

Evaluation of Sediment from Douglas Harbor in Juneau, Alaska

Sampling Analysis Plan/Quality Assurance Project Plan

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ACRONYMS/ABBREVIATIONS

ARI	Analytical Resources Incorporated
ASTM	American Society for Testing and Materials
BP	bioaccumulation potential
COC	chain of custody
DGPS	differential global positioning system
ID	identification
ITM	Inland Testing Manual
MLLW	mean lower low water
OTM	Ocean Testing Manual
POC	point of contact
SAP	sampling and analysis plan
SM	Standard Methods
SOP	standard operating procedure
SP	solid phase
SVOC	semivolatile organic compound
TOC	total organic carbon
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
NewFields	NewFields Northwest LLC.

UNITS OF MEASUREMENT

°C	degree(s) Celsius
ft	feet
g/kg	microgram(s) per kilogram
µg/L	microgram(s) per liter
µm	micrometer(s)
ng/kg	nanogram(s) per kilogram
cm	centimeter(s)
L	liter
m	meter(s)
mg/kg	milligram(s) per kilogram
mL	milliliter(s)
mm	millimeter(s)
ng/L	nanogram(s) per liter
ppb	parts per billion
ppm	parts per million
ppt	parts per thousand
v/v	volume per volume
yd ³	cubic yards

1 INTRODUCTION

Douglas Harbor (Figure 1), located in Juneau Alaska, is undergoing expansion to accommodate increased moorage demands. The expansion involves removal of existing moorings, creosote pilings, and dredged material to return the harbor to its original design depth of -14 ft MLLW. This involves the removal and disposal of approximately 30,000 yd³ of sediment.

PND Engineering conducted a chemical assessment of Douglas Harbor in March 2007, referred to as the 2007 survey (Figure 2 – PND07- 13, 14, 15, and 16 were samples collected in the New Harbor Dredge Area and the New Surface Dredge Areas). The Concentration of mercury detected in all of the individual samples and the composites were above the project screening level of 0.41 mg/kg. Five of the seven composites had mercury concentrations detected above the Puget Sound Dredged Disposal Analysis Users Manual (PSDDA) maximum level of 2.1 mg/kg. The mercury concentrations were consistent throughout the entire harbor. Mercury was the only contaminant above regulatory guidance values. Biological testing was not a part of the 2007 survey.

A new sampling and testing program will occur in Douglas Harbor with the principal objectives to *verify* the concentrations of mercury present in the sediment, identify the potential forms of mercury present in the system, and determine if the forms of mercury present in the sediment are toxic or bioavailable to selected species of aquatic life. The State of Alaska does not currently have their own dredged material evaluation program, therefore, federal guidance provided in the Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. – Inland Testing Manual (ITM; USEPA/ USACE1998) will be used to conduct field sampling and laboratory testing. The results of this study will facilitate the determination of suitability of Douglas Harbor sediment for aquatic disposal.

The *confirmatory* chemistry and performance of biological and bioaccumulation testing of the sediment within Douglas Harbor is a Tier III evaluation with some Tier IV assessment of the bioavailability of mercury toxicity and bioaccumulation. The results of the chemical and biological analysis will be evaluated according to performance criteria outlined in the ITM (USEPA/USACE 1998) and the Dredged Material Evaluation and Disposal Procedures (Users Manual – July 2008 when applicable). The performance criteria for the ITM are summarized in more detail in Section 7 of this SAP/QAPP.

1.1 BACKGROUND AND HISTORY

Douglas Harbor has undergone a number of renovations, investigations, and dredging operations since the 1940's. The last dredging program occurred in 2003 and at that time, dredged material was placed in the Gastineau Channel disposal site. A summary of activities related to Douglas Harbor includes:

- 1940's: Rock fill material was placed from Douglas Island to create a street out to the City wharf near the harbor entrance.

- 1948: Juneau Island Causeway was constructed along the south margin of the basin to provide vehicle access between the mining facility and Douglas Island.
- 1961: US Army Corp of Engineers (USACE) conducted site investigations for the proposed dredging of the harbor basin and for wave protection at the entrance to the harbor.
- 1962: Harbor basin was dredged to -12 ft MLLW and an entrance breakwater was constructed. Dredged material was placed on the Douglas Island side of a containment berm located along the western limits of the basin. The placement of dredge material provided a foundation for the roadways, parks, and recreational areas known today as Savikko Park.
- 1962-65: Inner harbor facilities were designed and constructed by the State of Alaska. They included Floats A, B & C, an access dock and gangway at Float B, a tidal grid and a boat ramp.
- 1995: US ACOE Civil Works conducted Tier II sampling of the harbor basin in preparation of maintenance dredging (USACE 1995).
- 1997: The US ACOE dredged approximately 25,000 yd³ of material in the entrance channel and northern areas of the basin. Dredged material was disposed in an unconfined manner just outside the harbor in Gastineau Channel, an inland waterway.
- 1998: The City and Borough of Juneau (CBJ) constructed seven stall floats along the north side of Float C.
- 2001-03: The CBJ expanded the Douglas Harbor basin and installed Floats D&E resulting in the current condition. Approximately 65,000 yd³ of material was dredged during this effort. A majority of the dredged material (roughly 90%) was disposed behind a geotextile lined containment berm on site creating a boat launch ramp and parking area. The remaining dredged material was disposed in an unconfined manner outside the harbor in Gastineau Channel.
- 2007-08: The CBJ is currently planning to renovate the original section of Douglas Harbor constructed during the period 1962-65. The existing harbor facilities are severely deteriorated and need to be replaced to provide safe public moorage. The current harbor basin elevation has risen, likely due to glacial rebound and dredging is necessary to maintain safe navigational depth for vessels moored in the harbor.

Figure 1. Douglas Harbor



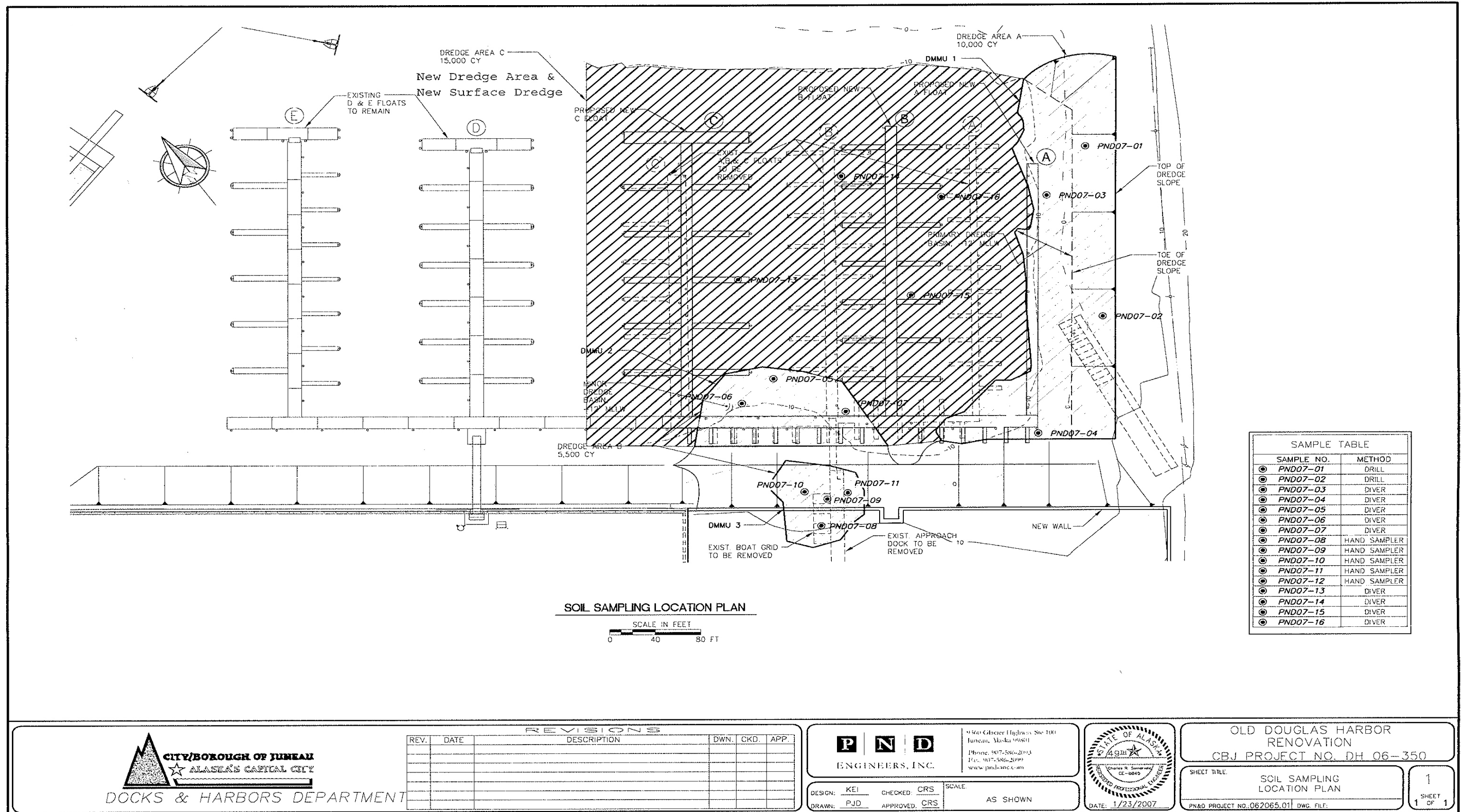


Figure 2. Douglas Harbor Site Map from 2007 Field Survey.

