety nets, shall be specified in the site HASP, and shall be based on site-specific conditions. Appropriate PPE may include lifelines or personal flotation devices (PFDs). If sampling from a vessel is required, appropriate protective measures may be required, and shall be detailed in the site HASP.

The samples collected at the site will be transported to predesignated Contract Laboratory Program (CLP) or Delivery of Analytical Services (DAS) laboratories according to either Department of Transportation (DOT) Hazardous Materials Regulations or International Air Transport Association (IATA) Dangerous Goods Regulations, or hand-delivered to the laboratories. Samples will be transported in a manner that will maintain their integrity, as well as protect against detrimental effects from sample breakage or leakage. The Roy F. Weston, Inc. *Manual of Procedures for Shipping and Transporting Dangerous Goods* will be followed whenever samples are shipped.

Decontamination wastes will be collected and secured on site. Separate containers will be used for the aqueous wastes and for flammable, non-chlorinated solvents (methanol and hexane) wastes. Proper personal protection will be worn during decontamination procedures and will include gloves, eye protection, and splash-resistant protective clothing. Off-site disposal of decontamination wastes and contaminated PPE will be conducted through the Subcontract Agreement established by Region X START for disposal of investigation-derived wastes (IDW). Non-contaminated wastes will be tightly sealed, double-bagged, and disposed of appropriately.

5.0 Interferences

Some interferences or potential problems associated with surface water 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. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve the use of contaminated equipment, disturbance of bottom sediments resulting in the entrainment of sediment fines in the surface water sample, or inadequate homogenization of the samples where required, resulting in variable, non-representative results.

6.0 Personnel Qualifications

Training of START members will be provided to ensure that technical, operational, and quality requirements are understood. START personnel are trained in-house to conduct surface water sampling activities. Prior to sampling from a vessel, the WESTON Operating Procedures relevant to the Operation and Use of Boats shall be reviewed. Training includes reviewing this SOP and other applicable SOPs and/or guidance documents; health and safety training; "hands-on" experience conducting sampling activities with more experienced START personnel, and with performing instrument calibrations.

7.0 Apparatus and Materials

Equipment/materials for collecting surface water samples includes, but is not limited to the following:

- Site-specific SQAP.
- Standard Operating Procedure for Surface Water Sampling.
- Safety equipment specified in the site HASP.
- Field map of site.
- Logbook.
- Waterproof pen.
- Field data sheets (e.g. sample description sheets, etc.).
- Tape measure.
- Compass.
- Survey stakes, flags (white), and buoys and anchors.
- Camera and film.
- Kemmerer bottles.
- Bacon bomb sampler.
- Dip sampler.
- Line and messengers.
- Sampling jars.
- Preservatives.
- Safety equipment.
- Decontamination fluids (supplies)/equipment (pump sprayers, brushes, etc.).
- Plastic sheeting (Visqueen).
- Sealable plastic bags.
- Bottle labels.
- Chain-of-custody forms and seals.
- Coolers and ice.
- Vermiculite.
- Strapping tape.

7.1 Reagents

Reagents used for the preservation of surface water samples include hydrochloric acid (HCl), nitric acid (HNO₃), sodium hydroxide (NaOH), sodium sulfite, and ascorbic acid. HCl, HNO₃, and NaOH are used to preserve surface water samples collected in the field for volatile organic compound (VOC), metals, and cyanide analyses, respectively. It is not expected that surface water samples will contain free (residual) chlorine unless they are treated effluent samples or are collected near the outfall of a treated water effluent. If chlorination is suspected, the water will be tested for free chlorine using chlorine test strips according to the manufacturer's directions. If samples are collected for VOC, semivolatile organic compound (SVOC), or cyanide analyses, samples that test positive for residual

chlorine will require treatment with a reducing agent before sample preservation. Ascorbic acid is used as a reducing agent for samples collected for VOC and cyanide analyses, and sodium sulfite is used as a reducing agent for samples collected for SVOC analysis. Samples collected for oil analysis may sometimes be preserved with HCl or sulfuric acid to prevent degradation by microbial action.

Reagents used for decontamination of sampling equipment are discussed in *Section 4.0, Health and Safety Warnings*. Non-aqueous samples are typically not preserved due to the uncertain nature of the matrix, which may evolve harmful gases upon addition of acid or base to the sample.

