Quality Assurance Project Plan

Little Squalicum Creek Biological Toxicity Assessment

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October 2003

Publication No. 03-03-110

This plan is available on the Department of Ecology home page on the World Wide Web at http://www.ecy.wa.gov/biblio/0303110.html

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Biological Toxicity Assessment

October 2003

303(d) listings addressed in this study: None

Waterbody number: WA-01-0050 (Bellingham Bay)

Ecology EIM number: NBLA0002

Approvals

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Abstract

This project will investigate sediments from Little Squalicum Creek for contamination and toxicity to aquatic life. Little Squalicum Creek is a small stream in Bellingham, Washington, that empties into Bellingham Bay. It has a history of pollution, including spills from an adjacent wood treating plant. Sediment samples will be evaluated for toxicity using Microtox, amphipod (*Hyalella*), and midge (*Chironomus*) bioassays.

Results from this study will assist the City of Bellingham in planning an investigation of pollution in Little Squalicum Creek prior to the redevelopment of the area into a community park and trail corridor.

Background and Problem Statement

Little Squalicum Creek is a small stream, approximately 1500 feet long, in Bellingham that discharges to Bellingham Bay (Figures 1 and 2). Depth is generally less than one foot, with a channel width ranging from about three to six feet (Ecology and Environment, Inc., 2001). The creek is fed by stormwater outfalls, two springs, and several small seeps. During wetter seasons, flow is estimated at one to 10 cfs, but the creek bed may be exposed during drier seasons. The stream is normally not tidally influenced because it discharges through a beach culvert above high tide levels.

Over the last century, Little Squalicum Creek and the surrounding ravine have been subjected to considerable physical disturbance and episodes of pollution. The City of Bellingham is now proposing to redevelop this site into a community park and trail corridor. The city plans to divert the creek into a meandering path through the park and to remove a culvert that is blocking fish from moving up the creek. However, before work begins on this project, the city will investigate the extent of remaining pollution with a grant from the U.S. Environmental Protection Agency's Brownfields program.

Past physical alteration of the creek has included a shortening of its length and reduction of its flow due to the diversion of the upper reach through a stormwater drain to nearby Squalicum Creek (Ecology and Environment, 2001). Sand and gravel were mined from parts of the ravine until the late 1960s. The Ecology and Environment report states, "The entire ravine has been altered substantially from natural conditions with rerouting of the original creek bed and significant changes to the soils and lithology (e.g., backfilling of gravel pit excavations, release of log storage debris, landfilling activities, temporary road maintenance, rail bed and track placement and subsequent track removal, and filling and paving of some areas)."

Known or suspected historical pollution of the creek and ravine is varied (Ecology and Environment, Inc., 2001). The city operated a landfill in the ravine beginning in 1936. Refuse from an adjacent sugar plant also was reportedly dumped in the ravine. Burlington Northern Railroad disposed of wastes (possibly oil wastes) in the vicinity that may have migrated into the ravine through groundwater transport. Storm drain discharges may also have been contributors. An adjacent wood treating facility, Oeser Company, has had spills into the creek. At least some of these spills were of pentachlorophenol preservative in carrier oil.

A preliminary site characterization of the Oeser Company Superfund Site included sediment sampling from Little Squalicum Creek at 11 stations (Figure 3). All but one of these stations exceeded a dioxin criterion for the protection of human health (6.7 ng/kg reported as 2,3,7,8-TCDD TEQ, MTCA Method B direct contact value). Polycyclic aromatic hydrocarbon (PAH) concentrations exceeded Sediment Quality Standards (SQS) for the protection of marine aquatic life[1] at eight stations (Table 1), and the pentachlorophenol SQS was exceeded at four stations. However, from a bioassay evaluation of these stations (10-day *Hyalella azteca* growth and survival) it was concluded in the Site Characterization Report that current levels of sediment

[1] Marine standards were used for comparison because freshwater sediment quality standards have not been adopted by the Washington State Department of Ecology.

contamination in the creek do not pose a hazard to aquatic life (Ecology and Environment, 2001).

There are some uncertainties relating to the Site Characterization Report's evaluation of Little Squalicum Creek sediments, for several reasons:

- Sampling was not conducted immediately downstream of the Oeser storm drain discharge point (i.e., gap between SD10 and SD08), and near the mouth of the stream (gap between SD01 and SD02). See Figure 3 for locations of these stations.
- Only one bioassay test (10-day *Hyalella azteca)* was conducted, and testing with *Hyalella* was not performed by an Ecology-accredited laboratory. Ecology recommends testing with a suite of three bioassays, two acute effects and one chronic effects test, consistent with procedures for marine and estuarine sediments (MEL, 2001).
- Despite the Report's conclusion of no hazard to aquatic life, the reported *Hyalella* test results do show evidence of toxic effects. At five of the stations, there was a reduction in *Hyalella* growth relative to a test results for the "background" station immediately upstream of the Oeser outfall (Figure 4-95 in Ecology and Environment, 2001).