The 2007 PND field survey conducted sediment sampling and physical characterization combined with chemistry analyses of the following chemicals of potential ecological concern:

- Grain size
- Total volatile Solids
- Gasoline Range Organics, Diesel Range Organics, Residual Range Organics
- Benzene, Toluene, Ethylene, and Xylene
- PAHs
- Metals
- Chlorinated hydrocarbons
- Organotins

Mercury was the **only** contaminant determined to be of potential ecological concern with concentrations above the project screening level of 0.41 mg/kg and the PSDDA maximum level of 2.1 mg/kg. Mercury concentrations in the test *composites* from the 2007 survey are summarized in Table 1 (PND 2007) (Data taken from PND Report #062065, p10). Individual sediment sample concentrations ranged from 0.47 to 5.4 mg/kg.

Table 1. Mercury Concentrations in Composite Sediment Samples, 2007.

Sample Location	Mercury Concentration (mg/kg dry weight)
PND-11	1.3
PND-2	2.4
PND-4	2.5
Harbor Dredge	3.5
New Surface Dredge	2.2
PND-1	1.8
PND-3	2.7

Concentrations of the other potential contaminants of concern were below screening levels and will not be analyzed as part of this program (see PND Report #062065 Page 10 for summary of data).

2 SAMPLING STRATEGY AND TESTING OBJECTIVES

The purpose of this 2008 project is to characterize the proposed dredged material from Douglas Harbor to determine suitability for aquatic disposal using guidelines established in the ITM (USEPA/USACE 1998). The testing strategy parallels the tiered testing approach (Section 3) of the ITM.

Specific tasks necessary to accomplish this objective are:

- Collection of test sediment to project depth using two coring devices, vibratory hammer or push core.
- Collection of reference sediment from the proposed reference area (five spatial replicates and one reference composite made from five spatial replicates) using a van Veen grab.

- Toxicity testing of test, reference, and control sediments using ITM methods for benthic toxicity, suspended particulate phase toxicity, and bioaccumulation potential.
- Measurement of a selected suite of potential contaminants of concern in sediment, pore water, and tissue.
- Provide a detailed interpretative report that includes methods, results, and a comparison of test and reference materials using ITM guidance for test acceptability and performance criteria.

Detailed sediment chemistry analysis for a variety of potential contaminants of concern was performed in 2007 as part of the Tier II assessment. The concentrations of mercury were above project screening levels, therefore, a Tier III evaluation will be conducted which includes quantification of the mercury concentrations along with biological and bioaccumulation testing. Figure 3 illustrates the tiered testing approach for this study, (figure taken directly from the ITM (USEPA/USACE 1998)).

The proposed site for receipt of dredged material from Douglas Harbor is the Gastineau Channel (GC) disposal site. To determine suitability of Douglas Harbor material at this disposal site, chemical and biological analysis will include a control for test validation and reference area samples collected and tested concurrently with the test sediment for comparative purposes following ITM procedures.

The native control sediment is specific to each type of toxicity test and is collected in areas not influenced by contaminants and appropriate for each test species. The control sediment should be collected from places where the test organisms naturally reside or sediment that is used to culture the test organisms in the laboratory. The response of the test organism to this sediment serves to confirm the health of the test animals and to validate the acceptability of the tests that are performed.

The purpose of a reference sediment is to provide a point of comparison (reference point) to which benthic effects of dredged material are compared. Reference sediment is collected *outside* the influence of previous disposal operations at a dredged material disposal site, but near enough to the disposal site that the reference sediment is subject to all the same natural influences as the disposal site (USEPA/USACE 1998).

A designated reference site for the purposes of dredged material evaluation does not exist in Juneau, Alaska area. PND and the regulatory agencies (Figure 4 and Figure 5) chose five different locations to represent the reference area. The five locations will be tested separately and has part of a reference composite made from the five locations. There is a possibility that sediment previously disposed of at the Gastineau Channel may have migrated outside the disposal site, therefore, the location of the proposed reference area was chosen from an area not influenced by previous disposal operations.

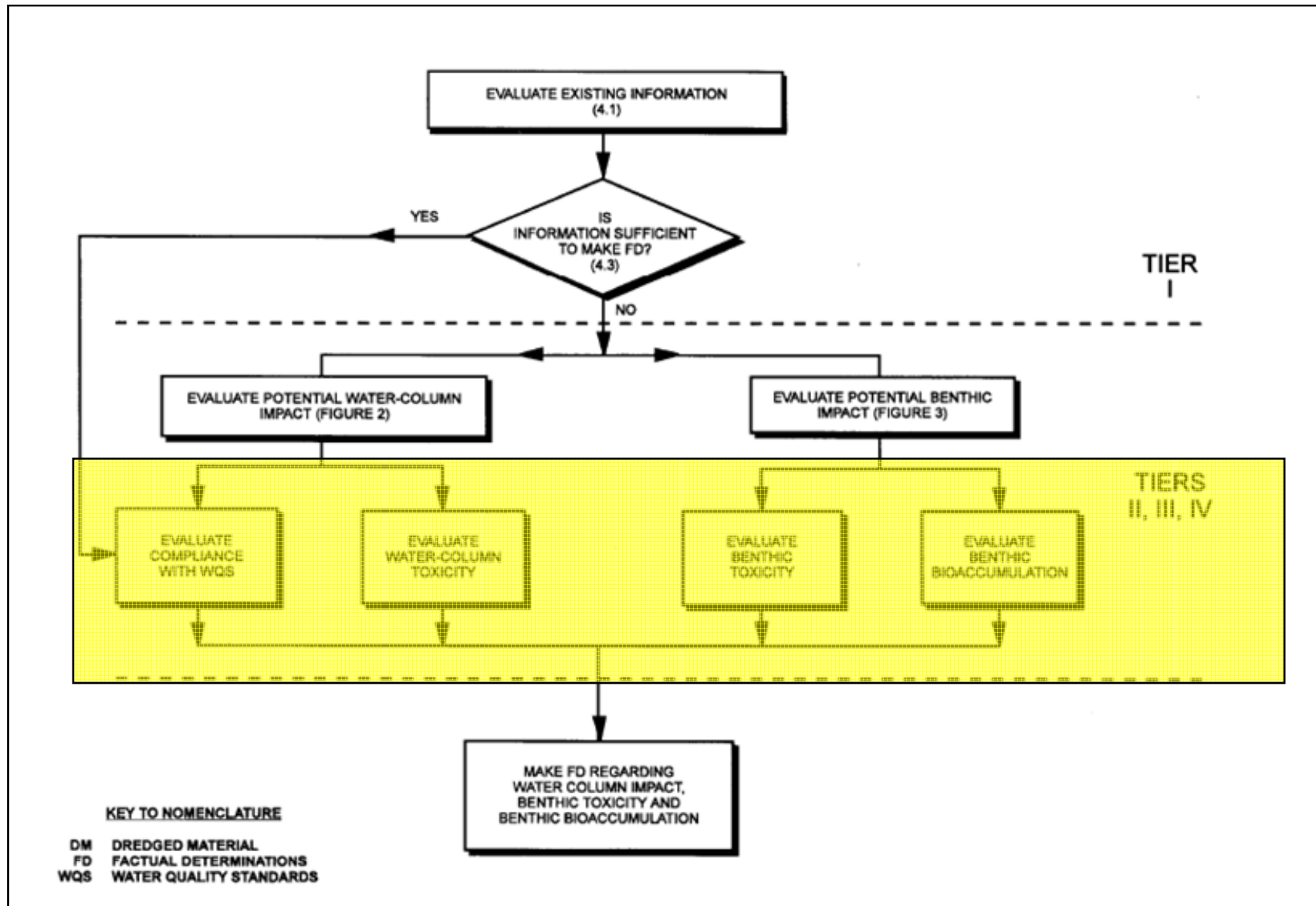


Figure 3. Tiered Testing Approach (ITM 1998).