8.0 Method Calibration

This section is not applicable to this SOP.

9.0 Sample Collection

Under the Removal Program, surface water samples may be collected to determine if any hazardous substances are present on site at such concentrations that a removal action is warranted or if contaminants from on-site sources are migrating off site. Under the Site Assessment Program, surface water samples are generally collected to determine if concentrations of hazardous substances document an observed release to the surface water pathway and can be used to document actual contamination to drinking water supplies (surface water intakes), fisheries (food chain), and/or sensitive environments (wetlands, Clean Water Act-protected bodies, endangered and/threatened species, etc.) under the Hazard Ranking System.

In order to collect a representative sample, the hydrology and morphometrics (e.g., measurements of volume, depth, etc.) 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.

Water quality data should be collected in impoundments to determine if stratification is present. Measurements of dissolved oxygen, pH, and temperature can indicate if stratification exists which would affect analytical results. Measurements should be collected at 3-foot intervals from the substrate to the surface using an appropriate instrument, such as a Hydrolab (or equivalent).

Water quality measurements such as dissolved oxygen, pH, temperature, conductivity, and oxidation-reduction potential can assist in the interpretation of analytical data and the selection of sampling sites and depths any time surface water samples are collected.

Factors in the selection of a sampling device for sampling liquids in streams, rivers, lakes, ponds, lagoons, and surface impoundments include whether the sample is to be collected from the shore or from a boat, the desired depth from which the sample is to be collected, and the overall depth and flow direction of river or stream.

The appropriate sampling device must be of a proper composition. Samplers constructed of glass, stainless steel, polyvinyl chloride (PVC), or Teflon should be used based upon the analyses to be performed.

9.1 Presampling Preparation

Following EPA approval of the site-specific SQAPs, and prior to conducting sampling activities, a pre-sampling meeting will be held by START team members to discuss the proposed sampling strategy and site health and safety issues. Attendees of the pre-sampling meeting will include the Project Manager, Site Leader, samplers, and the START Health and Safety Officer. During the pre-sampling meeting, the Site Leader will discuss the site history, contaminants of concern, sampling methodology, individual responsibilities, sample shipment or delivery, health and safety issues, and lines of communication anticipated during the sampling event.

Prior to mobilizing to the site to conduct sampling activities, the START Site Leader will fill out an equipment/supply list and transmit the list to the WESTON Equipment Stores technician one week prior to the sampling event. Necessary sampling equipment, sample containers, PPE, and vehicles are therefore reserved. Analytical services are procured for water samples through CLP or subcontract DAS laboratories at least one week prior to the sampling event by START analytical staff members under the supervision of the START Lead Chemist.

9.2 Surface Water Sample Collection

Prior to conducting any on-site activities, START members review and sign the site-specific HASP. The START field team will establish a command post upwind of suspected source areas, if possible. START members will perform calibration checks of air monitoring instruments and document background ambient air monitoring levels. The samplers will decontaminate the sampling equipment. Decontamination will be conducted in accordance with the HASP, and/or applicable SOPs. Decontamination generally consists of an alconox and water wash followed by a distilled water rinse, followed by an isopropanol rinse, followed by a de-ionized water final rinse, and air drying. Additionally, where high concentrations of specific substances are anticipated, chemicals such as methanol, hexane, and/or HNO₃ may be used in the decontamination process. Equipment decontamination fluids and PPE generated during sampling activities will be containerized and disposed appropriately based on the results of laboratory analyses of samples collected.

The number of samples and the locations of surface water samples to be collected will be detailed in the site-specific SQAP. For streams, rivers, lakes, and other surface waters, a direct sampling method may be utilized to collect the samples. This method is not to be used for sampling lagoons or other impoundments where contact with contaminants is a concern. The direct method may also be used when collecting samples from outfall pipes where

effluent flow is sufficiently low to avoid exposure from splashing. A dipper with a long handle may be used to collect surface water samples from outfall pipes, lagoon banks, or any other location where direct access is limited. The long handle allows access from a safe, discrete location.

It is not expected that surface water samples will contain free (residual) chlorine unless they are treated effluent samples or are collected near the outfall of a treated water effluent. If chlorination is suspected, the water will be tested for free chlorine using chlorine test strips according to the manufacturer's directions. When collecting samples for VOC, SVOC, or cyanide analyses, samples that test positive for residual chlorine will require treatment with a reducing agent before sample preservation. These procedures are discussed in the following sections.

