

# **PTI**

*ENVIRONMENTAL SERVICES*

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## **Puget Sound Dredged Disposal Analysis**

### **BASELINE SURVEY OF PHASE II DISPOSAL SITES**

**APPENDIX A**  
Sampling Cruise Safety Plan and  
Sampling & Analysis Procedures

For

**Washington Department of Ecology  
Olympia, Washington**

June 1989

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**PUGET SOUND DREDGED DISPOSAL ANALYSIS  
BASELINE SURVEY OF PHASE II DISPOSAL SITES**

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For

Washington Department of Ecology  
Olympia, Washington 98504-8711

Ecology Contract C0089128  
PTI Contract C878-04

June 1989

## APPENDIX A

### SAMPLING CRUISE SAFETY PLAN AND SAMPLING AND ANALYSIS PROCEDURES

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## SAFETY PLAN

Safety hazards are associated with the equipment and supplies in use, as well as the general rigors of work at sea. The purpose of a cruise safety plan is to identify the potential hazards, institute procedures for minimizing them, prepare the proper responses in case of accident and injury, and make this information known to all shipboard personnel. The following plan was used during the Phase II baseline study.

### SAFETY PROCEDURES

To ensure safe and efficient shipboard operations, the chief scientist was designated the safety officer responsible for all shipboard operations, including evaluation of hazardous conditions, ensuring compliance with safety precautions, and suspension of shipboard operations if necessary. A halt to or suspension of operations could also be dictated by the vessel master.

#### Hazards

Hazards encountered during sampling were generally classified as either chemical or physical. Chemical hazards were associated with the materials used to decontaminate sampling gear and preserve samples. Physical hazards were associated with the gear and the conditions of work at sea.

During field operations, several heavy pieces of equipment were used to collect field samples. This gear included a REMOTS® camera, a mini-Soutar box corer (0.06-m<sup>2</sup>), and a dual van Veen grab sampler (0.1-m<sup>2</sup>).

**Chemical Hazards**—No need for extraordinary precautions (e.g., Tyvek suits, respirators) was anticipated for PSDDA Phase II sampling. Stations to be sampled during the survey were not expected to contain hazardous materials. During future field operations, if evidence of contaminated sediment is observed (by odor, color, petroleum product presence, or excessive organic enrichment), the sample should be discarded and the sampling plan should be reassessed. This reassessment may involve instituting suitable protective measures for the crew.

Ten percent buffered formalin was used to fix benthic samples. Formalin is a colorless liquid with a pungent odor. Symptoms of exposure to formalin fumes or liquid include irritation of the eyes, throat, and nose. Contact with formalin was not expected to be a health concern during normal operations. During the baseline survey, formalin was stored in a tip-and-measure dispenser that allowed easy measure of the desired volume of formalin. To ensure adequate ventilation, all formalin was used and stored in the open and not permitted in the cabin or below deck.

A 1N solution of zinc acetate was used to preserve sediment samples for sulfide analysis. Zinc acetate was dispensed from a narrow-mouth bottle with a plastic eyedropper and was used only on deck to provide adequate ventilation and reduce the severity of any spills.

Acetone and methylene chloride were used to decontaminate sampling equipment. Both are clear, colorless, volatile organic solvents with strong odors. Methylene chloride is a suspected carcinogen. Acetone and methylene chloride were used only on the open deck, and personnel were required to wear protective gloves whenever handling these liquids.

**Physical Hazards**—Gear deployment and retrieval present hazards because of the heavy weight of the sampling gear, its suspension above the deck, and the risk of accidental and premature closure. Safety pins were required to be in place on all pieces of gear whenever they were inboard of the vessel rail. Setting the triggering mechanism was always performed when the equipment was resting on a stable surface. Each safety pin for the gear used in the PSDDA Phase II baseline survey was equipped with a ring by which the pin may be grasped for removal. If the gear or winch slips while a finger is inserted through this ring, the finger may be severed. Consequently, personnel were required to remove the safety pin only by grasping the outer edge of the ring between finger and thumb.

During retrieval, at least one crew member was required to watch for the appearance of the sampling gear and alert the winch operator. Failure to observe the gear and stop the winch can lead to breakage of the cable, loss of the gear, and possible injury from either the falling gear or the snapped cable end.

After repeated use, individual strands of a hydrowire may break and project from the cable. Sampling personnel were reminded to never contact the moving cable unless protected by work gloves. On a periodic basis over the length of the sampling cruise, the chief scientist inspected the cable for wear, especially where the wire was attached to the sampling gear.

The winch drum, the blocks, and the area between the gear and the rail, deck, or other large equipment all represent significant pinching and crushing hazards. Personnel were instructed to keep their hands, feet, and clothing clear of these points.

Lines, hoses, hatch covers, and mud on the deck all present tripping, slipping, and falling hazards. Every crew member was instructed to take care to keep the working surfaces of the deck clear and clean by coiling hoses and lines and rinsing accumulations of mud from the deck. Awareness of the positions of hatch covers and other gear in use should be maintained at all times.

A drowning hazard exists during work at sea. Sampling operations do not require any personnel to work on or over the rail of the vessel. The drowning hazard is therefore associated with either tripping (discussed above) or conditions of excessively rough weather. Life vests were available for all personnel working on the rear deck and were donned when directed by the chief scientist/safety officer or the vessel operator. The vessel was also equipped with throwable life rings, and each crew member was briefed on their use and storage location.

Fatigue presents a hazard when working at sea. It can be compounded by the motion of the vessel, exposure, or heat stress. Personnel monitored their own condition and capabilities and were responsible for taking appropriate measures (discussed below) to relieve fatigue, exposure, or heat stress. The chief scientist/safety officer could also direct any member of the crew to cease working.

## **Safe Work Practices**

Precautions taken in the handling of chemicals included restricting their use to the deck, storing and dispensing them from narrow-mouth bottles, and exercising care in their use. When adding formalin to a benthic sample jar, the sample jar was placed on a stable surface so that two hands were free to manipulate the dispensing jug. Solvent rinsing of sampling gear was conducted on deck over an open basin of water, so that the excess solvent was not spilled on the deck and so that the vapors were not trapped. Gloves were always worn when handling solvents. Attention was given to the sea state and presence of wakes or other disturbances that could lead to spillage.