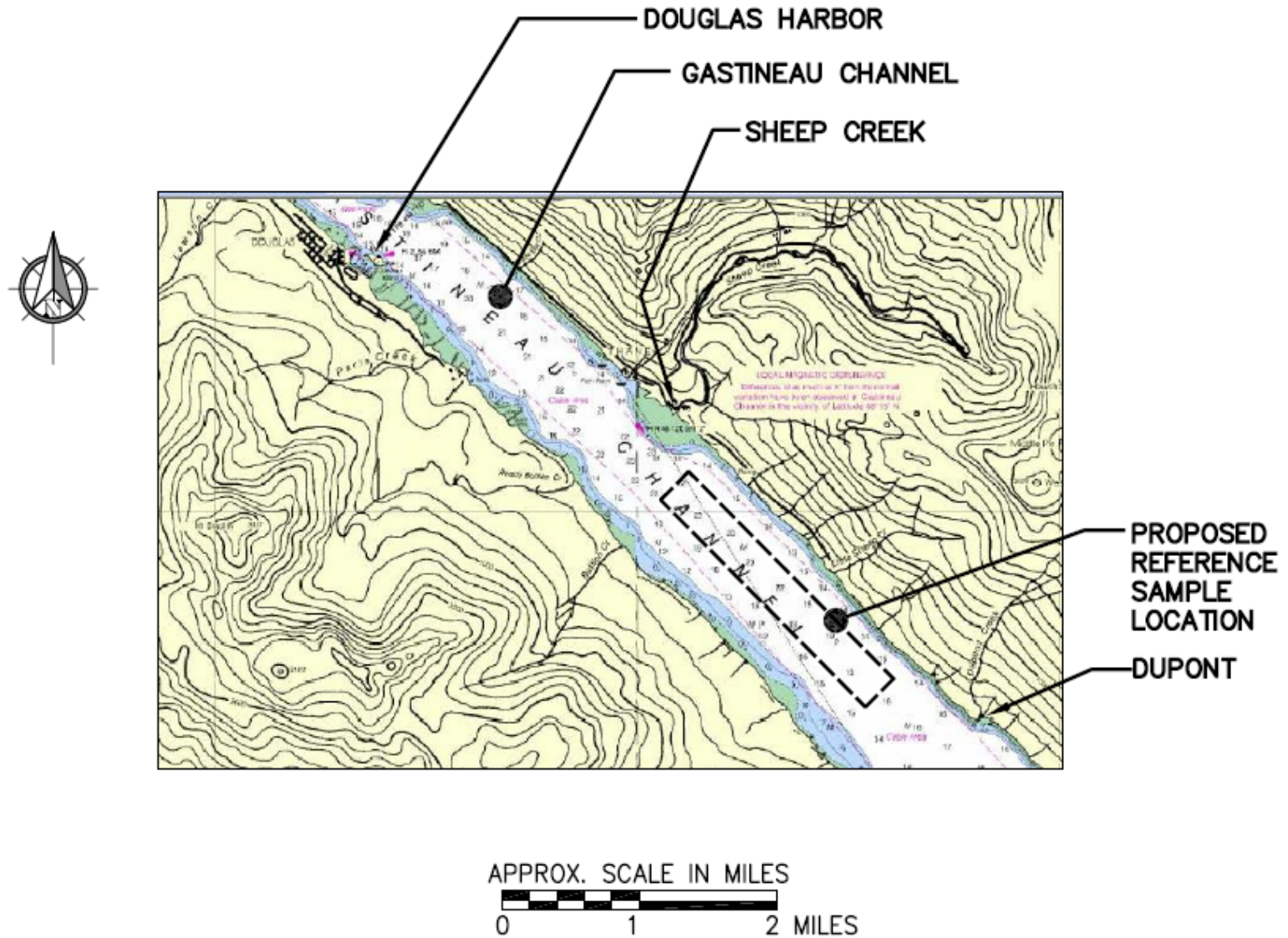


Figure 4. Nautical Chart of Proposed Reference area.

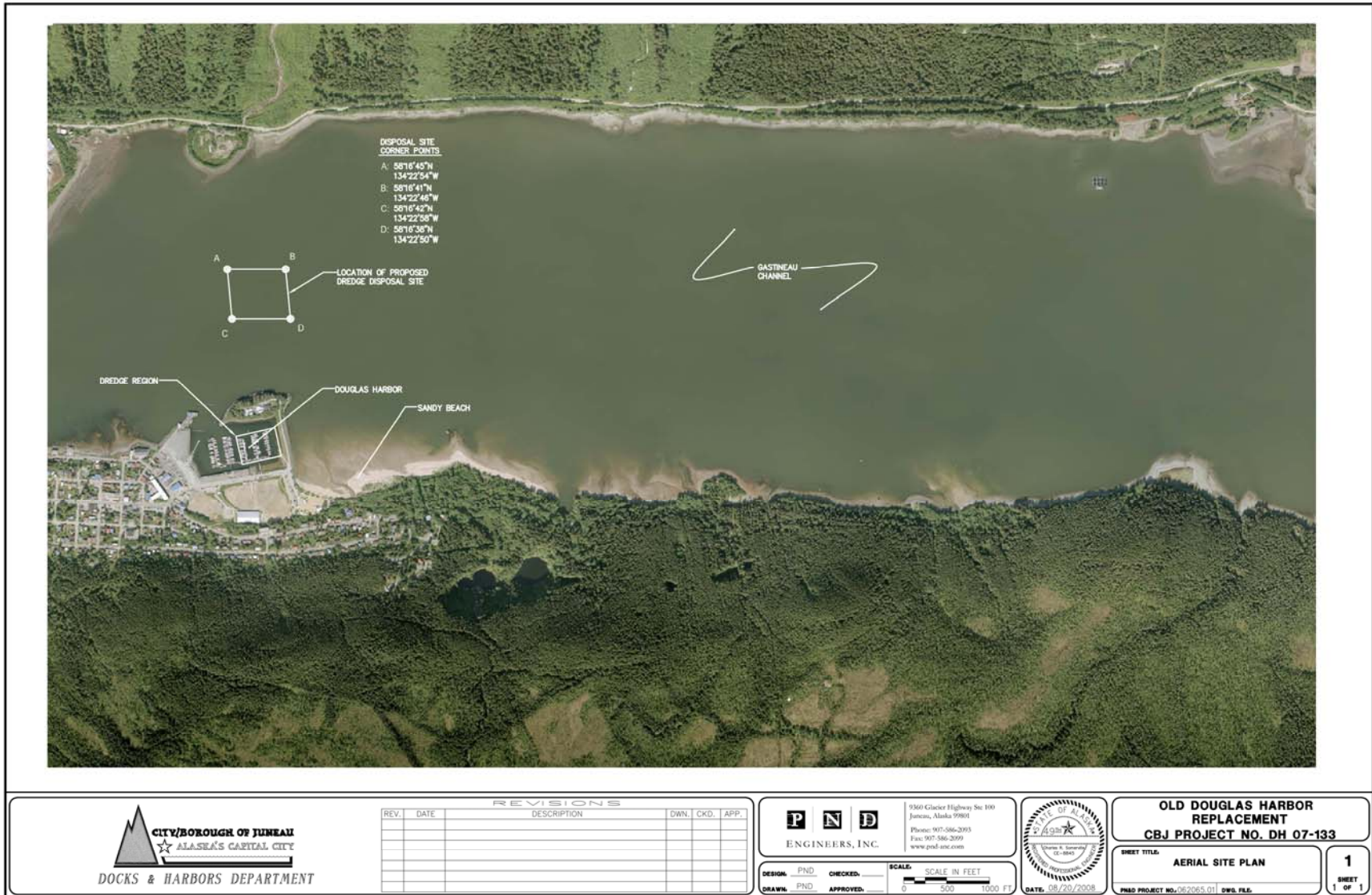


Figure 5. Aerial View of Douglas Harbor and the Proposed Disposal Site. The reference area is not shown on this map.

The five reference samples will be treated as individual spatial replicates for biological testing and will be submitted as individual samples for chemical analysis. These five reference samples will be tested concurrently with the Douglas Harbor sediment treatments and the biological results will be statistically compared to the test sediments. The comparison of reference and test sediment data provides the framework for determining suitability of the Douglas Harbor sediment for disposal at the GC site. Using the five spatial replicates in the comparison incorporates the inherent natural variability of the channel.

The five reference samples will also be combined into one reference area composite based on guidance provided in the ITM when the disposal site is considered heterogeneous in nature (field investigation of the disposal site confirmed heterogeneity of disposal site, data provided as an appendix to this report). This reference area approach is “used when the disposal site is known to be heterogeneous and more than one reference location must be sampled to adequately characterize the disposal site.

2.1 STRATEGY FOR TESTING COMPOSITES AND STATION LOCATIONS

The estimated volume of Douglas Harbor dredged material is approximately 30,000 yd³. Based on the project footprint, four testing composites will be prepared and submitted for toxicological testing (Figure 6 and Section 4). This compositing scheme is consistent and more frequent than guidance provided in the ITM (see excerpt of ITM, Table 2 below) requiring a minimum of two sediment composite from eight sampling locations for volumes of 20,000-100,000 yd³. The previous sediment investigation of Douglas Harbor 2007 identified four different dredged material management units (DMMU -is the smallest volume of dredged material that is capable of being dredged independently from adjacent sediments) 1, 2, 3, and 4 (PND Report #062065). Three of these DMMU areas 1, 2, and 4 are part of this investigation (for consistently and comparison to 2007 data and are shown in Figure 6).

Table 2. Number of Samples and Number of Composites per Dredge Volume (USEPA/USACE 1998).