9.2.1 Sample Collection Procedures for Direct Surface Water Sampling

The sampling station will be accessed by appropriate means. For shallow stream stations, the sample will be collected by placing the appropriate sample container under the water surface, pointing the container upstream. The container must be upstream of the collector. The sampler(s) will avoid disturbing the substrate. For lakes and other impoundments, the sample will be collected under the water surface avoiding surface debris, any boat wakes, and contact with the sampler's gloves. When collecting samples from several stations along a stream, always begin collecting samples from downstream to upstream locations.

When using the direct method, do not use pre-preserved sample bottles as the collection method may dilute the concentration of the preservative necessary for proper sample preservation.

Surface water samples suspected of containing chlorine (for example, samples collected downstream of a wastewater treatment plant) must be tested for residual chlorine before the samples are placed into containers and preserved. To test for chlorine, use potassium-iodide paper dipped in a volatile organic analysis (VOA) vial containing 2 to 5 milliliters (ml) of the sample. A blue coloration on the wet paper indicates the presence of residual chlorine. The potassium-iodide paper should be kept away from light during storage. Samples that test positive for residual chlorine must be pre-treated with either sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) or ascorbic acid. [Note: Samples are only to be treated using $\text{Na}_2\text{S}_2\text{O}_3$ or ascorbic acid if the samples test positive]. Add 0.008% (a few crystals) of $\text{Na}_2\text{S}_2\text{O}_3$ to an empty VOA vial. Add the sample to the VOA vial, and preserve as directed in the section below. The sample aliquot submitted for cyanide analysis must be pre-treated with 0.6 grams of ascorbic acid per liter of sample. The following section discusses collecting surface water samples for typical analytical parameters under the START Program.

VOC Sampling - The proper collection of a sample for VOC analysis requires minimal disturbance of the sample to limit volatilization, and therefore prevent loss of volatile compounds from the sample. VOC samples shall not be collected and/or preserved near a running motor or any type of exhaust system due to possible contamination by discharges, fumes, or vapors.

If the sample tests positive for residual chlorine, it must be treated with $\text{Na}_2\text{S}_2\text{O}_3$ prior to pH adjustment using HCl preservative. Each sample container will be filled to just overflowing (forming a convex meniscus) in such a manner that no air bubbles pass through the sample as the bottle is being filled. Care will be taken to avoid flushing the ascorbic acid from the sample container.

The pH of the sample will be adjusted to < 2 by carefully adding the appropriate number drops of 1:1 HCl. Generally one drop of 1:1 HCl for each 20 ml of sample volume is adequate; however, some samples collected in the field have different buffering capacities and may require additional drops of HCl to achieve a pH of < 2 . Alternately, the sample containers can be pre-preserved with HCl and the sample carefully filled to just overflowing to avoid washing out the preservative.

The sample container is then sealed so that no air bubbles are entrapped. The sealed bottle will be inverted, tapped gently on the side, and observed for 10 seconds for the presence of air bubbles. If an air bubble appears, the sample will be discarded and the collection procedure repeated. The sample containers will then be shaken vigorously to mix the preservative; placed in a sealable plastic bag; and placed into a cooler with ice.

The holding time for VOC samples is ten (10) days in the CLP. Samples should be shipped or delivered to the laboratory daily so as not to exceed the holding time. Ensure that the samples remain at 4 degrees Celsius ($^{\circ}\text{C}$), but do not allow them to freeze.

One trip blank sample (organic-free water) will be collected prior to the sampling event. Preparation of trip blanks is discussed in *Section 14.3, Trip Blanks*.

SVOC Sampling - Samples will be collected as described above into appropriate size glass containers. CLP does not specify addition of preservative. The container cap(s) will then be secured, and the samples will be preserved by immediately placing the containers in a cooler on ice. In the CLP, aqueous samples are to be extracted in 5 days following VTSR, solid samples extracted in 7 days. VTSR and all extracts analyzed within 40 days.

Pesticides (pest)/polychlorinated biphenyls (PCBs) Sampling - Samples will be collected as described above into appropriate size glass containers. The container

cap(s) will then be secured and the samples will be preserved by immediately placing the containers in a cooler on ice.