To address these uncertainties, sediment sampling will be conducted in the two "gap" stream segments noted above. At each of the new sampling locations, sediment samples will be characterized chemically and evaluated for toxicity using three bioassay tests. Results from this investigation may be used by the City of Bellingham to plan further studies under the Brownfields grant.

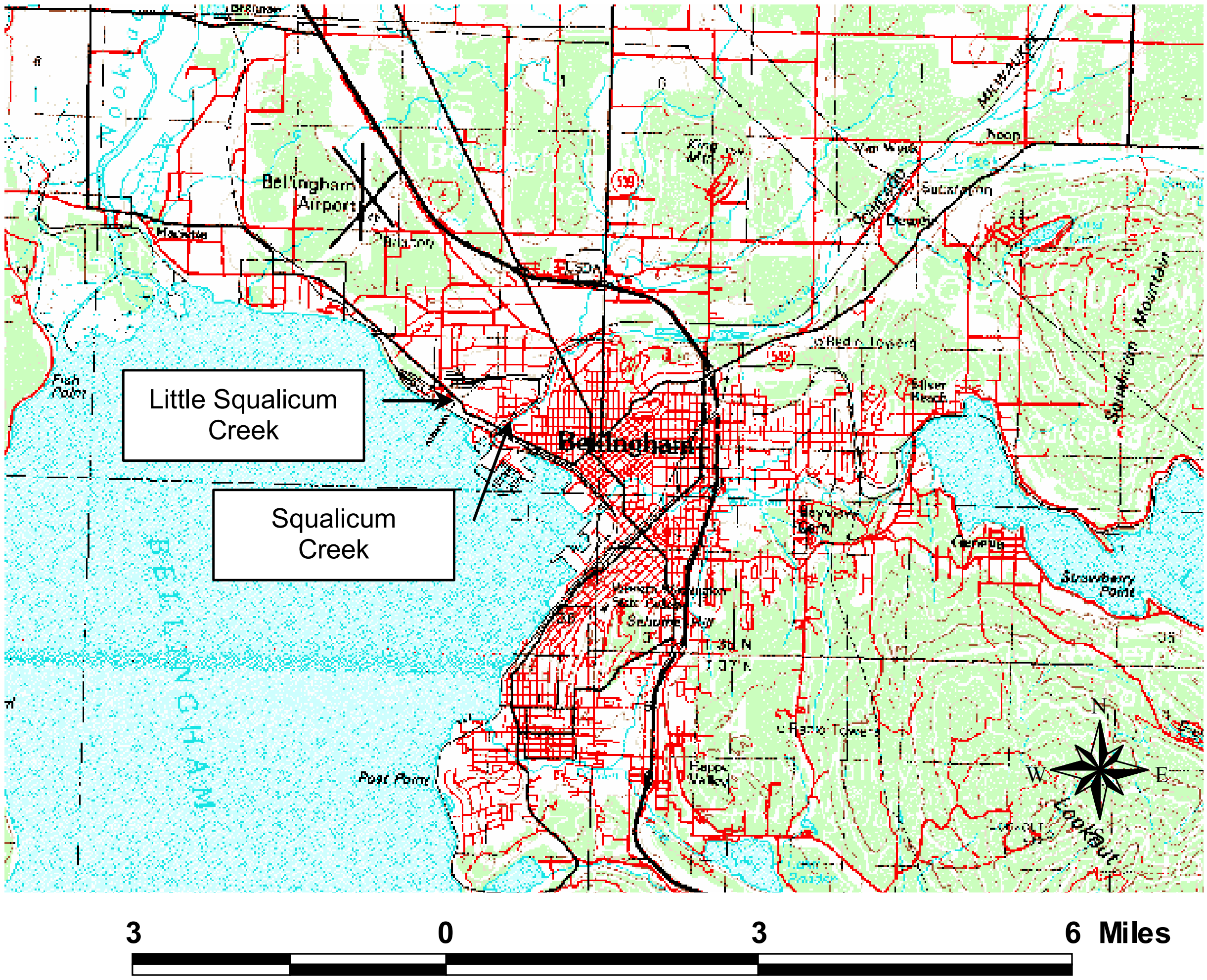

Figure 1. General Location of Little Squalicum Creek

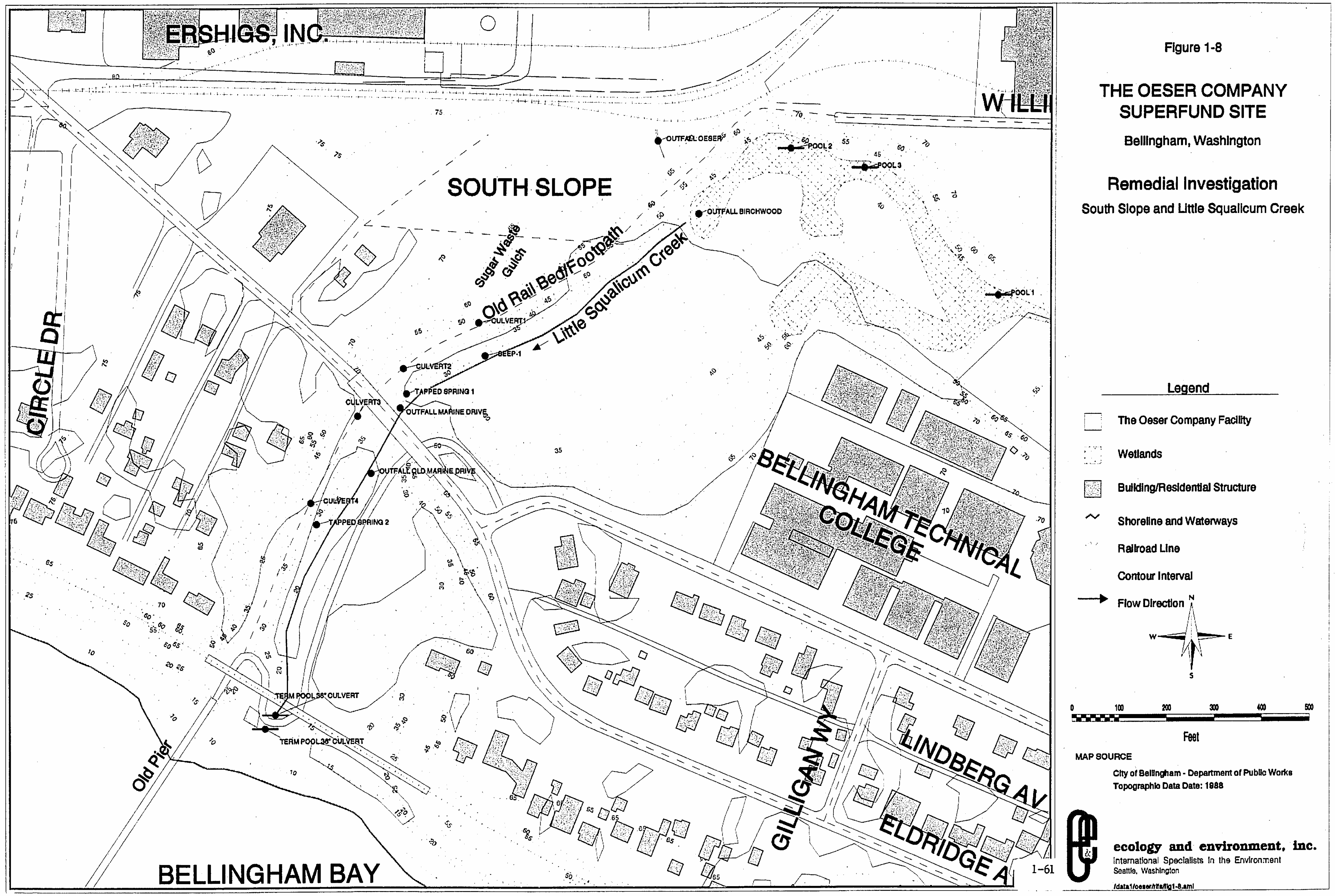

Figure 2. Map of Little Squalicum Creek, with locations of outfalls, culverts and springs. Source: Ecology and Environment, Inc., 2001.