All crew members were required to wear hardhats when working on the rear deck. Work gloves were available but not required (latex gloves were required when using acetone or methylene chloride). Flotation vests were provided and could be worn by crew at their own discretion. They may be required by the chief scientist/safety officer or vessel operator if weather or work conditions warrant.

Each crew member was expected to bring clothing appropriate to the weather to minimize the hazards of exposure and heat stress. Boots and rain gear or other waterproof clothing were recommended, particularly when sieving benthic samples.

During gear deployment and retrieval, personnel paid close attention to the position of the gear, the motion of the boat, obstructions on the deck that could impede their own mobility, and actual or potential fouling of the gear. Hands and feet must never be placed underneath sampling gear. Safety pins were removed only when the gear was at or just over the rail.

Weather conditions were monitored by the chief scientist and vessel operator. The vessel was supplied with life rings or other emergency flotation equipment and fire extinguishers. Food and access to shelter (the vessel's cabin) were provided to the sampling crew.

The number of excess personnel (observers or guests) was minimized. Only one extra person in addition to the sampling crew of four (i.e., chief scientist, navigator, two sampling crew members) was ordinarily allowed onboard each day.

## **Emergency Planning**

If an emergency or accident had occurred during sampling, the chief scientist/safety officer and vessel operator would have been responsible for determining the appropriate response. This includes an assessment of the severity of the incident and, if appropriate, contacting the appropriate personnel for emergency assistance. All crew members were instructed in the use of the cellular telephone and marine radio in the event that the vessel operator and chief scientist were incapacitated. The vessel operator was responsible for moving the boat to an appropriate position for receipt of emergency aid, if necessary. A basic first-aid kit was kept onboard for the treatment of minor cuts or scrapes. All accidents were required to be reported to the chief scientist and recorded in the cruise log.

Contact information for local emergency services, hospitals, and ambulance services was aboard the boat in a location known to and accessible to all personnel.

## **Dissemination of Information**

All sampling crew and observers were aware of potential hazards, safe work practices, and emergency response. A number of planning measures were undertaken to ensure appropriate response to emergency situations, including the following:

- A precruise briefing of cruise members was conducted prior to initiation of sampling activity. The meeting included all personnel scheduled to participate in the sampling cruises. The meeting provided an overview of safety and cruise plans.
- Each crew member was given a copy of this safety plan for final review prior to participating on the cruise.
- Copies of this safety plan were made available on the vessel and at the shore support station. A copy of emergency contacts was posted near the ship-to-shore radio and kept with the cellular phone.
- Prior to the start of each day's sampling activities, the chief scientist checked to ensure that all safety equipment was onboard.
- The chief scientist briefed all guest observers onboard concerning the safety requirements and procedures to be adhered to during sampling operations.

During the baseline survey, contact information was provided to aid the chief scientist in making contact with the appropriate emergency personnel. Table A-1 lists the emergency contact information used during the Phase II baseline survey. Prior to conducting future field cruises at these sites, the emergency contact information listed in Table A-1 must be verified.

**TABLE A-1. EMERGENCY CONTACT INFORMATION**

**Anderson/Ketron Site**

Hospitals

Lakewood General Hospital, 5702 - 100th Street S.W.	588-1711
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Emergency Medical Information and Transportation

Firecomm Emergency Dispatch	911
Pierce County Emergency Services	593-4797
Steilacoom Fire Department	581-0110
U.S. Coast Guard	858-9998
Washington State Patrol	593-2424

**Bellingham Bay**

Hospitals

St. Luke's General Hospital, 809 E Chestnut	734-8300
St. Joseph's Hospital, Squalicum Parkway	734-5400
Whatcom Medic I	911

Emergency Medical Information and Transportation

Bellingham Police Department, Emergency	911
Bellingham Fire Department	911
U.S. Coast Guard	1-800-592-9911

## SAMPLING AND ANALYSIS PROCEDURES

A variety of activities were conducted during the baseline cruise including the collection of sediment for chemical analysis and bioassays, and sieving of sediments to collect organisms for benthic abundance and tissue body burden analysis. In addition, sediment vertical profile samples were taken using the REMOTS® camera.

### STATION LOCATION AND IDENTIFICATION

The number, identity, and location of all sampling stations were established prior to field operations. The locations were plotted on nautical charts and the actual positions were picked off the charts as latitude and longitude.

Each station was identified by a five-character name or code. The first two characters identify the disposal site or reference area. The following site identification codes were used:

BB - Bellingham Bay  
AK - Anderson/Ketron site  
CR - Carr Inlet.

The third character identifies the sampling location. Stations included the disposal site target zone (Z), other stations within the disposal site (S), perimeter stations located at a distance 0.125 miles from the site boundary (P), transect stations located in the downcurrent direction from each site (T), benchmark stations located in the general vicinity of the sites (B), and reference stations located in uncontaminated areas of Puget Sound (R). The fourth and fifth characters make up a two-digit number used to discriminate between different stations of the same type. For example, perimeter Station number 10 in Bellingham Bay was identified as BBP10.

### VESSEL POSITIONING

To meet the goals of the PSDDA monitoring plan for disposal sites, precise positioning at station locations was required. Both absolute accuracy (e.g., ability to define position) and repeatable accuracy (e.g., ability to return to a sampling station time after time) were essential. To meet these positioning goals a microwave navigation system was used.

During the baseline survey, precise station positioning was achieved through the use of a Del Norte Technology, Inc. Trisponder® system with a Model 542 digital distance measuring unit (DDMU). This system depends upon accurately known positions for the shore stations at which transponders are placed. Three microwave transponders were used at each sampling site. The criteria for selecting transponder locations were as follows:

- At least two locations must form a line-of-sight angle of 30-150 degrees from each sampling station

- The coordinates of each location must be accurately known, either from a survey or by reference to an existing benchmark
- Locations should be as secure as possible to eliminate the need to remove and replace the transponders each day
- Locations with AC power available are preferable, to reduce the need for battery changes and the possibility of battery failure.

Transponders were placed at the selected locations at the beginning of operations in each bay. Transponder locations are described in the main body of this report. Power was supplied by a pair of 12-volt car batteries (in series) or AC power where available. Each transponder location was checked daily and batteries were replaced as necessary. The DDMU was calibrated with the transponder in use at the beginning of operations in each sampling area and each time a transponder was changed. At the beginning of each sampling day the calibration was checked by moving the vessel to a known position. The DDMU was checked and adjusted as necessary.