Metals Sampling - Samples will be collected as described above into appropriate size plastic or glass containers and preserved with HNO₃ to a pH of < 2. The container cap(s) will then be secured; and the samples will be preserved by immediately placing the containers in a cooler on ice.

Cyanide (Total and Amenable) Sampling - Samples will be collected as described above into the appropriate glass or plastic containers to which 0.6 gm of ascorbic acid has been added. Care will be taken to avoid flushing the ascorbic acid from the sample container. The samples will then be preserved with NaOH to a pH of > 12. The container cap(s) will then be secured, and the samples will be preserved by immediately placing the containers in a cooler on ice.

pH Sampling - Samples will be collected as described above into glass or plastic containers. The container cap(s) will then be secured, and the samples will be preserved by immediately placing the containers in a cooler on ice. Samples collected for pH analysis will be tested in the field and must be transported to a laboratory for analysis as soon as possible after collection. A field pH may be determined by touching a piece of pH paper to a drop of sample in the cap or around the lip of the sample container. The field pH should then be recorded on the Chain-of-Custody (COC) record.

Oil Identification Sampling - It is recommended that wherever possible, liquid samples for this parameter be collected directly into the appropriate glass container that is shipped to the laboratory for analysis. Since oil tends to adhere to the walls of the container or samplers, results may be biased low when oil in the sample clings to the surfaces of the sampling device upon transfer of the liquid into the sample bottle. Oil can also be degraded by microbial action, and therefore must be preserved with HCl or sulfuric acid to a pH of < 2, and then placed in a cooler with ice. If an oil layer is present on top of the water surface, a representative amount of the oil should be collected with the sample.

9.2.2 Sample Collection Procedures for Dipper Sampling

The dipper device is assembled by fastening a wide-mouth glass sampling container to a long handle. The device is then extended to the sample location, and the sample collected by allowing the material to flow into the sample container. When the sample container is full, the dipper device is retrieved and the material transferred into the appropriate, pre-labeled sample container(s). Collect samples for VOC analysis first, followed by SVOC, pest/PCB, metals, and cyanide following the procedures described in Section 9.3.1 - *Sample Collection Procedures for Direct Surface Water Sampling*.

When using the dipper method, one wide-mouthed glass sampling container is dedicated to each sampling station to avoid the cross-contamination of samples.

9.2.3 Sample Collection Procedures Using a Kemmerer Bottle

The 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 be sampled to pass through this tube.
- 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.
- Retrieve the sampler and discharge the first 10 to 20 ml to clear any potential contamination on the valve. Collect samples for VOC analysis first, followed by SVOC, pest/PCB, metals, and cyanide following the procedures described in Section 9.3.1 - *Sample Collection Procedures for Direct Surface Water Sampling*.

9.2.4 Sample Collection Procedures Using a Bacon Bomb Sampler

A Bacon Bomb sampler may be used in similar situations 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.
- Release the trigger line and retrieve the sampler.
- Transfer the sample to the appropriate sample container by pulling the trigger. Collect samples for VOC analysis first, followed by SVOC, pest/PCB, metals, and cyanide following the procedures described in Section 9.3.1 - *Sample Collection Procedures for Direct Surface Water Sampling*.

10.0 Handling and Preservation

Trip blanks are used to assess the degree of VOC contamination introduced into samples during sample handling, shipment, storage, and analysis. One pair of trip blanks is included in each cooler containing VOC samples. Trip blank samples prepared from organic-free water will be collected in 40-ml VOA vials prior to the sampling event. The glass sample container(s) used for the trip blanks will be from the same lot as the corresponding sample vials. Each bottle of the trip blank sample will be preserved with one drop of 1:1 HCl per 20 ml of sample. The trip blank sample will be handled in the same fashion as the samples collected in the field, labeled, placed into a sealable plastic bag, transported to the field with the empty sample containers, and shipped to the laboratory in the same cooler as the VOC field samples.

Contamination may also be introduced from the use of poorly cleaned non-dedicated sampling equipment, chemical preservatives, sample bottles, or from improper shipping and handling. Chemicals that may be used during the decontamination process include Alconox detergent, HNO₃, hexane, and isopropanol. Chemical preservatives commonly used to preserve aqueous samples and equipment rinsate blanks include HCl, HNO₃, NaOH, and sulfuric acid. START routinely purchases high-quality certified-clean sampling containers to prevent contamination from sample bottles. Sample jars are also sealed in plastic bags and/or sleeve-protectors to avoid bottle breakage and provide containment in the event that a bottle is broken in shipment. The samples are carefully placed in coolers lined with vermiculite packing material and ice packs, to avoid cross-contamination due to breakage of samples during shipment.