DREDGE VOLUME* (<i>in situ</i> cubic yards)	MINIMUM # OF SAMPLE STATIONS	# OF COMPOSITES ANALYZED **
5,000 – 20,000	4	1
20,000 – 100,000	8	2
100,000 – 200,000	12	3

Dredged Volume (cubic yard) for Douglas Harbor	Number of Sampling Stations	Number of Composites
30,000	18	4

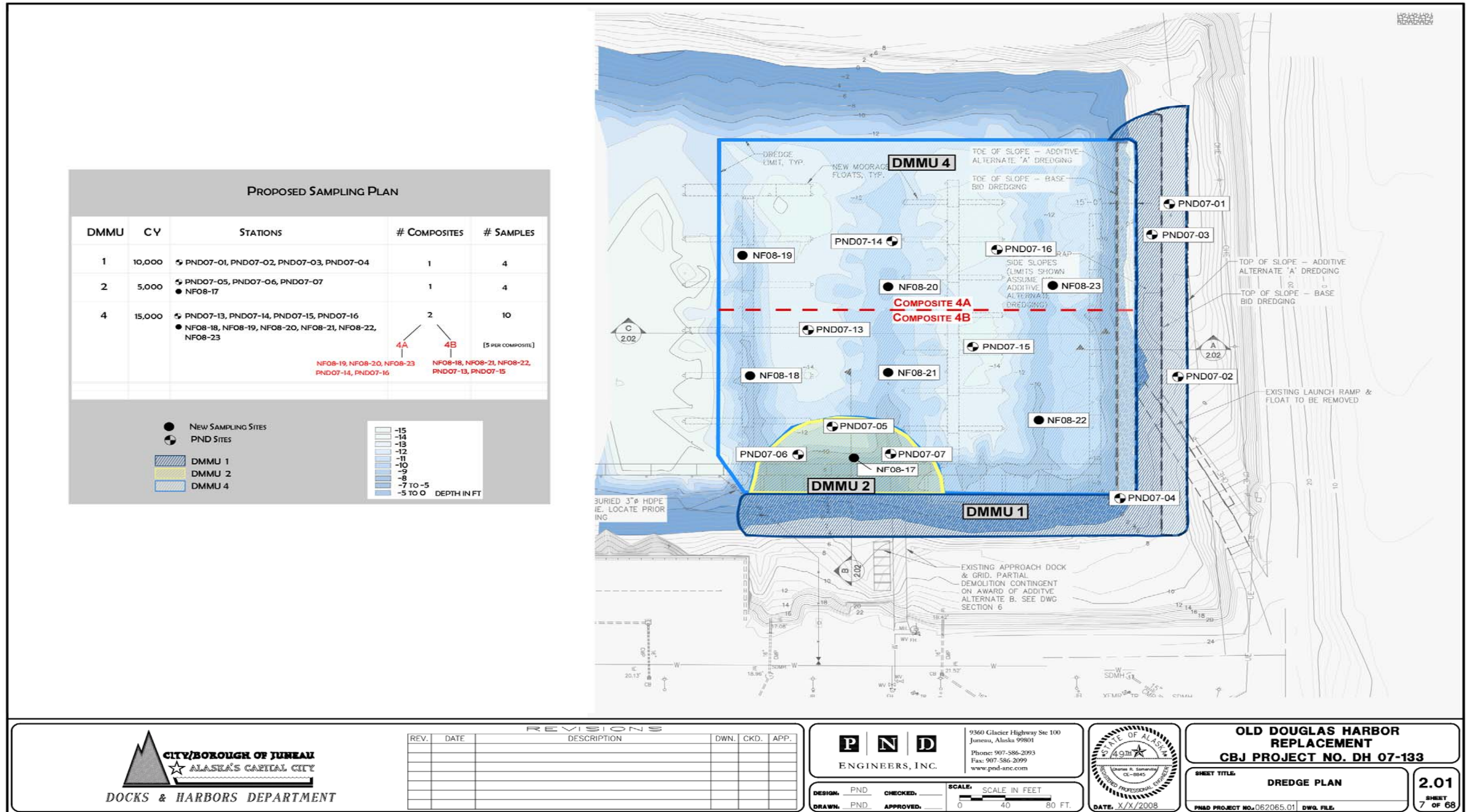


Figure 6. Douglas Harbor Site Map with Field Sampling Locations and Compositing Strategy.

The ITM (USEPA/USACE 1998) and the ITM Supplement (USEPA et.al. 2001) provide guidance on compositing strategies and are summarized as follows:

- Combining locations from contiguous portions of the project area, using sediment that is similar and is exposed to the same influences and pollutant sources.
- The amount of material taken from individual cores for allocation to the test composite is directly proportional to the length of core collected. The amount of test material required for each test composite (including sediment chemistry biological testing and bioaccumulation testing) is approximately ten gallons.
- The procedure for compositing will include sediment from the entire length of core to project depth, however, if individual core samples contain distinct layers the core may be composited vertically to separate any effects that might occur from differing sediment profiles. The proposed project depth is -14 ft. MLLW. The sampling locations reflect the areas previous sampled in 2007. In addition, a few new stations with the NF prefix are included mainly to refine areas where sediment is currently accumulating.

2.2 OVERVIEW OF FIELD ACTIVITIES AND LABORATORY ANALYSES-

Eighteen core samples will be collected to a project depth of -14 MLLW using a push core (possibly with diver assistance) or a vibratory hammer core. The proposed field sampling locations include those previously collected by PND and several new stations (NF designation) that are generally associated with the mounded sediment where the majority of dredging will occur. It is possible that additional analytical samples *will* be required from vertical segments of the cores if the core visually appears to be different based on either color or grain size within the dredge prism. The sediment that is left after proposed dredging (Z-layer) would be sampled for potential chemical analyses. If distinct vertical samples are present from a given location, the distinct layers could be segregated and archived for potential chemical and biological analysis to address specific questions and additional costs would be required for the subsequent analysis. Previous experience has shown compositing field samples leads to a cost effective testing strategy. Moreover, archived individual samples allow for further refinement of contaminant profiles without the need to conduct additional sampling and often reduces the amount of sediment considered unacceptable for aquatic disposal. Therefore, an archive of each samples as well as each composited sample will be retained for potential future assessment.

In addition to collection of sediment from Douglas Harbor, five individual reference sediment samples will be collected from Gastineau Channel using a modified van Veen grab sampler. These five reference samples will be chemically analyzed and biologically tested as individual spatial replicates. A composite of the five reference locations will also be chemically analyzed and tested. Control sediment will be supplied by aquatic vendors who culture or collect the proposed test organisms.

Chemical analyses of the test and reference material for each area include mercury for sediment and percent moisture, lipid analyses, and mercury for the tissues. Physical measurements on the sediment include percent moisture, TOC, grain size, and SEM/AVS analysis.

Biological evaluation following ITM protocols of the dredged material proposed for aquatic disposal will include the 10-day amphipod test, the 10-day juvenile polychaete test, and the water column tests with three species representing different phyla and differences in species sensitivities. The chosen species will be based on availability and spawning season and will most likely include the mysid (*Americamysis bahia*), the silverside fish (*Menidia sp.*) and the bivalve larvae of the mussel (*Mytilus edulis*). Dredging site water from Douglas Harbor will be used to prepare suspended particulate phase material and clean seawater from Port Gamble Bay will be used as the dilution water. Detailed testing methods are provided in Section 5 of this SAP/QAPP.

In addition, bioaccumulation testing will be conducted using a polychaete, *Nephtys caecoides*, and a bivalve clam, *Macoma nausta*, to determine the ability of contaminants to accumulate in the tissues of organisms. Test duration for metal bioaccumulation will be 28 days, as recommended in the ITM.

3 PROJECT MANAGEMENT AND TEAM RESPONSIBILITIES

3.1 PROJECT MANAGEMENT

Meg Pinza will be the Project Manager for NewFields and will serve as the point-of-contact (POC) for this project. She will provide oversight for planning and implementing the project, as well as coordinating with PND, along with Dr. Jack Q. Word (Senior Project Manager from NewFields). She will coordinate the efforts of the various team members, respond to requests, provide technical consulting and coordination with USEPA and/or USACE, and ensure that project goals, budgets, and schedules are met. Mr. Jack D. Word of NewFields will serve as the Field Operations Manager. He will assist Ms. Pinza in coordinating team efforts and will provide oversight for all field activities. Mr. Brian Hester is NewFields' Laboratory Manager for the biological evaluations of sediment. Ms. Lucinda Word of NewFields will serve as the quality assurance/quality control (QA/QC) officer and will be responsible for adherence to QA/QC requirements specified for collection, handling, and analyses. Mr. William Gardiner of NewFields will provide QA/QC review of all chemical data and interact with the analytical laboratories. Ms. Susie Watts will oversee the statistical analysis that will be performed for this program.