Prior to using the transponder for the baseline survey, the DDMU was calibrated in Port Gardner at a range whose length was known with a high degree of certainty. A Hewlett Packard Series 200 computer was used to acquire, display, and record data received by the DDMU. The software displayed the sampling area at a user-selected scale, showing the station and vessel positions and the range and bearing to the station at all times. This equipment was used to achieve consistent positioning of the vessel within 50 feet of the chosen location.

Distance measurements from three transponders were ordinarily used to locate the vessel. In some cases, signals from one shore station were interrupted or intermittent due to shadowing, range holes, or equipment malfunctions. Sampling was halted whenever accurate positioning could not be achieved using at least two shore stations. Shore support personnel were available at all times to inspect, adjust, and replace the transponders as necessary. Communication with shore personnel was maintained by radio.

In Carr Inlet, the level of station positioning accuracy was not as critical as that required at the disposal sites. At this site, a station position was established on the basis of Loran-C and radar ranges.

## **GEAR DEPLOYMENT**

Gear was deployed after the vessel was positioned appropriately relative to the sampling station (depending upon drift caused by wind and tidal currents). Sampling equipment was swung outboard, readied for triggering (safety pins removed), lowered to the water surface or a known distance above it, and the meter block was adjusted. The gear was then lowered to 5-10 meters from the bottom. If necessary, the vessel position was adjusted to within 50 feet of the station. The DDMU was attached to the boom just above the block so that no offset was required to determine the position of the sampling gear relative to the position indicated by the navigation system. Once within the station target (i.e., 50 feet of station coordinates), the vessel position was further adjusted, if required, so that a minimal wire angle (<5 degrees) was achieved. The gear was then lowered to the seafloor. Position (in feet, as generated by the DDMU, and in Loran-C), time, depth, and length of wire out were recorded at the moment gear reached the bottom.

Position was recorded by the computer on a magnetic disk and on paper and was entered by hand into the sampling logs as well. This procedure was repeated for each field replicate or composite sample taken at a station.

## SAMPLE IDENTIFICATION AND LABELING

Each sample and field replicate was assigned a unique identifier. The first five characters always consisted of the station ID, and the sixth was used to identify the type of sample, as follows:

- I - infauna
- C - sediment chemistry or bioassay
- A - bioaccumulation.

When field replicates were collected (i.e., for infauna samples), a dash (-) and the replicate number were appended to the sample ID. For example, a sediment sample taken for chemical testing from the fourth perimeter station in Bellingham Bay was identified as BBP04C.

Various types of containers were used to collect samples, as described in following sections. All samples were labeled uniformly. Included on the label was the name of the survey (PSDDA2), the sample ID, and the date of collection. Each container for chemical analysis also listed the chemicals to be analyzed for that sample. All labels were covered with clear waterproof tape to prevent damage or alteration.

## SAMPLE INFORMATION CONTROL

It was important throughout the baseline survey to maintain integrity of each sample from the time of collection to the point of data reporting. Proper recordkeeping and chain-of-custody procedures allowed the possession and handling of samples to be traced from collection to final disposition. The forms used to identify and catalog station and sample information were:

- **Station Log** (Figure A-1)--The station log was maintained by the navigation systems operator. At the time the sampling gear contacted the seafloor, all pertinent information was recorded in the station log.
- **Chain-of-Custody Record** (Figure A-2)--Each chain-of-custody record was made in triplicate and was completed by the chief scientist at the end of the sampling day. One copy was maintained by the chief scientist when samples were released to the chain-of-custody officer. One copy was retained by the chain-of-custody officer when samples were released to the testing laboratory. The final copy was returned by the laboratory with data results.

## COLLECTION OF SEDIMENT VERTICAL PROFILE PHOTOGRAPHS

Sediment profiles were collected at a matrix of stations spanning the site boundary in each basin. A REMOTS® camera system was equipped with a Model 3731 sediment-profile camera (Benthos, Inc.). The REMOTS® system was used in conjunction with a Hewlett Packard Series 200



**CHAIN OF CUSTODY RECORD**

<b>PROJECT</b>				<b>SAMPLERS:</b> <i>15 signature</i>						
LAB #	STATION	DATE	TIME	SAMPLE TYPE						REMARKS
				WATER	SEDIMENT	ISSUE	AIR	DIRT	OTHER	
<b>RELINQUISHED BY:</b> <i>15 signature</i>			<b>RECEIVED BY:</b> <i>15 signature</i>				<b>DATE/TIME</b>			
<b>RELINQUISHED BY:</b> <i>15 signature</i>			<b>RECEIVED BY:</b> <i>15 signature</i>				<b>DATE/TIME</b>			
<b>RELINQUISHED BY:</b> <i>15 signature</i>			<b>RECEIVED BY:</b> <i>15 signature</i>				<b>DATE/TIME</b>			
<b>RELINQUISHED BY:</b> <i>15 signature</i>			<b>REC'D BY MOBILE LAB FOR FIELD ANAL:</b> <i>15 signature</i>				<b>DATE/TIME</b>			
<b>DISPATCHED BY:</b> <i>15 signature</i>		<b>DATE/TIME</b>		<b>RECEIVED FOR LAB BY:</b> <i>15 signature</i>			<b>DATE/TIME</b>			
<b>METHOD OF SHIPMENT:</b>										

Distribution: Original - Accompanying Sherman  
 One Copy - Survey Coordinator Field Files

Figure A-2. Chain-of-custody record form used in baseline survey

computer running the SAIC integrated navigation and data acquisition system (INDAS). Image analysis of the REMOTS® camera photographs was carried out using a video digitizer and image analysis software.

The REMOTS® camera consists of a wedge-shaped prism with a front faceplate and a back mirror mounted at a 45-degree angle (Figure A-3). The camera and prism are retracted within the frame while on deck and are held by safety pins. When the gear is deployed, the pins are removed and the hydrowire tension keeps the camera retracted. When the frame contacts the seafloor, the release of tension allows the prism to penetrate the bottom. A piston ensures that the prism enters the bottom slowly and does not disturb the sediment-water interface. On impact, a trigger activates a 13-second time delay on the shutter release, giving the prism a chance to achieve maximum penetration before a picture is taken. The sediment photographed is directly against the faceplate so that the turbidity of the ambient seawater does not affect image quality. When the camera is raised, a wiper blade cleans the faceplate, the film is advanced by a motor drive, and the strobe is recharged. The depth of the vertical profile can be up to 20 cm depending on the substrate type.