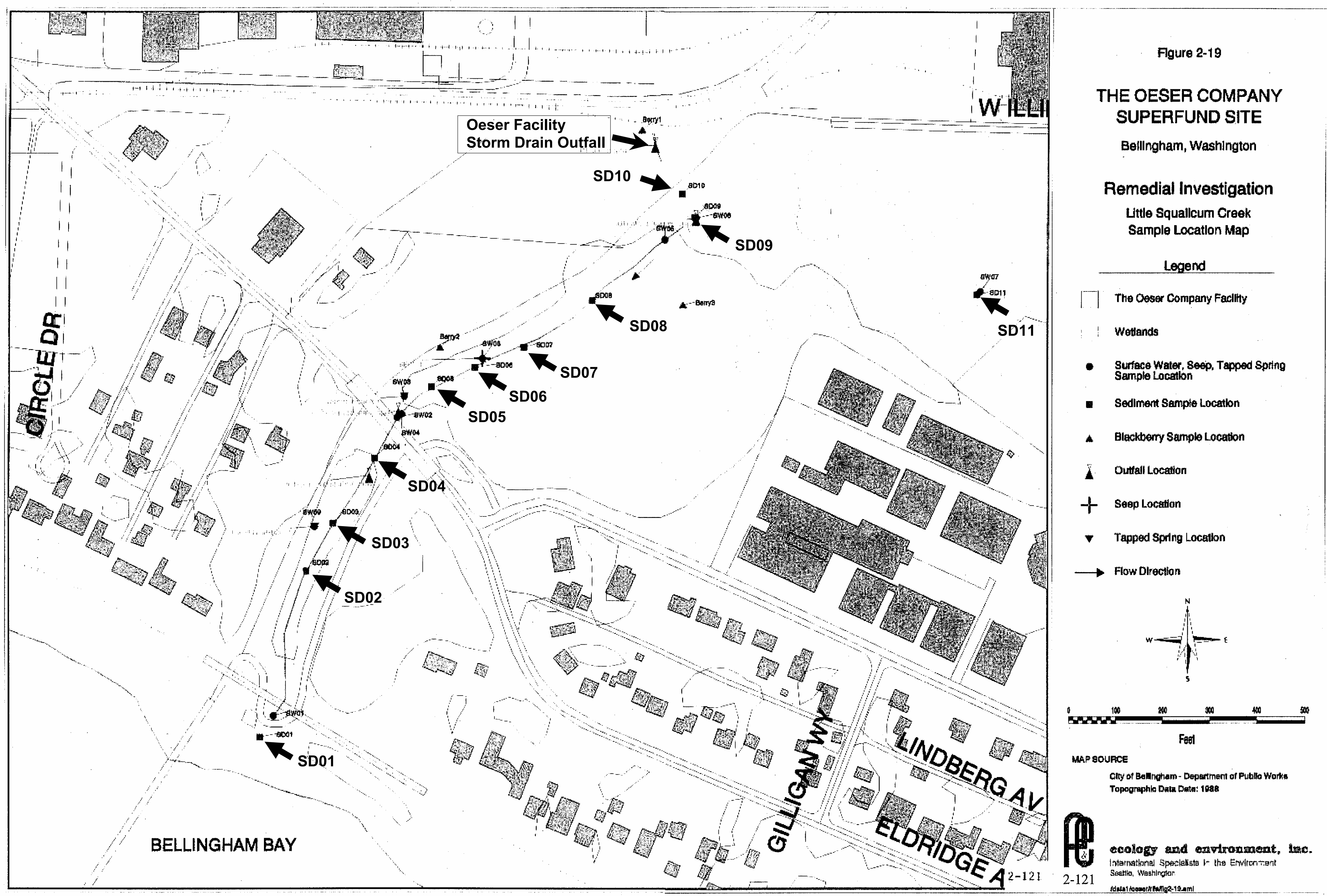

Figure 3. Locations of Little Squalicum Creek Sediments Sampled (arrows) During the Oeser Company Superfund Site Remedial Investigation. Source: Ecology and Environment, Inc., 2001.

Table 1. PAH Concentrations in Little Squalicum Creek Sediments (adapted from Table 4-119 in Ecology and Environment, Inc., 2001).

	SQS	SD01	SD02	SD03	SD04	SD05	SD06	SD07	SD08	SD09	SD10	SD11
TOC (mg/kg)	--	0.3%	1.3%	0.75%	0.5%	1.8%	11%	0.7%	0.5%	1.3%	1.5%	12%
Pentachlorophenol (mg/kg)	0.360	0.0037	0.033	**2**	0.024	0.056	**0.46**	0.015	0.16	**1.1**	**2.9**	ND
PAH (mg/kg OC)[b]												
Acenaphthene	16	ND[c]	0.9	**19**	0.7	1.6	0.7	1.5	2.6	0.4	1.2	ND
Benzo(a)anthracene	110	1.1	**285**	7.9	4.2	6.7	16	19.4	69	5.6	87	ND
Benzo(a)pyrene	99	1.5	**185**	12	8.1	19	13	38.9	**159**	5.5	**140**	ND
Benzo(g,h,i)perylene	31	1.1	**32**	5.6	1.9	8.9	3.6	15.3	**56**	ND	**58**	ND
Chrysene	110	3.5	**639**	25	9.8	13	20	36.1	**135**	9.2	**207**	ND
Indeno(1,2,3-cd)pyrene	34	0.8	**37**	6.4	4.6	10	4.5	16.7	**69**	ND	**64**	ND
LPAH[d]	370	0.9	**588**	**2,700**	299	168	26	**620**	**2,400**	94	**1,100**	0.2
HPAH[e]	960	**1,300**	**40,000**	**3,500**	**2,800**	**2,000**	420	**10,000**	**53,000**	905	**20,000**	0.1

[a] Marine Sediment Quality Standard. Exceedances of the SQS are shown in **bold**.
[b] TOC normalized (mg/kg organic carbon). Only PAH analytes for which one or more samples exceeded the SQS are shown.
[c] ND = not detected.
[d] "Low molecular weight polynuclear aromatic hydrocarbons" as defined in WAC 173-204-320.
[e] "High molecular weight polynuclear aromatic hydrocarbons" as defined in WAC 173-204-320.