3.2 TEAM RESPONSIBILITIES

NewFields will provide field-sampling equipment that is unavailable in Juneau, coordinate field logistics with PND, and conduct the field sampling and the laboratory testing. The Battelle Marine Sciences Laboratory in Sequim, Washington will conduct the analytical chemistry for sediment and tissues. ARI in Seattle, Washington will conduct physical analysis of the sediment. The NewFields office in Port Gamble, Washington will perform biological testing, review all analytical data, and perform all data analyses. NewFields will produce the final reports, with review and approval by PND and CBJ.

4 FIELD COLLECTION PROGRAM FOR SEDIMENT CORE SAMPLES

4.1 SAMPLING LOCATIONS AND SAMPLING DEPTHS

Sediment cores will be collected from eighteen stations to a project depth of -14 ft MLLW. If the sampler cannot penetrate to project depth due to sediment type, then vessel will be moved and a second attempt will be made to collect a sample. If project depth is not

achieved on the second attempt, then additional cores will not be attempted unless equipment operational problems are suspected.

A subsample of each field station sample will be collected and individually archived for possible future analysis (one 16-ounce archive for chemistry only unless a vertical compositing strategy is necessary to separate distinct sediment layers). The remainder of the sediment from each station will contribute *proportionally* to the test composite based on the length of core collected. For example, a 7 ft core would contribute more sediment to the composite than a 2 ft core. This method provides a dredge material sampling composite that is comparable to the projected volume of dredged material produced from each location. Table 3 provides a summary of the field stations, expected core lengths, amount of material allocated to the composite, compositing strategy, and type of field sampler proposed for each station.

Table 3. Proposed Stations and Locations in Douglas Harbor Juneau, Alaska.

Site Location: 58°16'30"N 134°23'8"W							
Field Station	DMMU	Estimated Core Length (ft)	Type of Sampler	Volume Contribution (gal) ¹	Length of Core Needed for Volume (ft) ²	Number of Cores/Grabs to Collect	Sediment Composite
RS-01 – RS-05	NA	NA	Van Veen grab	10		3 to 4 grabs per station	5 samples
PND07-01	1	TBD rip rap area	Vibratory hammer core	TBD in field	TBD in field	TBD in field	1
PND07-02	1	TBD rip rap area	Vibratory hammer core	TBD in field	TBD in field	TBD in field	1
PND07-03	1	TBD rip rap area	Vibratory hammer core	TBD in field	TBD in field	TBD in field	1
PND07-04	1	TBD rip rap area	Vibratory hammer core	TBD in field	TBD in field	TBD in field	1
PND07-05	2	3	Push core / diver assistance	2.1	4.2	2	2
PND07-06	2	4	Push core / diver assistance	2.9	5.6	2	2
PND07-07	2	4	Push core / diver assistance	2.9	5.6	2	2
NF08-17	2	3	Push core / diver assistance	2.1	4.2	2	2
PND07-14	4	3	Push core / diver assistance	1.2	2.4	1	4A
PND07-16	4	2.5	Push core / diver assistance	1.0	2.0	1	4A
NF08-19	4	7	Vibratory hammer core	2.9	5.7	1	4A
NF08-20	4	6	Vibratory hammer core	2.5	4.9	1	4A
NF08-23	4	6	Vibratory hammer core	2.5	4.9	1	4A
PND07-13	4	1.5	Push core / diver assistance	0.7	1.3	1	4B
PND07-15	4	2.5	Push core / diver assistance	1.1	2.2	1	4B
NF08-18	4	6	Vibratory hammer core	2.6	5.1	1	4B
NF-08-21	4	6	Vibratory hammer core	2.6	5.1	1	4B
NF08-22	4	7	Vibratory hammer core	3.0	5.9	1	4B

¹ Contribution of each station to a 10 gallon composite
² Assumes 1 ft of core is = 0.5 gallons (based on diameter of core liner = 3.125 in)

Two sampling devices, a push core and a vibratory hammer core, will be used for this program based on their ability to work in a variety of sediment types and water depths.

These types of samplers allow for collection of the large sediment volumes necessary to accommodate both chemical and biological analyses.

As stated previously, a reference area approach will be used for determination of suitability of the material for disposal. Individual reference sediment samples will be collected from areas expected to be outside of the influence of the disposal site using a van Veen grab sampler. The exact locations of the reference sites will be chosen in consultation with the regulatory agencies, PND, CBJ, and NewFields. The reference area samples and the reference area composite will serve as a point of statistical comparison to the test data.

Native control sediment will be collected or supplied by the aquatic organism vendor and will be tested along with the reference and control samples.

4.1.1 Core Collection Techniques

All sampling will occur onboard the tug vessel *WALDO* that has deck space and crane lifting capabilities to accommodate the field collection equipment.

The process for sediment collection is similar using either the push core or the vibratory hammer core except that the push core is pushed through the sediment and the vibratory hammer core is vibrated through the sediment using a vibrating hammer. The procedure involves lowering the coring device to the sediment surface and then driving the core through the sediment to project depth. Once onboard the vessel, the core is placed horizontally on the deck and the core liner is extruded, cut into smaller sections that are capped on either end and placed in coolers containing blue ice to provide temperatures approximately 4°C.

A diver may assist with collection of the sediment using the push core to ensure efficient penetration in the sediment at a vertical angle and to aide with sediment retrieval in areas under the moorings. The diver will position over the sampling location, help with manually pushing the core into the sediment, lift the core back out of sediment and quickly cover tube with hand or cap prior to returning to the water surface.

To minimize cross contamination between station locations, individual core liners will be used for each station. The core liners are non-contaminating Lexan[®] liners that are preferred for core sampling based on their durability and proven success from previous testing programs.

4.1.2 Van Veen Grab Collection

A stainless steel van Veen grab will collect the reference sediment samples. The tug *Waldo* will be used for transit to the proposed Gastineau Channel reference area. Sediment representing the upper 10 - 12 centimeters within a sampling area of 0.1 square meters will be collected and transferred to labeled polyethylene bags that are stored in coolers maintained approximately 4°C during all aspects of shipping and handling. This is equivalent to approximately three gallons of sediment per sample, which requires three grab samples per site.

4.1.3 Water Collection for Water Column Test Preparation

Douglas Harbor site water will be collected into pre-cleaned polycarbonate carboys. The carboy with the lid attached will be submersed below the water surface, the lid will be

removed, water allowed to fill the carboy, then the lid will be replaced on the carboy, and the container will be brought to the surface. This procedure avoids collecting any surface water that may contain oil or other materials that could interfere with the test. Approximately 40 L of site water are required to conduct the three water column tests as described in Section 5. We will collect 80 L of water to serve as a backup in case additional testing is required.

4.1.4 Navigation

All station locations will be determined using a Differential Global Positioning System (DGPS). The system uses U.S. Coast Guard differential correction data, and is accurate to ± 3 meters. In the event of differential failure, stations will be located using a land surveying system consisting of two identifiable ranges, or laser range finder and compass. All final station locations will be recorded in the field using positions from the DGPS or through lineups on the field map.

4.1.5 Sediment Handling

The core stratigraphy will be recorded in the field log by viewing through the clear Lexan[®] core liner. The field observation will focus on homogeneity of the vertical samples. If the sediment layers appear different, then a second core will be collected to ensure that adequate volumes of sediment are available for potential vertical compositing, enough sediment would be collected to perform additional chemical and biological analysis. After inspection, the core will be cut into two to three foot sections and placed into labeled coolers maintained at approximately 4° C until delivery to the NewFields' laboratory in Port Gamble, Washington for processing. Upon return to NewFields in Port Gamble, a representative core from each sample location will be photographed and characterized for sediment characteristics. The geologic description of each core will include the texture, odor, color, length, approximate grain size distribution, and any evident stratification of the sediment.

Sediment collected from the reference sites will be placed into clean, polyethylene bags, labeled (project name, date, sampler ID, analysis, logged into a field chain-of-custody (COC) form, and placed into a cooler maintained at approximately 4° C until delivery to the NewFields' laboratory in Port Gamble, Washington for processing.

Every cooler will contain a temperature blank that is used to assess the temperature of the cooler upon arrival at the testing laboratory and a chain of custody form will be attached to the inside of the cooler lid (Section 4.3)

4.1.6 Sample Processing and Storage

Sample processing and compositing will be performed at the Port Gamble NewFields laboratory. Each sediment sample will be homogenized to a uniform consistency at the laboratory using a stainless steel mixing apparatus. Each test composite will be generated by allocating sediment from each station based on the length of core collected.

Samples for physical and chemical analysis will be placed into certified clean glass jars with Teflon-lined lids and shipped to the analytical laboratories. Sub-samples for archive will be placed in certified clean glass jars with Teflon-lined lids and frozen at -20°C for possible future chemical analysis in the event that further delineation of chemical contamination among stations is required. The remainder of the composite sample will be analyzed for

toxicity. All sediment samples will be stored in the walk-in cold room at the Port Gamble laboratory maintained at a constant temperature of approximately 4°C.