### **Collection of Photographs**

At the beginning of each survey day, the time on the data logger that was mounted on the REMOTS® camera was synchronized with the computerized INDAS system. The time was automatically recorded on each frame of film exposed and was simultaneously recorded by INDAS with the vessel position at which each exposure was made. Test exposures were made on deck at the beginning and end of every roll of film to verify that all internal electronic systems on the camera were working to design specifications. Spare cameras and charged batteries were kept on hand at all times to ensure uninterrupted sample acquisition.

Triplicate profile photographs were taken at each station to ensure the capture of at least one good image. Upon retrieval of the camera system at each station, the frame counter was checked to make sure that the proper number of exposures was taken. In addition, a prism penetration depth indicator on the camera frame was checked to see that the optical prism had penetrated the bottom to a sufficient depth to acquire a profile image. If images were missed or insufficient penetration was achieved, the equipment was redeployed. All film was developed at the end of every survey day to verify successful data acquisition.

### **Acceptability of Photographs**

Strict control was maintained over film development temperatures, times, and chemicals to ensure consistent emulsion density. The negatives were then visually inspected under magnification to identify any images of insufficient quality for image analysis. If necessary, the appropriate station would be reoccupied on the next survey day. No redeployment of REMOTS® was necessary during the baseline survey, as it rarely is when sampling granular sediments in the range of medium sand to silt-clay.

### **Analysis of Photographs**

All physical and biological measurements were made directly from the film negatives. Negatives were used instead of positive prints to avoid changes in image density that can

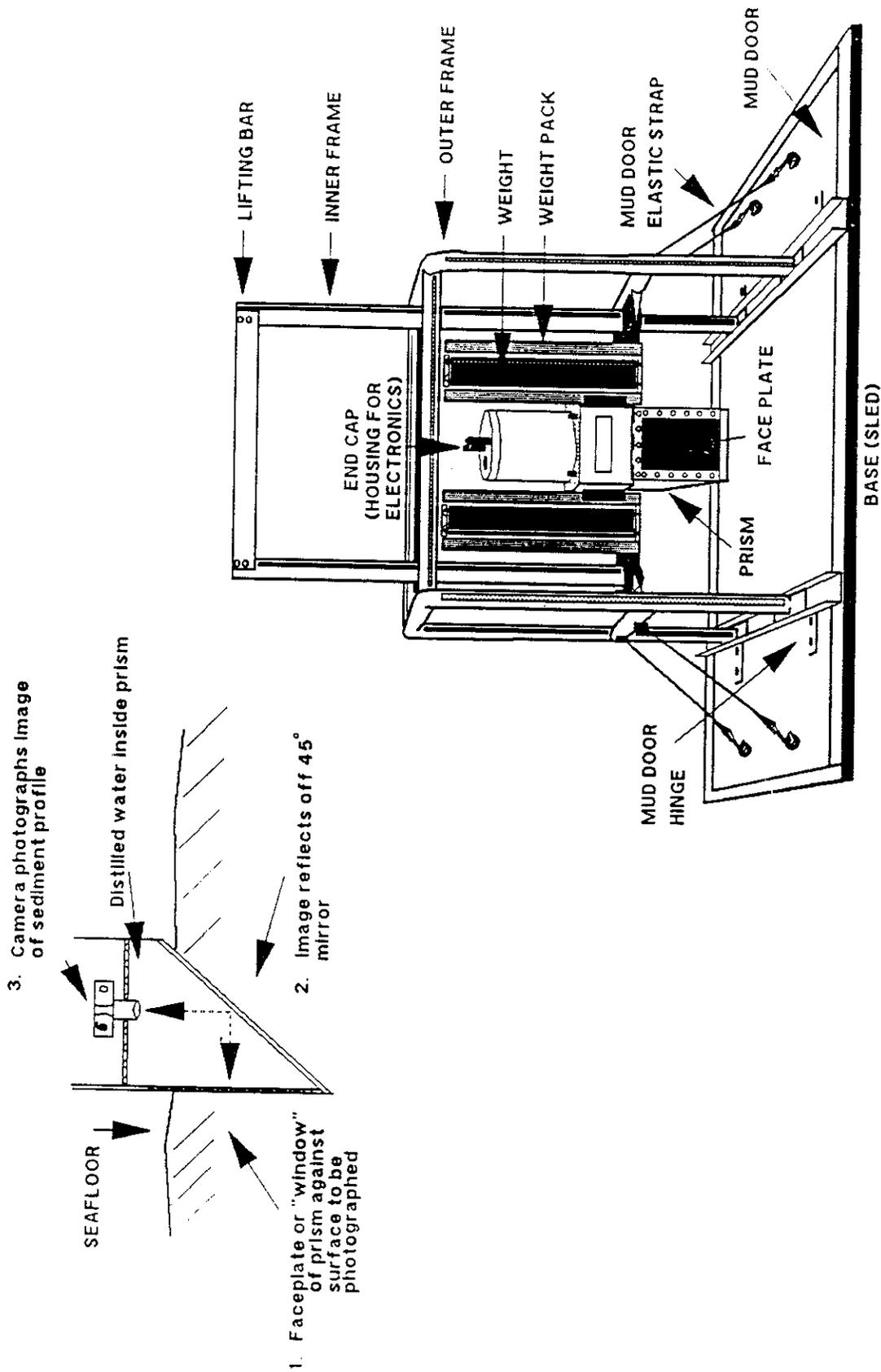


Figure A-3. Schematic illustration of REMOTS<sup>®</sup> camera system

accompany the printing of a positive image and the differential shrinkage of photographic paper. The computerized image analysis system used can discriminate up to 256 gray tones, allowing subtle features to be accurately digitized and measured

**Sediment Type Determination**—The mode and range of sediment grain size distribution was visually estimated from the photographs by overlaying a grain-size comparator at the same scale. This comparator was prepared by photographing a series of Udden-Wentworth size classes (equal to or less than coarse silt up to granule and larger sizes) through the REMOTS® camera. Seven grain-size classes are represented on the comparator: >4 phi, 4-3 phi, 3-2 phi, 2-1 phi, 1-0 phi, 0 to -1 phi, and <-1 phi. The lower limit of resolution of the photographic system is approximately 62  $\mu\text{m}$ , allowing recognition of grain sizes equal to or greater than coarse silt. The accuracy of this method has been verified by comparing REMOTS® estimates with sieve analyses (Germano 1983).