Project Description

This investigation will evaluate sediments from selected locations in Little Squalicum Creek for contamination and toxicity to aquatic life. The City of Bellingham has requested assistance from Ecology in its attempt to help identify and address uncertainties and data gaps regarding potential contaminants associated with the Little Squalicum Creek sediments. This investigation is therefore not intended to provide a complete or representative characterization of the sediments throughout Little Squalicum Creek. The objectives are:

- Evaluate sediments below the storm drain outfall of the Oeser Company's wood treating facility, between stations SD10 and SD08. This segment is of interest because of past releases of wood preservative chemicals from the facility, described in Ecology and Environment (2001).
- Evaluate sediments near the mouth of Little Squalicum Creek, between stations SD01 and SD02. This segment is of interest as a potential location where contaminants transported down the creek in past releases may have accumulated in the sediments.
- To the extent possible, compare results of this investigation with data from the previous study conducted as part of the Oeser Company Superfund Site Characterization (see discussion below under *Comparability*).
- Assist the City of Bellingham in identifying potential data gaps, and address uncertainties related to potential contamination of the sediments and associated alluvial soils to assess potential actions prior to redevelopment of the site into a recreational park.

Sediment samples collected in this investigation will be used to characterize organic chemical contamination, primarily PAHs and pentachlorophenol. These compounds are of particular interest based on results from the Oeser Company Superfund Site Characterization (Table 1) and the history of known past releases of wood preservative chemicals to the creek. The samples will also be evaluated for toxicity to aquatic life using three Ecology-approved freshwater sediment bioassays: 10-day amphipod (*Hyalella azteca*), 20-day midge (*Chironomus tentans*), and Microtox® (Ecology, 2003).

Responsibilities

Project Manager	Nigel Blakley (360) 407-6770	Project management, QAPP and report preparation
Project Assistant	Pete Adolphson (360) 407-7557	Assist in project planning, including selection of sampling sites and bioassays
Client (TCP-NWRO)	Lucy McInerney (425) 649-7272	Review QAPP and final report
TSU Supervisor	Dale Norton (360) 407-6765	Project review
WES Section Manager	Will Kendra (360) 407-6698	Project review
Manchester Laboratory	Stuart Magoon (360) 871-8801	Coordinate laboratory analysis
EIM Data Entry	Carolyn Lee (360) 407-6430	Data entry

Schedule

Field Sample Collection	September 2003
Laboratory Analysis Complete	December 2003
Draft Report	January 2003
Final Report	April 2004
EIM Data Entry	May 2004

Data Quality Objectives

Measurement Quality Objectives

Measurement quality objectives (MQOs) for accuracy (precision and bias) and required reporting limits for this investigation are shown in Table 2. The MQOs for accuracy, precision, and bias are in terms of maximum acceptable error.

Table 2. Measurement Quality Objectives for the Little Squalicum Creek Investigation.

Parameter	Accuracy (% deviation from true value)	Bias	Precision (RSD)	Required Reporting Limit
BNAs	65%	25%	20%	PAHs 45 ug/Kg, dry[a] Pentachlorophenol 360 ug/Kg, dry
TOC	NA[b]	NA[b]	14%	1%
Grain size	35%	5%	14%	1%

[a] Based on Sediment Quality Standard for acenaphthene and a TOC of 0.28%, reported in Ecology and Environment, Inc. (2001). Higher reporting limits are acceptable for some other PAHs.
[b] Evaluated qualitatively.

No criteria are included for the amphipod, midge, and Microtox bioassay data. The laboratory procedures for these bioassays include positive and negative controls, and they establish quantitative criteria for control data for bioassay results to be considered valid (PSEP, 1995; Ecology, 2003).

Representativeness

The primary objective for this study is to characterize surface sediments in two segments of Little Squalicum Creek with respect to chemistry (PAHs and pentachlorophenol) and toxicity to aquatic life. Sample data should be representative of sediments in these two segments to fill data gaps from previous studies.

In each segment, three sampling locations are considered adequate to characterize the instream sediments. A fourth location in each segment will characterize bank soil that appears to represent sediment deposited during high-flow periods. There are constraints on the selection of sampling locations, but these are not expected to invalidate the usefulness of the data for characterizing the stream segments. Constraints include access barriers at some stream locations (e.g., logs and dense vegetation across the streambed) and the lack of sediment fines in areas with a stony stream bed. Because of the latter problem, at some locations it may be necessary to subsample from small neighboring pockets of sediment fines and composite the subsamples to obtain sufficient material for analysis.