4.1.7 Shipping

Chemistry jars for mercury analysis will be provided by the analytical laboratory. The analysis jars are cleaned according to methods outlined for mercury analysis that include collection in acid-cleaned Teflon™, or glass bottles with Teflon-lined lids. Briefly, the cleaning process involves washing the bottles or glass jars and then boiling them in concentrated HNO₃ for 48 hours. Bottles are rinsed in tap water shown to contain negligible concentrations of methyl mercury, and then filled with 0.5% HCl in low Hg water and heated to 65°C for a minimum of 24 hours. This water is then poured off and the bottles are refilled with 0.5% HCl in low Hg water, and then stored until use. Prior to use, the vessels are emptied and dried in a clean drying oven at 65°C.

After the sediment is composited and sampled for chemical analysis, the chemistry samples will be placed in sealable plastic bags and securely packed inside the cooler with blue ice. The COC forms will be completed (see Section 4.3), and the original signed COC forms will be placed in a sealable plastic bag and placed inside the cooler. The cooler lids will be securely taped shut.

Table 4 lists the laboratories, the particular analyses to be performed by each, and the point of contact and pertinent shipping information for each laboratory.

Table 4 . Analytical Laboratories, Point of Contact, and Shipping Information.

Laboratory	Analyses Performed	Point of Contact	Shipping Information
NewFields Northwest LLC.	All biological testing including the toxicity tests and the bioaccumulation tests	Ms. Meg Pinza Mr. Brian Hester (360) 297-6040	NewFields Northwest LLC. 4729 NE View Drive Port Gamble, WA 98364
Pacific Northwest National Laboratory – Battelle Marine Sciences Laboratory	Mercury Analysis Acid Volatile Sulfides (AVS) Simultaneously Extracted Metals (SEM) TCLP extraction and analysis	Ms. Brenda Lasorsa (360) 681-3650	Battelle Marine Sciences Lab 1529 West Sequim Bay Road Sequim, Washington 98362
Analytical Resources Inc	TOC Total solids Grain Size	Ms. Sue Dunninghoo (206) 695-6200	Analytical Resources, Inc. 4611 S.134 th Pl, Suite 100 Tukwila, WA 98168

4.2 DECONTAMINATION OF FIELD AND LABORATORY EQUIPMENT

All sampling and laboratory equipment will be cleaned prior to sampling. In the field the core and grab, samplers will be rinsed between stations with site water. To avoid cross contamination between stations, individual core Lexan® liners will be used to collect the sediment samples.

Sediment composting will be conducted at the Port Gamble laboratory using clean sampling techniques. All stainless steel utensils (bowls, spoons, spatulas, mixers, and other utensils) will be cleaned with soapy water, rinsed with tap water, and then rinsed three times with deionized water. The final cleaning step involves a rinse with methylene chloride to remove any trace of soap or organic residue. Glassware will be cleaned with soapy water, rinsed

with deionized water, soaked in a nitric acid bath and the rinsed with methylene chloride prior to use.

4.3 DOCUMENTATION AND CHAIN OF CUSTODY (COC)

This section describes the program requirements for sample handling and COC procedures. Samples are in custody if they are: (1) in the custodian's possession or view, (2) retained in a secured place (under lock) with restricted access, or (3) placed in a secured container. The principal documents used to identify samples and to document possession are COC records, field logbooks, and field tracking forms. COC procedures will be used for all samples throughout the collection, transport, and analytical process, and for all data and data documentation, whether in hard copy or electronic format.

The COC procedures will begin during sample collection. A COC record will be prepared for each sample. Each person who has custody of the samples will sign the form and ensure that the samples are in custody unless properly secured. Minimum documentation of sample handling and custody will include the following:

- Sample identification
- Sample collection date and time
- Any special notations on sample characteristics
- Initials of the person collecting the sample
- Date the sample was sent to the laboratory
- Shipping company and waybill information

The completed COC form (Figure 7 is an example of the NewFields COC form) will be placed in a sealable plastic envelope that will travel inside the ice chest containing the listed samples. The person transferring custody of the samples will sign the COC form, and the condition of the samples will be recorded by the receiver. COC records will be included in the final analytical report prepared by the laboratory, and will be considered an integral part of that report.

CHAIN OF CUSTODY
13180

NewFields Northwest, LLC.
Shipping: 4729 NE View Dr.
Mailing: P.O. Box 216
Port Gamble, WA. 98364
Tel: (360) 297-6040, Fax: (360)297-7268



Destination Lab:		Sample Originator:		Report Results To:	
Destination Contact:		Contact Name:		Phone:	
Date:		Address:		Fax:	
Turn-Around-Time		Phone:		Email:	
Project Name		Fax:		Invoicing To:	
Contract/PO:		E-mail:		Comments or Special Instructions:	
				Preservation	
				Sample Temp Upon Receipt	
				LAB ID	
No.	Sample ID	Matrix	No. & Type of Container	Date & Time	
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
Print Name:	Relinquished by:	Print Name:	Received by:	Print Name:	Received by:
Signature:		Signature:		Signature:	
Affiliation:		Affiliation:		Affiliation:	
Date/Time:		Date/Time:		Date/Time:	
Matrix Codes					
FW = Fresh Water					
WW = Waste Water					
SB = Soil & Sediment					
SS = Soil & Sediment					
TB = plant & Animal Tissue					
OT = Other					

Example

WHITE - return to originator • YELLOW - lab • PINK - retained by originator

Figure 7. Chain of Custody Form

5 BIOASSAY TESTING

This section summarizes the test methods that will be used to conduct the benthic, water column and bioaccumulation potential (BP) tests. All sediment samples will be evaluated in accordance with procedures outlined the Inland Testing Manual (USEPA/USACE 1998). This program will include bioassay analysis of four test composite samples and five individual reference samples. In addition, appropriate laboratory control samples will be run with each of the selected test species. Ammonia and sulfide concentrations in composite sample pore-water will be analyzed prior to bioassay testing in the bulk sediments. Bioassay testing for this project consists of two benthic toxicity tests, three water-column toxicity tests, and two BP tests. The bioassays proposed for this project are summarized in Table 5.

Table 5. Bioassay Testing Proposed for Suitability of Dredged Material for Douglas Harbor

Test Type	Type of Organism	Taxon	Project Sediments	Individual Reference Sediments ¹	Control Sediment/ Seawater	Reference Toxicant ¹
Water column	Bivalve larvae	<i>Mytilus</i> sp	X		X	X
	Fish	<i>Menidia beryllina</i>	X		X	X
	Mysid shrimp	<i>Americamysis bahia</i>	X		X	X
Benthic	Amphipod	<i>Ampelisca abdit</i> , <i>Eohaustorius estuarius</i> or <i>Rhepoxynius abronius</i>	X	X	X	X
	Polychaete	<i>Neanthes arenaceodentata</i>	X	X	X	X
BP	Bivalve	<i>Macoma nasuta</i>	X	X	X	
	Polychaete	<i>Nephtys caecoides</i>	X	X	X	

¹Shaded areas indicate tests or treatments that are not applicable to the selected tests.

5.1 WATER COLUMN TESTING

Water-column bioassay tests will be performed to estimate the potential impact of aquatic disposal of dredged material to organisms that live in the water column. Table 6 provides of summary of testing conditions for three different water column species. The water column test will be performed using a 4:1 dilution by volume seawater to test dredged material. Sediment from each composite will be combined with dredging-area site seawater in a 4:1 ratio by volume, vigorously agitated for 30 minutes, and then centrifuged for 30 minutes at 980 g. Following settling, the supernatant will be gently decanted. This supernatant represents the 100% test concentration and is used to create serial dilutions with clean seawater (0.45- μ m-filtered Hood Canal seawater) to create subsequent test concentrations for the water column tests. Three species will be tested: *Mytilus* sp. (bivalve larvae), *Americamysis bahia* (mysid shrimp), and *Menidia beryllina* (inland silverside fish).

The bivalve larvae test will be run on the test sediment elutriates at 100%, 50%, 10%, 1% and a seawater control. There will be five replicates per elutriate; a surrogate replicate will also be set up for use in water quality measurements. The test will be run for 48 hours, or longer if necessary, to ensure development of the bivalve larvae to the D-hinge stage in the

control. At the termination of the study, survival and normal development will be compared between the control and test groups to determine if significant mortality or abnormal development exists. If bivalve larvae are not available (due to seasonal availability), an echinoderm species (either the sea urchin *Strongylocentrotus purpuratus* or the sand dollar *Dendraster excentricus*) will be used for the larval test.

For the mysid and the fish, the water column will be tested at 100%, 50%, 10% and a seawater control under static conditions. Each of these tests will be conducted in accordance with procedures outlined in the ITM (USEPA/USACE 1998). Ten animals will be used per replicate with five replicates per elutriate concentration and a surrogate replicate for water quality measurements. Each test will be run for 96 hours.

Daily water quality monitoring of test chambers will be carried out for pH, dissolved oxygen, salinity, and temperature. Ammonia and sulfides will be analyzed at the start and end of the test in the 100% concentration. Measurements in other concentrations will only be performed if the readings in the 100% concentration are greater than 4 mg/L total ammonia. To evaluate the relative sensitivity of the organisms, reference toxicity tests will be performed using ammonia reference toxicants (Lee 1980).