**Surface Boundary Roughness**—Surface boundary roughness was determined by measuring the vertical distance (parallel to the film border) between the highest and lowest points of the sediment-water interface. In addition, the origin of small-scale topographic relief was indicated when it was evident (physical or biogenic).

**Apparent Redox Potential Discontinuity Depth**—The apparent redox potential discontinuity depth was measured at the boundary between the more highly reflective oxygenated sediment and the underlying reduced sediment. Oxygenated sediment is more reflective because it contains particles coated with ferric hydroxide (an olive color when associated with particles). Anoxic sediments are grey to black. The area of oxidized sediment is determined by digitizing its unique reflectance value. The area is measured to scale and divided by the prism window width to obtain a mean depth for the apparent relative percent difference.

**Infauna Successional Stage**—The successional stage of the benthic infauna was determined by examining the REMOTS® images for the presence of invertebrates belonging to specific functional types. Depending upon the evidence of organisms belonging to certain types, the infauna community is classified as either a Stage I, Stage II, or Stage III assemblage. Stage I assemblages are pioneers on disturbed habitat, usually consisting of dense aggregations of near-surface, tube-dwelling polychaetes or opportunistic bivalves. In the absence of further disturbance, these assemblages are eventually replaced by infauna deposit feeders such as shallow-dwelling bivalves and tubicolous amphipods (Stage II). Stage III taxa are typically found in low-disturbance regimes. Many of these infauna species feed in a head-down orientation, resulting in distinctive feeding voids. Diagnostic features of feeding voids include a generally semicircular shape with a flat bottom and arched roof, and a coarser grain size overlying the floor. Stages I and III are easily recognized in REMOTS® images by the presence of dense assemblages of near-surface polychaetes or the presence of subsurface feeding voids. Both types of assemblages may be present in one image

## COLLECTION OF SEDIMENT CHEMISTRY AND BIOASSAY SAMPLES

Chemistry and bioassay samples were collected with a pair of 0.1- $\text{m}^2$  van Veen grab samplers welded together. The gear was specially constructed for Ecology by Kahl Scientific. An extended

bridle was attached so that the samplers would close simultaneously as they were pulled out of the sediment. Separate safety pins were provided for each grab sampler.

Sediment chemistry and bioassay samples were collected from the same composite, which was composed of material collected from a minimum of six grabs (a minimum of three casts). The only exception was in collection for volatile organic compounds and sulfide samples, which were collected from one of the six grabs prior to sediment removal from the sampler. Stations within the disposal site were sampled to a depth of 10 cm. Other stations were sampled to a depth of 2 cm. Additional grabs were taken whenever sufficient material could not be obtained from the first six grabs (e.g., at benchmark stations where sediments for bioassays were collected).

Approximately 250 grams of sediment were collected at each station for chemical analysis. This amount was made up of the sum of the six grabs. In future sampling at the PSDDA disposal sites, sample sizes should not be smaller than 150 grams, and they may be larger than 250 grams to ensure a sufficient sample for analysis. One liter of sediment was collected at each station for conducting bioassays.

### **Sample Acceptability**

Material collected in the van Veen grab sampler was evaluated for acceptability using the criteria set forth in the PSEP (1986) protocols. Each grab was evaluated separately. Acceptability was evaluated on the basis of the following criteria:

- The sampler was not overfilled
- Overlying water was present over the sample
- The overlying water was not excessively turbid
- The sediment surface was relatively undisturbed
- The depth of penetration was at least 2 cm below the depth of the sample to be taken.

The chief scientist and quality assurance officer onboard evaluated all samples collected. If a grab failed to meet any of the criteria, a sample was not collected from that grab.

### **Sample Collection**

After a grab was judged acceptable, the overlying water was siphoned off and the sample collected according to PSEP protocols. Two stainless steel spatulas and a stainless steel ruler were used to collect the samples from the van Veen grab sampler. The ruler was used to ensure that all samples were collected from the same depth. Sediment touching the sides of the van Veen grab sampler was not collected, and approximately equal volumes were collected from both halves of each grab.

Sediment for analysis of volatile organic compounds was collected and transferred directly to sample jars before any sediment was removed for compositing. All grab samples forming a composite were mixed in a stainless steel bowl until homogeneity in texture and color was achieved.

Sediment was then transferred with a stainless steel spoon directly to the appropriate sample containers.

### **Sample Handling**

All sample containers were prepared according to PSEP protocols prior to field operations. The type of container used to store material for each type of analysis is shown in Table A-2.

All samples were placed on ice immediately after sample jars were filled. Tributyltin samples were frozen within 24 hours of sample collection. Chain-of-custody forms were completed onboard at the end of each day and were shipped with the samples to the analytical laboratories. Samples for volatile organic compounds, semivolatile organic compounds, pesticides, and PCB analyses were hand-delivered to Analytical Research Inc. in Seattle, Washington. Samples for bioassay analyses were transported by courier to E.V.S. Consultants, Ltd. in Vancouver, British Columbia. Samples for metals and conventional analyses and archive samples were transported by bus to Columbia Analytical Services, Inc. in Longview, Washington. Samples for tributyltin analyses were transported by bus to Battelle Northwest Laboratory in Sequim, Washington.

All samples were delivered to the laboratories on ice. Samples traveling by courier or bus were packed in bubble-wrap plastic to prevent breakage, and chain-of-custody seals were placed across the cooler lids. Chain-of-custody forms, enclosed with the samples traveling by courier or bus, were signed at the laboratory and returned to the shipper. A packing list also accompanied each shipment to the analytical laboratories. The packing list enumerated all the samples contained in the shipment and specified which analyses were to be conducted on each sample.

### **Sediment Chemistry**

Chemical analyses of sediment samples were performed for all PSDDA chemicals of concern and tributyltin (Table A-3). Concentrations of organic compounds were determined following modified EPA Contract Laboratory Program (CLP) protocols (PSEP 1986). Concentrations of metals were determined using a strong acid digestion extraction technique. Analysis of all conventional variables except ammonia followed the PSEP protocols (PSEP 1986). The technique used for the extraction of ammonia is described by Plumb (1981).

The following sections provide an overview of the techniques and protocols used in chemical analysis of sediments collected during the Phase II baseline survey. More detailed descriptions of methods employed in this study are included in the QA/QC reports (Appendix D).