Samples will be collected, stored, and transported so as to minimize the potential for cross-contamination and to assure that they remain unchanged until analyzed. Where appropriate, sampling will be conducted from downstream to upstream to avoid contaminating the downstream samples.

Comparability

It is desirable but not essential to be able to compare data from this investigation with previous sampling results reported in the Oeser Company Superfund Site Characterization Report (Ecology and Environment, 2001). Sampling methods to be used in this investigation appear to be essentially the same as those described in the Site Characterization sampling plan (EPA, 1999). This investigation will use the same analytical method as that used previously (Base/Neutral/Acids analysis). The *Hyalella* bioassay used in the previous study is also included in this investigation.

Study Design

As indicated earlier in the Problem Statement, two stream segments were chosen for this study based on data gaps in the Oeser Company Superfund Site Characterization Report. Within these segments, sediment samples will be collected from the approximate locations shown in Figure 4 (see descriptions in Table 3). These locations were chosen to be distributed along the stream segment within the constraints imposed by streambed characteristics (e.g., stony areas lacking fines preclude sampling) and access barriers (e.g., downed logs and dense vegetation). One station (LSC05) is located in a pond above Little Squalicum Creek and serves as a reference station. For each sample, TOC will be measured for use in normalizing PAH concentrations. Grain size measurements will be used to characterize the sample composition for comparison with other samples from different areas of the stream.

Table 3. Sample Stations.

Station	Location
Sediment	
LSC01	Downstream segment of Little Squalicum Creek
LSC02	Downstream segment of Little Squalicum Creek
LSC03	Downstream segment of Little Squalicum Creek
LSC04	Headwater segment of Little Squalicum Creek
LSC05	Reference station. Pond near Bellingham Technical College parking lot (Pool 1 in Figure 2). Corresponds to SD-11 station used as background in previous study – see Figure 3 and data in Table 1.
LSC06	Channel from Oeser Outfall to Little Squalicum Creek
Soil	
LSCS1	Right bank, downstream segment of Little Squalicum Creek
LSCS2	Right bank, downstream segment of Little Squalicum Creek

Field Procedures

A sample will be collected at each of the sampling points shown in Figure 4. Sampling locations will be measured from reference points using a laser rangefinder, and approximate coordinates will be determined from GPS readings. Stakes and survey flagging will be used to mark each sampling location

At each sampling location, the top 10 cm of sediment (or soil) will be removed using a stainless steel scoop, placed in a dedicated precleaned stainless steel mixing bowl, homogenized, and then transferred to containers listed in Table 4. Large pieces of organic material and gravel will be removed from the samples. Although discrete samples will be collected where possible, at some stations it may be necessary to composite subsamples from neighboring pockets of sediment to obtained the required volumes. All samples will be placed in coolers with ice immediately after collection for transportation to Ecology Headquarters, where the samples will be stored at 4º C. The samples will be transported to the Ecology Manchester Environmental Laboratory (MEL) within five days of collection. Storage temperatures and holding time requirements are listed in Table 4. Chain-of-custody will be maintained throughout the study, including archived samples.

Stainless steel scoops and mixing bowls will be precleaned with Liquinox detergent, rinsed with deionized water, 10% nitric acid, and then methanol. After cleaning, the scoops and bowls will be wrapped in aluminum foil.

Table 4. Containers, Preservatives, and Holding times for Sediment and Soil Samples.

Analyte	Container	Preservation Techniques	Holding Time	Source[1]
TOC	2 oz glass jar	Cool to 4°C	14 days	MEL
Grain Size	8 oz plastic jar	Cool to 4°C	6 months	MEL
BNAs	8 oz glass jar[2]	Cool to 4°C	14 days	MEL
Amphipod Bioassay (*Hyalella* 10-day)	1 liter glass jar	Cool to 4°C	2 weeks	SAPA
Midge Bioassay (*Chironomus* 20-day)	1 liter glass jar	Cool to 4°C	2 weeks	SAPA
Microtox Bioassay	0.5 liter glass jar	Cool to 4°C	2 weeks	SAPA

[1] Sources. See Reference section for full citation.

MEL Manchester Lab Users Manual, Sixth Edition – July 2002

PSEP Recommended Guidelines for Sampling Marine Sediment, Water Column, and Tissue in Puget Sound. PSEP, 1997.

SAPA Sediment Sampling and Analysis Plan Appendix. Washington Department of Ecology, 2003. (Tables 9 and 10).

[2] Organic free with Teflon lined lids, with certificate of analysis.