Table 6. Summary of Test Conditions for the Water Column Tests

Test Condition	Water Column Test Species		
Test Organism:	<i>A. bahia</i>	<i>M. beryllina</i>	<i>M. edulis</i>
Age of Organism:	1-5 day; 24 h range	9 – 14 days day; 24 h range	larvae, post fertilization
Test Type:	Static non-renewal	Static non-renewal	Static non-renewal
Duration:	96 h	96 h	48 h
Test Chamber:	250 mL minimum	250 mL minimum	20 mL scintillation vials
# Organisms /Jar:	10	10	15 to 30 /mLs
Test Volume:	200 mL minimum	200 mL minimum	10 mL
Replicates:	6 (5 + WQ rep)	6 (5 + WQ rep)	6 (5 + WQ rep)
Sediment Holding Time	< 8 weeks	< 8 weeks	< 8 weeks
Water Quality:			
Temperature:	20°C ± 1°C	20°C ± 1°C	16°C ± 1°C
Salinity:	Ambient ± 2 ppt	Ambient ± 2 ppt	Ambient ± 2 ppt
Dissolved Oxygen:	≥ 40 % saturation	≥ 40 % saturation	≥ 4.0 mg/L (no air unless needed)
Dilution water	Natural seawater	Natural seawater	Natural seawater
Test concentrations	Three concentrations 100, 50, and 10%	Three concentrations 100, 50, and 10%	Three concentrations 100, 50, and 10%, and 1%
Feeding Schedule:	Daily	Daily	none
Ration/Diet	<i>Artemia</i> solution 0.2 mL of <24 h old (about 100 nauplii)	<i>Artemia</i> solution 0.2 mL of <24 h old (about 100 nauplii)	none
Lighting quality	Ambient lighting	Ambient lighting	Ambient lighting
Photoperiod	16L/8D	16L/8D	16L/8D
Endpoints:	Survival	Survival	Survival to normal larvae to D-stage
Test acceptability criteria	≥ 90% survival in control	≥ 90% survival in control	≥ 70% survival and ≥70% normal shell development in control
Reference Toxicant	Yes –NH ₃ and Copper Sulfate	Yes –NH ₃ and Copper Sulfate	Yes –NH ₃ and Copper Sulfate

5.2 BENTHIC TESTING

Benthic bioassays will be performed to estimate the potential impact of aquatic disposal of the proposed dredged material on benthic organisms that attempt to re-colonize the area. Sediment will be tested in 10-day benthic tests using an amphipod species, *Ampelisca abdita*, *Eohaustorius estuarius* or *Rhepoxynius abronius* depending on sediment grain size) and polychaete worm, *Neanthes arenaceodentata*. Amphipod testing will be conducted in accordance with procedures described in Appendix E of the ITM (USEPA/USACE 1998) and ASTM Standard E1367-99 (ASTM 2003c) and outlined in Table 7. Tests with the polychaete will be conducted in accordance with procedures outlined in the ITM (USEPA/USACE 1998). Each sediment type (test and control) will be run with five replicates. Control sediment will be sediment from the area where the organisms were collected (i.e., native sediment).

Test organisms will be exposed in a static system to the sediment for ten days in 1-liter glass test chambers. Two centimeters of sediment (approximately 150 mL) will be placed into each chamber with 800 mL of overlying water. Initial stocking densities in each replicate will be 20 organisms per test chamber for the amphipod test, and 5 organisms per test chamber for the polychaete test. Trickle-flow aeration will be provided through glass pipettes, in such a way as to avoid disturbing the sediment surface. Water quality measurements will be taken in one chamber from each test treatment daily and will include pH, salinity, temperature, and dissolved oxygen. Ammonia and sulfides will be measured in both interstitial (pore water) and overlying water at the start and finish of the test from one replicate for each test sample. Sediment pore water will be extracted via centrifugation. All instruments used will be calibrated and logged daily. Using methods described in the ITM (USEPA/USACE 1998), the sediments will be carefully sieved to remove the test organisms, and then survivorship will be assessed. To evaluate the relative sensitivity of the organisms, reference toxicity tests will be performed using standard reference toxicants (Lee 1980).

Table 7. Summary of Test Conditions for the Benthic Tests

Test Condition	Test Species	
	<i>N. arenaceodentata</i>	<i>A. abdita</i>
Test Organism:		
Age of Organism:	2 to 3 weeks	Mature, 3 – 5 mm mixed sexes
Test Type:	Static, Non-renewal	Static, Non-renewal
Duration:	10-d	10-d
Test Chamber:	1-L	1-L
# Organisms /Jar:	5	20
Test Sediment Volume/ Seawater Volume	200 mL/800 mL	2 cm minimum/900mL
Sediment holding time	< 8 weeks	< 8 weeks
Replicates:	6 (5 + WQ rep)	6 (5 + WQ rep)
Water Quality:		
Temperature:	20°C ± 1°C	20°C ± 1°C
Salinity:	28 to 30 ppt	28 to 30 ppt
Aeration	Trickle-flow(<100 bubbles/min.)	Trickle-flow (<100 bubbles/min.)
Ammonia/Sulfides:	Day 0 and 10	Day 0 and 10
Lighting quality	ambient	ambient
Photoperiod	12L/12D	Continuous
Endpoints:	Survival	Survival
Test acceptability criteria	≥ 90% survival in control	≥ 90% survival in control
Reference Toxicant	Yes	Yes

5.3 BENTHIC BIOACCUMULATION POTENTIAL (BP) TESTING

Assessment of BP will be carried out using the polychaete worm *Nephtys caecoides* and the bivalve *Macoma nasuta* over a 28-day test period. BP tests will be conducted in accordance with Appendix E of the ITM (USEPA/USACE 1998) and summarized in Table 8. Each test will be initiated using test, reference, and control sediments in the same manner as the 10-day benthic test. Background tissue samples will be archived for both in the event that a baseline tissue concentration is required. Five replicate tests will be performed for each composite sample. *N. caecoides* and *M. nasuta* will be tested in the same aquaria using a minimum of 20 animals per replicate for the polychaete and a minimum of 10 animals per replicate for the bivalve. The test chambers will be maintained under flow-through conditions and daily water quality measurements will be taken on each chamber. On Day 28, the sediment will be sieved to remove the worms and clams. The surviving animals will be placed in clean flow-through aquaria to purge their gut contents for 24 hours, after which tissues will be placed into certified-clean glass sample jars, frozen and sent to the chemistry laboratory for tissue analysis. If mortality exceeds 25 percent in reference or test dredged material, discussion with the regulatory agencies will occur regarding reduced tissue volume for chemical analysis (which could result in the necessity of increased chemical detection limits and/or compositing of replicates).

The analysis of bioaccumulation will be made by statistically comparing tissue levels from the test group to data from the reference area for each species. The analysis will be conducted using Analysis of Variance, t-tests, or non-parametric tests, depending on the assumptions of the individual tests (i.e., homogeneity of variance) as specified in the ITM (USEPA/USACE 1998). Contaminant concentrations found to be significantly elevated above reference will be interpreted in light of criteria specified in the ITM, comparisons to Federal Drug Administration limits, and to Alaska specific fish consumption guidance provided as written comment by ADEC to be 0.32 ppm wet weight of total mercury (State of Alaska Division of Public Health, 2007).

Table 8. Summary of Test Conditions for the Bioaccumulation Potential Tests.

Test Conditions	
Test Procedures	ITM (USEPA/USACE 1998)
Test type/duration	28-day static with flow through
Water Quality	
Test temperature	Recommended: 14 ± 2 °C
Test Salinity	Recommended: 32 ± 2 ppt
Test dissolved oxygen	Recommended: > 4.5 mg/L
Test pH	Recommended: 7.8 ± 0.5
Test photoperiod	16 hours light: 8 hours dark
Test chamber	Glass Aquaria (9.5 L volume)
Replicates/treatment	5
Organisms/replicate	<i>M. nasuta</i> = 10, <i>N. caecoides</i> = 25
Exposure Volume	1 L sediment
Feeding	None
Water renewal	Flow-through 14±4 ml/sec

5.4 POTENTIAL CONTRIBUTING FACTORS TO TOXICITY

Additional testing is recommended to address acclimation of sediment to testing conditions. The acclimation efforts will focus on the four test composites and the reference composite. Deeply buried sediments that have been isolated from biogenic processes (deeper than 10

cm below mud line depths), and any of the sediment treatment composites that have pore water salinity values that are less than 20‰ should be considered for additional testing. These sediments may have additional contributions to toxicity that are related to the changes in microbial processes that occur under the new conditions established for toxicity testing. The acclimation process will be performed on an additional five replicates of each test composite sample and the reference composite samples. The testing will be conducted at the same time as the standardized tests. The only difference will be the time at which test organisms are added to the sediment treatments. In the standard tests, the organisms are added one day after the sediment is placed into test containers while the addition of test organisms in the acclimated samples will wait until acclimation to the testing conditions has occurred. The amount of time required for acclimation follows the ammonia cycle. Sediment taken from one environmental regime to another (e.g., fresh water to marine or from deep non-biogenic materials to biogenic surface material) undergoes natural microbial changes to accommodate to the new environment. A surrogate measure of the success of this process is to measure the overlying water ammonia concentration through time. The premise for using ammonia as a surrogate assumes that ammonia will increase in concentration until the microbial community adjusts to the new environment. Once the microbial community is established, the overlying water ammonia concentration will decrease to levels below species-specific threshold concentrations. Although, ammonia is a surrogate measure to indicate when the acclimation process is complete, acclimation of test sediment addresses other potential contributing factors including sulfide toxicity. After sediment acclimation, the test organisms are added to the test containers. The differences in survival and growth of test organisms between acclimated and unacclimated testing are attributed to the acclimation process. The premise of acclimation is that effects from the acclimated sediment represents contaminant related effects, effects from unacclimated sediment represent contributions from contaminants as well as effects observed from abrupt changes in for example, temperature or salinity.