TABLE A-2. CONTAINER TYPES USED FOR COLLECTING  
 SAMPLES DURING THE PHASE II BASELINE CRUISE

Analysis	Container
Tributyltin	8-ounce glass jars with Teflon-lined lids (acid and solvent rinsed)
Metals Grain-size Total solids Total volatile solids Total organic carbon Ammonia	16-ounce polyethylene jars (rinsed, acid-soaked, and rinsed 3 times with metal-free water)
Total sulfides	4-ounce polyethylene jars [preserved in the field with 2N zinc acetate (approximately 5 mL per 30 grams of sediment)]
Volatile organics	2-ounce glass sample containers with Teflon-lined lids (detergent-washed, rinsed, fired at 450° C, filled in field to leave no headspace)
Semivolatile organic compounds Pesticides Polychlorinated biphenyls Archive sample	16-ounce glass jars with Teflon-lined lids (rinsed and fired at 450° C)
Bioassay	32-ounce glass jars with Teflon-lined lids (rinsed, fired at 450° C, filled in field to leave no headspace)

TABLE A-3. PSDDA CHEMICALS OF CONCERN

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<b>Metals</b>		
antimony	copper	nickel
arsenic	lead	silver
cadmium	mercury	zinc

<b>Phenol and Substituted Phenols</b>	
phenol	2,4-dimethylphenol
2-methylphenol	pentachlorophenol
4-methylphenol	

**Low Molecular Weight Polycyclic Aromatic Hydrocarbons (LPAH)**

naphthalene	fluorene
2-methylnaphthalene	phenanthrene
acenaphthylene	anthracene
acenaphthene	

**High Molecular Weight Polycyclic Aromatic Hydrocarbons (HPAH)**

fluoranthene	benzo(a)pyrene
pyrene	indeno(1,2,3-c,d)pyrene
benz(a)anthracene	dibenz(a,h)anthracene
chrysene	benzo(g,h,i)perylene
benzofluoranthenes (b and k)	

**Chlorinated Benzenes**

1,2-dichlorobenzene	1,2,4-trichlorobenzene
1,3-dichlorobenzene	hexachlorobenzene (HCB)
1,4-dichlorobenzene	

**Polychlorinated Biphenyls**

total polychlorinated biphenyls (PCB)

**Chlorinated Aliphatic Hydrocarbons**

hexachlorobutadiene	hexachloroethane
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TABLE A-3. (Continued)

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Phthalate Esters	
dimethyl phthalate	butyl benzyl phthalate
diethyl phthalate	bis(2-ethylhexyl)phthalate
di-n-butyl phthalate	di-n-octyl phthalate

Miscellaneous Oxygenated Compounds	
benzyl alcohol	benzoic acid
dibenzofuran	

Organonitrogen Compounds	
N-nitrosodiphenylamine	

Pesticides	
total DDT (p,p'isomers)	aldrin
heptachlor	dieldrin
$\alpha$ -chlordane	$\gamma$ -hexachlorocyclohexane (lindane)

Volatile Organic Compounds	
trichloroethene	ethylbenzene
tetrachloroethene	total xylenes

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## Organic Analyses

The analysis of volatile compounds followed the EPA CLP protocol for heated purge-and-trap gas chromatography/mass spectrometry (GC/MS) that is recommended by PSEP. However, only the four PSDDA volatile compounds were analyzed (Table A-3). Detection limits for volatiles were typically <5 µg/kg dry weight.

The analysis of semivolatile compounds, including A/B/N extractables, PCB, and pesticides, followed modified EPA CLP protocols that were consistent with PSEP recommendations for analyses with relatively low detection limits. In particular, modifications included larger sample size (typically 50-100 grams dry weight), smaller final extract volume for GC/MS analyses, and as an added quality assurance measure, the use of all available stable isotope labeled surrogates for the A/B/N compounds listed in Table A-3, to monitor recovery in all samples. Separate sediment subsamples were used for A/B/N and pesticide/PCB extractions. Ultrasonic extraction was carried out as described by the CLP procedure. Gel permeation chromatography (GPC), an optional cleanup step under the CLP, was performed for all sediment A/B/N extracts to reduce interferences and attain the necessary detection limits. Elemental sulfur cleanup was performed as necessary to reduce interferences in sediment samples. Detection limits for organic compounds in sediment samples without substantial interferences were typically well within PSEP guidelines.

Pesticide/PCB analyses were conducted with a slightly modified version of the EPA CLP method. These analyses included extract cleanup by alumina column chromatography and, when necessary, elemental sulfur cleanup, followed by gas chromatography/electron capture detection (GC/ECD) analysis. GC/ECD quantification and confirmation analyses were conducted with fused silica capillary columns rather than the packed columns routinely used in the CLP. Calibration procedures were consistent with the EPA CLP, although additional multipoint PCB calibrations specified in the laboratory contract were not performed. In addition to the standard CLP surrogate, 4,4'-dibromooctafluorobiphenyl (DBOBF) was added to monitor recovery on a sample-by-sample basis. Attempts were made to confirm PCB and pesticides by GC/MS when the compounds were detected at sufficient concentration by GC/ECD. The A/B/N extract was used for these confirmation analyses.

## Metals Analyses

For metals of concern, specific analytical procedures have been recommended by PSEP (1986). In accordance with these procedures, each sediment was digested using the strong acid technique in the EPA CLP program. Metals in sediment digestates were determined by graphite furnace atomic absorption (GFAA) or direct flame atomic absorption (DFAA) spectrometry. Cold vapor atomic absorption (CVAA) was required for determination of mercury in sediments.

## Tributyltin Analyses

Protocols for tributyltin used in this study were based on procedures and recommendations discussed in the EPA Region 10 methods memorandum that resulted from a meeting of the Subcommittee on Organotin Analysis Methods held 25 September 1987.

Sediment samples to be used for tributyltin analyses were frozen (-20° C) within 24 hours of collection as recommended at the tributyltin workshop. Sample holding times prior to extraction

or analysis have not been established, but it was recommended that analyses be completed as soon as possible to prevent potential degradation of the sample.

Sediment samples were acidified and mixed with  $\text{Na}_2\text{SO}_4$  prior to extraction with tropolone/methylene chloride. The extracts were concentrated, exchanged into hexane, and treated with a Grignard reagent to form hexyl derivatives. Derivatized extracts were subjected to Florisil column chromatography cleanup and analyzed by gas chromatography/mass selective detector (GC/MSD).