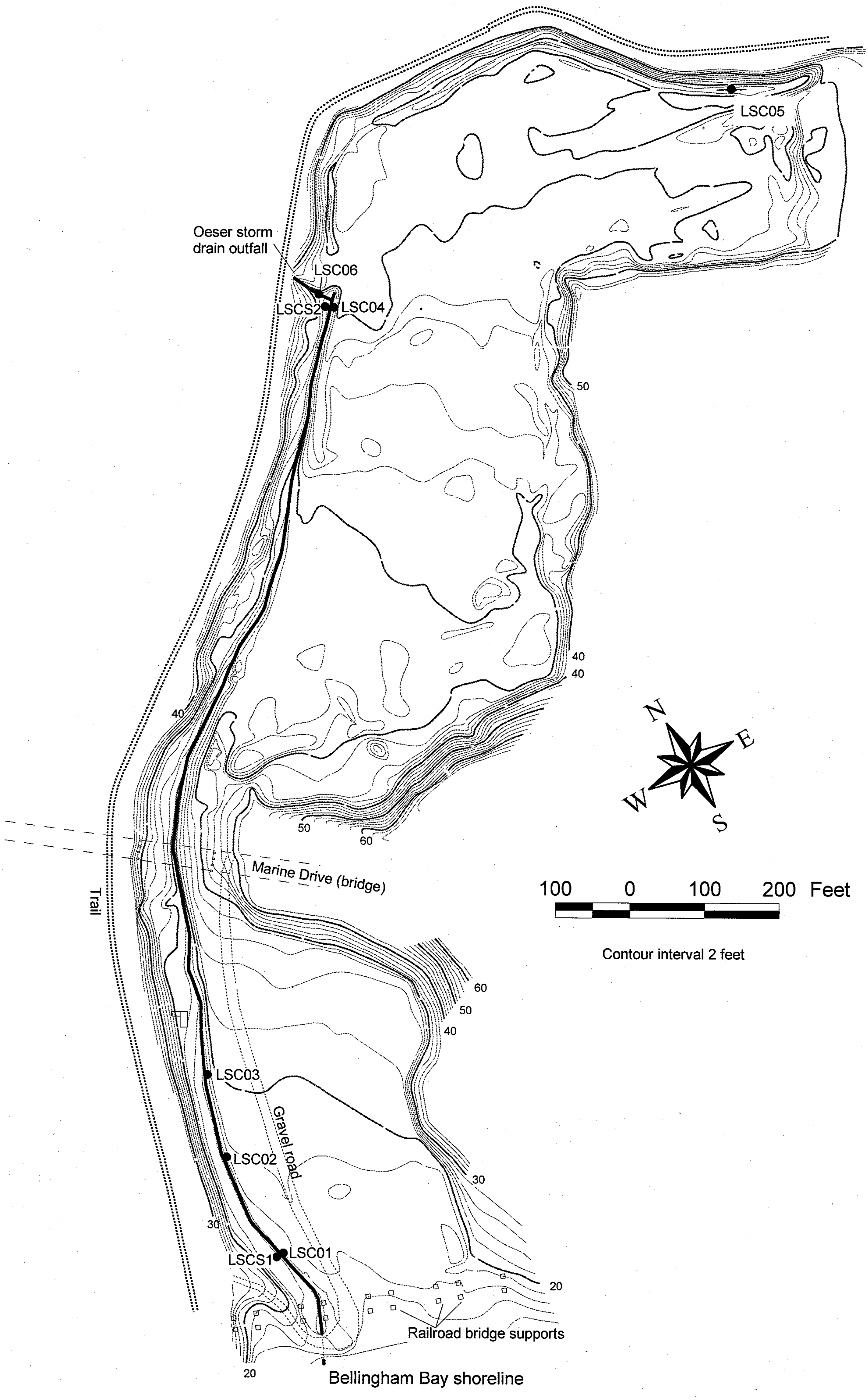

Figure 4. Approximate locations of proposed sediment sampling locations in Little Squalicum Creek (LSC01-LSC06) and soil (stream bank) locations (LSCS1-LSCS2).

Laboratory Procedures

Analytical methods and laboratory reporting limits for analysis of samples from this project are shown in Table 5. BNA and TOC analyses will be conducted at the Manchester Environmental Laboratory (MEL). Grain size analysis and bioassays will be conducted at contract laboratories selected by MEL. Estimated analytical costs for this investigation are shown in Table 6.

Table 5. Analytical Methods and Lab Reporting Limits for This Study.

Analyte	Method	Reference	Lab Reporting Limit	Laboratory
Total Organic Carbon	Combustion/CO^2 Measurement @ 70°C (9060)	PSEP, 1997	0.1%	MEL
Grain Size	Sieve and Pipet	PSEP, 1986	0.1%	Contractor
BNAs	Capillary GC/MS, EPA 8270	EPA, 1996	Varies[1]	MEL
Amphipod Bioassay (*Hyalella* 10-day)	ASTM E-1706 and Method 100.1	EPA, 2000	NA	Contractor
Midge Bioassay (*Chironomus* 20-day)	Method 100.5	EPA, 2000	NA	Contractor
Microtox Bioassay	Ecology Protocol	Ecology, 2003	NA	Contractor

[1] Varies with compound and sample characteristics. MEL can achieve the required reporting limits for PAHs and pentachlorophenol shown in Table 2 (Huntamer, 2003).