Samples are shipped to analytical laboratories expeditiously to ensure that holding times are not exceeded. A cooler temperature blank, consisting of a small container of tap water, is included in each cooler to monitor the temperature of samples upon receipt at the laboratory. Samples are considered to be properly preserved if they are maintained within the 2 to 6 °C temperature range. Microbial activity is minimized at these low temperatures, thereby preventing biodegradation of organic contaminants in the sample matrix.

11.0 Sample Preparation and Analysis

This section is not applicable to this SOP.

12.0 Troubleshooting

All field screening 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/operation, and they must be documented.

13.0 Data Acquisition, Calculations, and Data Reduction

13.1 Computer Hardware and Software

Surface water sample locations may be located using a Trimble Pro XRS or GeoExplorer Global Positioning System (GPS) receiver. Prior to conducting field work, Trimble Pathfinder Office 2.51 (Pathfinder software) may be used to generate a data dictionary to be used during collection of sample locations. The data dictionary is then transferred from the computer to the GPS datalogger. Upon returning from the field, GPS data are downloaded from the GPS datalogger to a computer using the Pathfinder software. GPS data undergo differential correction, with base station data obtained from a variety of community base stations depending upon geographic location to improve location accuracy. Pathfinder is used to export GPS data in a variety of formats [i.e. ArcView shapefile, ARC/INFO, dBASE, and AutoCAD export (DXF) files]. Typically, GPS data are exported into dBASE files, and then manipulated within Microsoft Excel 2000 software. Data are then manipulated where they can be utilized in site diagrams to display sample locations.

13.2 Data Management and Records Management

Field observations made during the sampling event will be recorded in a site logbook and/or field data sheets, including description of sampling locations and any deviations from the site-specific SQAPs. COC will be maintained until samples are relinquished to a courier or to the laboratories assigned to perform the analyses. Photographs will be taken to document site conditions. The location and direction from which photographs are taken will be noted in the field logbook, in accordance with the scope of work. Reports, site file memoranda, figures, tables, boring logs, etc. will be saved in site-specific Technical Direction Document (TDD) directories.

14.0 Quality Assurance and Quality Control Section

This section describes quality control/quality assurance (QA/QC) pertinent to surface water samples, and the types and uses of the QA/QC samples that are collected in the field. QA/QC samples are analyzed to provide information on the variability and usability of environmental sample results. They assist in identifying the origin of analytical discrepancies to help determine how the analytical results should be used. They are used mostly to validate analytical results.

A data quality review of the laboratory sample analyses will be conducted by U.S. EPA. At a minimum, a Tier I data validation will be conducted, which involves determining data completeness through a data package inventory check. A Tier II data validation is generally completed for Site Assessment projects, and may be done at the specific request of the EPA On-Scene Coordinator for Removal projects. Tier II data validation includes completing a data package inventory, and qualifying the data based on deficiencies in QC sample results. Tier III data validation includes all the elements of a Tier II data validation, plus a check of calculations and an evaluation of sample

results through an evaluation of the raw data. Deliverables resulting from the data validation process include a data validation memorandum and data tables. Qualifiers are applied to sample results for data packages which go through a Tier II or Tier III data validation, and provide the data user with a sense of the usability and limitations of the data.

Field duplicates, matrix spike/matrix spike duplicate (MS/MSD), laboratory duplicates (Dupl), trip blanks, rinsate (equipment) blanks, PE, and temperature blanks are discussed below. QA/QC results may suggest the need for modifying sample collection, preparation, handling, or analytical procedures if the resultant data do not meet QA objectives.

14.1 Field Duplicates

Field duplicates are used to assess the degree of sample heterogeneity and the reproducibility of the sample collection procedure and the laboratory analysis. Field duplicates are typically collected for surface water samples which are submitted for field screening analysis or for Site Assessment scoring activities. One field duplicate is typically collected for each matrix type and sample parameter for every 20 stations for Site Assessment projects. The field duplicate is assigned an individual sample number.