A standard reference material (SRM) (SQ-1 sediment) spiked with roughly 100  $\mu\text{g}/\text{kg}$  dry weight tributyltin was analyzed. Tripropyl tin chloride was spiked in all samples as a surrogate to monitor recovery. Detection limits were approximately 10  $\mu\text{g}/\text{kg}$  dry weight (as tin).

**Conventional Variable Analyses**—PSEP protocols (PSEP 1986) were used for analysis of conventional variables in sediment samples except for ammonia, which is not addressed in the protocols. Total organic carbon was measured using a combustion technique at 950° C. Grain size was determined on oxidized samples (using hydrogen peroxide) by wet sieving followed by dry sieving of the gravel and sand fractions (gravel consists of <-1 phi material, but the sand fraction was sieved to 1 phi intervals from -1 to 4 phi). The silt-clay fraction was subdivided into total silt (>4 to 8 phi) and total clay (>8 phi) fractions, using a pipet technique.

Total sulfides were determined using chemical treatment and color absorbance on a spectrophotometer at 650  $\mu\text{m}$ . Both undistilled and distilled standards were used to verify the calibration of the method. Total solids were determined on samples dried at 103° C in tared containers. Total volatile solids were determined by ignition at 550° C. Ammonia analyses are not addressed by the PSEP protocols. Extraction was performed in accordance with Plumb (1981) (Method 1 using KCl). Determination of ammonia concentrations was performed using the method of standard additions with a specific ion electrode for 10-gram aliquots of selected samples.

### Sediment Bioassays

Three sediment bioassays were conducted on zone, reference, and benchmark samples. Bioassays used, which were identified in the PSDDA monitoring plan, were the 10-day acute toxicity amphipod bioassay using *Rhepoxynius abronius*, the 48-hour bivalve larvae bioassay using *Crassostrea gigas*, and the Microtox bioassay using the saline extraction method. In all cases, the testing procedures described by PSEP (1986) were followed.

The sediment bioassays were conducted within 14 days of sediment collection as recommended in the protocols. Five replicate assays were made for each bioassay type for each of the sediments tested. Amphipods used for testing were collected from West Beach on Whidbey Island, Washington. Bivalves used to produce larvae were field-collected from Vancouver Island, British Columbia. Freeze-dried bacteria used in the Microtox bioassay were obtained from Microbics Corporation in Carlsbad, California. Where required, sediment collected from West Beach was used as a control. Samples collected from Carr Inlet and Port Susan were used as negative controls. Sodium pentachlorophenate (NaPCP) was used as a negative control.

Quality control procedures and documentation required for all four bioassays included:

- Use of negative and positive controls for each bioassay type
- Use of reference test samples for each bioassay type
- Measurement of water quality parameters including pH, dissolved oxygen, temperature, and salinity
- Summary of any testing anomalies.

## COLLECTION OF BENTHIC SAMPLES

Benthic infauna were collected with a 0.06-m<sup>2</sup> mini-Soutar box corer loaned by EPA. This frame-mounted spade corer is equipped with removable weights and two safety pins. The weight of the corer drives it into the bottom, and blades seal the bottom of the box corer upon retrieval. Five field replicates were collected at each benthic infauna station. A minimum depth of 10 cm was sampled at all benthic infauna stations.

PSEP (1987) protocols for benthic infauna were followed. The following steps were carried out during sample collection:

- The vessel was positioned on station and the gear deployed. At the time the gear contacted the bottom, the vessel position, length of wire deployed, and water depth were recorded in the field sample log.
- Upon retrieval of the box corer, it was opened from the top and the sample was evaluated. Insufficient penetration, overfilling, erosion, or other sample disturbance necessitated re-collection of the sample.
- The sample was removed from the corer and placed in a plastic tub.
- A sample label was prepared using waterproof paper and pencil or indelible ink. If the sample could be screened immediately, the label was placed in the tub containing the sample.
- The contents of each tub were transferred to a screen box and rinsed with a gentle stream of seawater.
- The sample was then transferred from the screen box to a 16-ounce or 32-ounce plastic jar using a gentle stream of seawater from a hose or wash bottle.
- The sample label was placed in the jar, against the side so that it could be read.
- Buffered formalin was added to the sample to make a final solution of approximately 10 percent, as follows. Formalin was prepared as a 30 percent solution, and a volume equivalent to half the volume of seawater used to collect the sample was added to each jar. After capping, the jar was inverted gently to mix the formalin solution and seawater.
- Before the next sample was sieved, the screen box was brushed and back-washed with a strong stream of water.

### **Sample Acceptability**

Material collected in the corer was evaluated for acceptability using the criteria set forth in the PSEP (1987) protocols. Each grab was evaluated separately. Acceptability was evaluated on the basis of the following criteria:

- The sampler was not overfilled or underfilled
- Overlying water was present over the sample
- The overlying water was not excessively turbid
- The sediment surface was relatively undisturbed.

The chief scientist evaluated all samples collected. If a grab failed to meet any of the criteria, a sample was not collected from that grab. The actual depth of penetration was recorded for each grab.

### **Sample Collection**

After a grab was judged acceptable, the overlying water was siphoned off. Depth, odor, color, and sediment type were recorded. The sediment was then emptied into a large plastic basin and transported to the sieve table. The inside of each grab was rinsed with seawater; the water and sediment were captured in a plastic basin and added to the sample. Each replicate was screened through a 1.0-mm sieve, washing away the sediment with seawater. The remaining material was rinsed into a polyethylene jar, and buffered formalin was added to make a final concentration in the sample of approximately 10 percent. Sample jars were labeled inside and out, and lids were sealed with plastic electrical tape to eliminate loosening or leakage.

Two sieves and extra plastic tubs were kept on hand to allow sampling to proceed even if sieving was delayed. Sieve boxes were back-washed and scrubbed following collection of each sample.

### **Sample Handling**

Chain-of-custody records were completed at the end of each sampling day. Samples were hand-delivered to E.V.S. Consultants in Seattle, Washington for analysis. Each shipment was accompanied by a packing list.

### **Sample Analysis**

Sample analysis followed PSEP (1987) protocols. Samples were transferred to isopropyl alcohol within 96 hours of collection. No stain was used during sorting. A 20 percent re-sort of each sample was carried out by the original sorter, and an additional 15 percent of all samples was totally re-sorted by another person. Total abundance and the abundance of major taxa (i.e., molluscs, polychaetes, and crustaceans) were determined for each sample.