NA = Not applicable

Table 6. Analytical Costs for the Little Squalicum Creek Screening Level Assessment[1]

Analysis	Cost per sample	Number of samples	Duplicate samples	Matrix spikes	Total analyses	Cost Subtotals
Bioassays						
Chironomus 20-day	$1,500	6			6	$9,000
Hyalella 10-day	$725	6			6	$4,350
Microtox	$200	6			6	$1,200
						$14,550
				25% contract fee		$3,638
						$18,188
Chemistry[2]						
BNAs	$300	9		1 MS & 1 MSD	11	$3,300
TOC	$39	9	1		10	$390
Grain size	$100	9	2		11	$1,100
						$4,790
				25% contract fee (grain size)		$275
						$5,065
					Total cost	**$23,253**

[1] Costs include 50% discount for Manchester Lab.

[2] Number of samples (9) includes 6 sediment stations, 1 field duplicate, and 2 stream bank stations. Bioassay testing applies only to the 6 sediment stations.

Quality Control Procedures

Field Quality Control

For each sampling location, a set of stainless steel implements to be used to collect and manipulate the sample will be cleaned by washing with Liquinox detergent and followed by sequential rinses with tap water, 10% nitric acid, and deionized water. The equipment will then be air dried and wrapped in aluminum foil.

Nitrile gloves will be worn when handling samples. Where there appears to be a potential for downstream cross-contamination, sampling will be conducted from downstream to upstream to avoid contaminating the downstream samples.

Homogenized samples will be placed in glass jars with Teflon lid liners and cleaned to EPA QA/QC specifications (EPA, 1990). All samples will be placed in coolers with ice immediately after collection for transportation to Ecology Headquarters, where the samples will be stored at 4º C. The samples will be transported in coolers with ice to MEL within five days of collection. Storage temperature and holding time requirements listed in Table 4 will be met. Chain-of-custody will be maintained throughout the study.

One sediment sample will be split in the field to estimate sampling precision. The split sample will be submitted as a blind field duplicate for analysis. A "background" reference station is also included in the sampling design. This station (LSC05), located in a pond near the Bellingham Technical College, served as a background station in the previous study (Station SD-11 in Figure 3). PAH and pentachlorophenol concentrations were below detection limits at this station (Table 1).

Laboratory Quality Control

Quality control procedures should conform to requirements provided by MEL (MEL, 2001) and the Ecology Sediment Management Unit (Ecology, 2003; see Tables 11, 13 and 15). Analysis requirements are shown in Table 7.

Table 7: Quality Control Analysis Requirements

Analyte	Method Blanks	Lab Duplicates	Matrix Spikes	Lab Control Samples
TOC	NA	≤28% RPD	NA	Within control limits
Grain Size	NA	≤14% RPD	NA	Within control limits
BNAs	NA	≤28% RPD	Within control limits	Within control limits

Control limits will be specified in the lab's report of the results of analyses.
NA = Not applicable.

Data Review, Verification, and Validation

MEL will review the Quality Assurance (QA) Project Plan and all of the sample and quality control data. MEL is responsible for verifying the data and providing a verification report, and the project lead is responsible for validating the data. Data validation involves detailed examination of the complete data package using professional judgement to determine whether the procedures in the methods, standard operating procedures, and QA Project Plan were followed (Ecology, 2001).

MEL reviews will be sent to the project lead in the form of case narratives and will include an assessment of the laboratories' performance in meeting the conditions and requirements set forth in this sampling plan.

On receipt of the bioassay data, the project lead will review the results for completeness, reasonableness, and usability. The bioassay data will be reviewed to assure that the methods and test conditions were followed and that results on negative controls and reference toxicants were acceptable.

The project lead will provide a draft report of the study results to the client in December 2003. At a minimum, the final report will include the following:

- A study area map showing the sampling sites.
- Latitude and longitude and other information describing the sampling sites.
- Descriptions of field and laboratory methods.
- A data quality synopsis and discussion of the significance of any analytical problems.
- Summary tables of biological and chemical data.
- An evaluation of significant findings. Analyte concentrations will be compared with marine Sediment Management Standards (WAC 173-204) and freshwater Apparent Effects Thresholds (Michelsen, 2003). Bioassay results will be compared with Sediment Quality Standards (WAC 173-204-315 and Ecology, 2003). A final report will be prepared following receipt of comments from the client and internal review by Ecology's Environmental Assessment Program. The final report goal is February 2004.

Data Quality Assessment

If the project data are complete and meet data quality requirements described above, they will be considered of acceptable quality for evaluating sediment quality in Little Squalicum Creek.

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