14.2 Laboratory Matrix Spike, Matrix Spike Duplicate, and Duplicate

MS and MSD samples are used to monitor laboratory performance and determine how effectively the analytical method is able to recover target compounds that are spiked into the matrix of interest. MS/MSD samples are spiked by the analyst with a known concentration of a target analytes to monitor recovery of the target compounds. It is necessary to provide extra volume of sample to the laboratory for spiking analyses. Generally, organic parameters (e.g., VOC, SVOC, and pest/PCBs) require triplicate sample volume for MS/MSD analysis. Inorganic analyses typically require MS and laboratory Dupl analyses. Laboratory duplicate precision monitors the laboratory's ability to reproduce its results. Inorganic parameters (e.g., metals and cyanide) require duplicate sample volume. The required frequency for MS/MSD and MS/Dupl analysis is generally one per every 20 samples of each matrix for each requested analytical parameter. Extra volume for the pre-designated QC sample must be provided to the laboratory for MS/MSD and MS/Dupl analyses. The QC sample, its MS/MSD, and MS/Dupl all receive the same sample number.

14.3 Trip Blanks

Trip blanks are used to assess the degree of contamination introduced into samples during sample handling, shipment, storage, and analysis. Contamination may be introduced from the sample bottle, the preservatives used, cross-contamination from highly contaminated samples, or from poor shipping and handling procedures experienced both in the field and in the laboratory. Trip blanks are typically collected for VOC analysis. Trip blanks prepared from organic-free water will be collected prior to the sampling event. Each bottle of the trip blank

sample will be preserved with one drop of 1:1 HCl per 20 ml of sample to achieve a pH of < 2. Trip blanks are handled, transported, and analyzed in the same manner as the other samples collected for that analysis. One set of trip blanks is collected for VOC analysis for each cooler in which VOC samples are shipped.

14.4 Rinsate (Equipment) Blanks

Dedicated or decontaminated sampling equipment will be used at each surface water sample location to minimize cross-contamination. Sampling equipment must be decontaminated prior to the start of sampling activities as well as between sample locations, unless the sampling activity is dedicated to one sample location. Rinsate blanks are used to assess contamination (typically, cross-contamination) brought about by improper decontamination procedures between sampling stations. Rinsate blanks are not required for dedicated, disposable sampling implements. Examples of equipment requiring decontamination and rinsate blanks include Kemmerer samplers and Bacon Bomb samplers.

Rinsate blanks are obtained by pouring analyte-free water over decontaminated sampling equipment to test for residual contamination. The water is collected into the appropriate sample containers which are handled (e.g., preserved), shipped, and analyzed for the same set of analytical parameters as the samples that were collected that day. Where non-dedicated sampling equipment is used, rinsate blanks must be collected at the rate of one per day per 20 stations for each parameter for which surface water samples are collected.

14.5 Temperature Blanks

Temperature blanks provide information on the preservation (temperature) of the samples during shipment to the laboratories. Temperature blanks are obtained by pouring tap water into a 40-ml glass vial and placing one temperature blank per cooler of samples.

15.0 Reference Section

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**REGION 10
SUPERFUND TECHNICAL ASSESSMENT AND RESPONSE TEAM
(START)**

**STANDARD OPERATING PROCEDURE
FOR**

**X-RAY FLUORESCENCE
FIELD SCREENING**

FEBRUARY 1999

Prepared by

Region 10 START
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 $\pm 20\%$ 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 K α) 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 K α and K β peak, or the L α and L β 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 $\pm 20\%$ 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 $\pm 20\%$ 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 $\pm 20\%$ of the true sample value, as determined by the appropriate EPA CLP-style analysis. If sample results from this analysis lie outside the $\pm 20\%$ 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 (heteroscedasticity), 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 B
SUPPLEMENTAL FORMS

CORRECTIVE ACTION CHECKLIST

Project Name and Number:

Sample Dates Involved:

Measurement Parameter(s):

Acceptable Data Range:

Problem Areas Requiring Corrective Action:

Measures Required to Correct Problems:

Means of Detecting Problems and Verifying Correction:

Initiators Name: _____ Date:

Project Approval: _____ Date:

QA Officer/Reviewer: _____ Date:



**UPPER COLUMBIA RIVER/LAKE ROOSEVELT ESI
SURFACE SEDIMENT FIELD SAMPLING RECORD**

Project Number <u>12644 001 002 0101 00</u>	Sampling Personnel _____
Date _____	Weather _____
Location Description _____	
Uncorrected GPS Coordinates _____	
Corrected GPS Coordinates _____	
GPS File Name _____	Sampling Method _____
WESTON Sample No _____	Water Depth to Sediment _____
EPA Sample No _____	Photograph roll _____ exposure: _____
Analyses _____	

Grab #	Time	Penetration Depth	Sample Material Physical Description [Grain Size (% gravel/sand/silt/clay), Color, Odor, Slag, Organics, Debris, Etc]

Notes _____

Sampler's Signature: _____



SURFACE WATER FIELD SAMPLING RECORD

Project Name: _____	Sampling Personnel: _____
Project Location _____	Project Number _____
Date _____	Weather: _____

Sampling Point Number: _____ Time: _____ Depth to Sampling Point: _____

Location Description: _____ Coordinates: _____

Access Method: _____ Sampling Method: _____

Description of Sample (color, turbidity, odor): _____

WESTON Sample ID: _____

EPA Sample Number: _____

<u>Sample Container(s)</u>	<u>Quantity</u>	<u>Analysis</u>
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Sampling Point Number: _____ Time: _____ Depth to Sampling Point: _____

Location Description: _____ Coordinates: _____

Access Method: _____ Sampling Method: _____

Description of Sample (color, turbidity, odor): _____

WESTON Sample ID: _____

EPA Sample Number: _____

<u>Sample Container(s)</u>	<u>Quantity</u>	<u>Analysis</u>
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Notes: _____

Sampler's Signature: _____

SAMPLE PLAN ALTERATION FORM

Project Name and Number: _____

Material to be sampled: _____

Measurement Parameter: _____

Standard Procedure for Field collection and Laboratory Analysis (cite references):

Reason for change in Field Procedure or Analytical Variance:

Variance from Field or Analytical Procedure:

Special Equipment, Materials, or Personnel Required:

Initiators Name: _____ Date: _____

Project Approval: _____ Date: _____

QA Officer/Reviewer: _____ Date: _____

APPENDIX C

SAMPLE DOCUMENTATION AND CHAIN-OF-CUSTODY FORMS

Receipt for Samples

United States
Environmental Protection
Agency, Region 10

Comprehensive Environmental Response, Compensation and
Liability Act as Amended by the Superfund Amendments
and Reauthorization Act

Superfund Program
U.S. Environmental Protection Agency
1200 Sixth Avenue, Seattle, WA 98101

Project No/TDD No.		Site Name		Name of Facility/Location			
Sampler(s)			Split Samples <input type="checkbox"/> Offered <input type="checkbox"/> Declined				
Date	Time	Sample Number	Description	Remarks			
Site Representative Signature		Telephone No.		Title		Date	Time

Original

United States Environmental Protection Agency

Region 10
1200 Sixth Avenue
Seattle, Washington 98101-9797



★ U S GPO 1995-692-882



Remarks:	CLP No.	Project Code	Month/Day/Year/Time	Tag No	133038
	Station Location / No		Sampler Name(s) (Please Print)		
Regional Tracking No.	Other (Write In)		Organics	Metals	Inorganics
	Phenols		Volatiles	Cyanide	Oil and Grease
	Herbicides		Semi-volatiles	Oil and Grease	COD, TOC, Nutrients
	PAHs		Pesticides/PCBs	BOD	General Chemistry
	Pesticides/PCBs		Volatiles	Solids (TSS, TDS, SS)	Acid Base
	Other (Write In)		Semi-volatiles	Oil and Grease	Other
	Phenols		Pesticides/PCBs	Oil and Grease	Chemical:
	Herbicides		PAHs	Oil and Grease	Ice <input type="checkbox"/> No <input type="checkbox"/>
	Other (Write In)		Herbicides	Oil and Grease	Acid <input type="checkbox"/> Base <input type="checkbox"/> Other <input type="checkbox"/>
	Phenols		Phenols	Oil and Grease	Preservative:



Custody Seal

Date: _____
Signature: _____

Signature: _____
Date: _____

Custody Seal



CUSTODY SEAL

Date: _____
Signature: _____



APPENDIX D

COMMERCIAL LABORATORY STATEMENT OF WORK

(To be attached following laboratory selection)