## COLLECTION OF BIOACCUMULATION SAMPLES

The van Veen grab sampler was used to collect organisms for bioaccumulation analysis. At both Bellingham Bay and the Anderson/Ketron Island site, the bivalve *Compsomyax subdiaphana* was selected for bioaccumulation analyses based on examination of abundance and biomass of organisms in infauna samples. A single sample was collected at each sampling station and was composed of up to 24 samples from the grab (12 deployments of the van Veen). A minimum of 5 grams (wet weight) of tissue was required for analysis (total sample sizes ranged from 5 to 173 grams wet weight).

The following steps were carried out during sample collection:

- Before sampling at each station, an aluminum plate fitted to the sorting table was rinsed with acetone, methylene chloride, and seawater.
- A sample label was prepared and placed on the sample jar.
- The vessel was positioned on station and the gear deployed. The vessel position, length of wire deployed, and water depth were recorded in the field sample log at the time the gear contacted the bottom.
- The grabs were emptied onto the sorting table.
- The sediment sample was then sorted for *Compsomyax subdiaphana*. Sorting was carried out with solvent- and seawater-rinsed gloves and stainless steel implements. Any specimens found in the sample were rinsed free of sediment and placed in the sample jar.
- After all sampling was completed at a station, the total weight (wet weight in shell) of the organisms was recorded.

### Decontamination

All equipment used to collect and handle the organisms was decontaminated prior to use at each station. This procedure consisted of rinses with acetone and methylene chloride applied to the van Veen grab sampler, the aluminum plate on which material was deposited, the stainless steel spatulas, and the latex gloves.

### Sample Acceptability

A minimum penetration depth of 5 cm was considered acceptable. Each side of the dual van Veen grab was evaluated separately. When penetration depth was unacceptable, the material (and organisms) were retained, but the grab was not counted toward the required maximum number of 24 grabs.

## Sample Collection

After the acceptability of a grab was judged, the sampler was emptied onto a solvent-rinsed aluminum plate. A worker with solvent-rinsed latex gloves searched through the sediment for *Compsomyax subdiaphana*. This technique was faster and less prone to contamination problems than sieving, and because of the large size of *Compsomyax subdiaphana* they were easily found. After all the specimens in a pair of grabs had been collected, they were rinsed with site seawater and transferred to acid-washed glass jars.

## Sample Handling

Organisms were kept on ice during collection (i.e., between grabs making up each replicate), storage, and transport. Chain-of-custody records were prepared at the end of each day. The samples were hand-delivered to EcoChem Inc., in Seattle, Washington. The clams were opened, rinsed with seawater to remove adhering sediment and debris, drained, and the tissue shucked into solvent-rinsed jars. Their intestines were removed to eliminate interference from ingested sediment. Samples were then hand-carried to Analytical Research, Inc. in Seattle for organic analyses, then shipped to Columbia Analytical Services, Inc. in Longview, Washington for metals analyses. Chain-of-custody forms and packing lists (including a list of analyses to be performed) accompanied each group of samples.

## Tissue Analysis

Whole *Compsomyax subdiaphana* were removed from their shells prior to homogenization. Sufficient tissue was not collected at most stations to conduct all proposed chemical analyses (i.e., for all PSDDA metals and semivolatile organic chemicals of concern) with appropriate detection limits. To perform as many chemical analyses as possible on the tissue collected from each station, tissue was apportioned for minimum analysis as identified in Table A-4.

Because of the substantial amount of water that is typically associated with whole clam tissue, the wet weight of the samples from each station was an overestimate of the actual wet tissue available for analyses. In order to allocate tissue for chemical analyses, a more accurate tissue wet weight was determined in the following manner. Prior to homogenization, excess water was decanted from the sample jar and saved in a contaminant-free container and the tissue weighed. After determining the wet weight of the tissue, the decanted fluid was recombined with the tissue and thoroughly homogenized.

Each sample was homogenized with a solvent (1:1 solution of methylene chloride and acetone) in a Tissuemizer®. Following homogenization, the material in each sample was apportioned for metals and organic chemical analysis according to the apportionment criteria previously discussed.

The general approach used for organic analysis of sediments was also used for tissue. All samples were processed using GPC cleanup, and the extract volume was reduced to the extent possible to enhance detection limits. Volatile compounds were not analyzed in tissue samples.

In addition to analysis of PSDDA chemicals of concern in tissues, lipid content was determined by gravimetrically weighing an aliquot of the extract for tissues prior to GPC cleanup.

**TABLE A-4. DECISION CRITERIA FOR CHEMICAL ANALYSIS OF TISSUE COLLECTED FROM *COMPSOMYAX SUBDIAPHANA***

Total Sample Weight	Preferred Analyses (in order of priority)
≤1.5 grams	Cd, Pb, and Zn (except in Bellingham Bay samples in which Hg is of highest priority followed by Cd if possible) (analyze other metals only if detection limits are not sacrificed)
1.5 to 3.5 grams	All PSDDA metals of concern (including Hg; in Bellingham Bay, Hg is of highest priority)
3.5 to 7 grams	1 grams - Cd, Pb, and Zn (except Hg is highest priority in Bellingham) (other metals if possible). Remainder of sample - analyze for polycyclic aromatic hydrocarbons (PAH) (analyze for PCB and other organic compounds if possible)
7 to 16 grams	2 grams - All PSDDA metals of concern 4 grams - PCB Remainder - PAH (other organic compounds, if possible)

Tissue samples used for metal analyses were subjected to digestion with nitric and perchloric acids. To attain the detection limits recommended in the PSEP (1986) protocols, GFAA analysis was required for most metals in tissue samples, although digestates were screened by inductively-coupled plasma (ICP) when possible. CVAA was required for determination of mercury.

As with chemical analyses conducted on sediments, quality control procedures required for tissue body burden analyses included:

- Adherence to all QA/QC requirements in accordance with PSEP protocols
- Control limits for accuracy (spikes and reference materials in accordance with PSEP protocols)
- Control limits for precision (replicates in accordance with PSEP protocols)
- Initial and ongoing calibration of instrumentation during analyses (calibration and control limits used, when available, were from PSEP protocols)
- Control limits for blanks in accordance with PSEP protocols.

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