5.5 SEAWATER FOR BIOASSAY TESTING

For the water-column tests, dredge site water from Douglas Harbor will be used to create the elutriate preparations. The elutriate preparations involves the use of the dredged material (sediment) and unfiltered dredging site water which are combined in sediment-to water volumetric ratio of 1:4. All other seawater used for the biological tests, including the flow-through studies, will come from the northern Hood Canal at Port Gamble, Washington. This seawater source has been used successfully on similar bioassay testing programs by the contracting team. Extensive testing on a variety of test species has shown that there is no significant potential for toxicity or bioaccumulation from these water supplies. The use of seawater from Port Gamble is allowed for in guidance provided in the ITM (1998- Table 8-1 and section 11.1.4). Good survival of organisms in control sediment has been achieved consistently in previous dredged material testing conducted by the laboratory.

6 PHYSICAL AND CHEMICAL ANALYSIS

Physical and chemical parameters to be measured in sediment for this testing program were selected to provide confirmatory data on potential chemicals of concern in the dredged material from Douglas Harbor in accordance with the ITM (USEPA/USACE 1998). Test and reference sediments will be analyzed for the parameters and target detection limits indicated in Table 9. All analytical methods used to obtain contaminant concentrations follow EPA or Standard Methods (SM; APHA/AWWA 1998).

Table 9. Chemical and Physical Measurements, Analytical Methods, and Detection Limits

Parameter	Method	Procedure	Sediment Target Reporting Limit (dry weight)	Water Target Reporting Limit	Tissue Target Reporting Limit (wet weight)
Grain Size	Plumb (1981)	Sieve/Pipette	1.0%		
Total Organic Carbon	ASTM D2579	Combustion IR	0.1%		
Percent Solids	EPA 160.3	Gravimetric	0.1%		
AVS/SEM	Allen et al 1991	ICP-MS	AVS: 0.0119 $\mu\text{mole/g}$ Cd: 0.0000661 $\mu\text{mole/g}$ Cu: 0.00257 $\mu\text{mole/g}$ Ni: 0.000512 $\mu\text{mole/g}$ Pb: 0.0000359 $\mu\text{mole/g}$ Zn: 0.000795 $\mu\text{mole/g}$ Hg: 0.000000278 $\mu\text{mole/g}$		
TCLP					
Ammonia	Standard Methods 4500 NH ₃ D ;ASTM Method D 1426-93 Test Method B; and USEPA Method 350.3	Ion Selective Method		0.5 mg/L	
Lipids	Bligh Dyer	Gravimetric			0.1%
Total Mercury (Hg) sediment and tissue	USEPA 7473	CVAA	0.002 $\mu\text{g/g}$		0.002 $\mu\text{g/g}$
Total Mercury (Hg) water	USEPA 1631	CVAF		0.2 (ng/l)	
Methyl Mercury (Hg) sediment, water	USEPA 1630	CVAF	0.00002 $\mu\text{g/g}$	0.03 (ng/l)	

6.1 PHYSICAL ANALYSES

To characterize the physical properties of the sediment, tests will be performed to predict the behavior of sediment after disposal and to compare reference and test sediment. Physical-chemical analyses of the sediment will include grain size, total organic carbon (TOC), and total solids.

Grain size is analyzed to determine the general size classes that make up the sediment (e.g., gravel, sand, silt, and clay). The frequency distribution of the size ranges (reported in millimeters [mm]) of the sediment will be reported in the final data report. Grain size will be conducted using the gravimetric procedure described in Plumb (1981). TOC, made up of volatile and nonvolatile organic compounds, will be determined as recommended in the ITM (USEPA/USACE 1998) or equivalent (modified SW846). This procedure involves dissolving inorganic carbon (carbonates and bicarbonates) with hydrochloric acid or sulfuric acid prior to TOC analysis (Plumb 1981). Total solids will also be measured to convert concentrations of the chemical parameters from a wet-weight to a dry-weight basis. Percent solids will be determined by USEPA Method 160.3 (USEPA 2001).

Acid Volatile Sulfide (AVS) and Simultaneously Extracted Metals (SEM) in sediments follow the published procedure (Allen et al. 1991) for the analysis of acid volatile sulfide (AVS) in sediment and total sulfide in aqueous samples. For sediment samples, sulfide is volatilized

after the addition of acid. The acid produced in this step is then analyzed for simultaneously extractable metals (SEM) when analyzing sediments; metals become soluble during the acidification step. As a precipitant with heavy metals, sulfide is fundamental in the determination of the bioavailability of metals in anoxic sediment. When the molar ratio of SEM to AVS exceeds one, the metals are potentially bioavailable. This method quantifies the concentration of AVS and results in an SEM extract which is analyzed by ICP-MS for Cd, Cu, Ni, Pb, and Zn and by CVAF for Hg. These metals form the most common sulfides. These data are then used to assess the concentration of metals associated with sulfide.

Acid volatile sulfides analysis uses a colorimetric method in which the sulfide in the sample is converted to hydrogen sulfide by the addition of hydrochloric acid at room temperature. The hydrogen sulfide (H₂S) is purged from the sample by an inert gas and trapped in a sodium hydroxide (NaOH) solution. With the addition of a mixed-diamine reagent (MDR), the sulfide is converted to methylene blue and measured on a spectrometer. The acid-sediment slurry is decanted into a centrifuge tube and centrifuged to settle the sediment. The supernatant is poured into an acid cleaned Teflon bottle, ready to be analyzed by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) for cadmium (Cd), copper (Cu), nickel (Ni), lead (Pb), silver (Ag) or zinc (Zn) following a modification of EPA Method 1638; and by Cold Vapor Atomic Fluorescence (CVAF) for Hg (if requested) following EPA Method 1631.

Toxicity Characteristic Leaching Procedures (TCLP) analysis will follow methods described in EPA Method 1311 (Toxicity Characteristic Leaching Procedure). Briefly, 10 grams of sediment will be added to 200 ml of extraction fluid (weak acetic acid and NaOH) in a 250 ml acid-cleaned teflon bottle (as described in the method). The samples will be slowly rotated end-over-end for 18 hours in a rotary agitation apparatus. The extracts will be then filtered at 0.45 µm and acidified to 0.2% HNO₃ to preserve. The samples will be analyzed by ICP-MS for Cd, Cu, Ni, Pb, and Zn by ICP-MS (EPA Method 1638M) and for Hg by EPA Method 1631e.

Ammonia will be measured in the overlying water and the in the pore water of the biological tests following methods referenced in Table 9. Ammonia concentrations can affect the test organisms if the levels are above established threshold levels. If ammonia concentrations are above threshold levels, then the agencies will be notified and appropriate corrective action may be taken to reduce ammonia concentrations prior to testing. In addition, ammonia reference toxicant tests will be conducted for each test species

6.2 METHYL MERCURY IN WATER, AND SEDIMENT BY COLD VAPOR ATOMIC FLUORESCENCE (EPA METHOD 1630 MOD)

The method used for methyl mercury (Hg) follows Bloom (1989) for the determination of methyl mercury in a wide range of biological and geological matrices. This CVAF technique is upon emission of 254 nm radiation by excited Hg atoms in an inert gas stream. This method is currently contained in 1600 series for trace metals analysis (EPA Method 1630).

Sediment and pore water samples are distilled in Teflon vessels using the methods of Horvat et al. (1993). Alternatively, sediment samples can also be prepared for analysis using the method of Bloom et al. (1997). This new extraction technique avoids the methylation artifact sometimes produced in sediment sample containing high levels of inorganic mercury and organic carbon. An ethylating agent is added to the digestate or

distillate to form a volatile methyl-ethyl mercury derivative and the derivative is purged onto graphitized carbon traps as a means of pre-concentration and interference removal. The mercury species are then separated using isothermal chromatography, broken down to elemental mercury by means of pyrolysis, and detected using a CVAF detector as described in Bloom and Fitzgerald (1988). The typical detection limit is 0.00002 µg/g for sediment (0.02 ppb), and 0.03 ng/l (0.03 ppt) for water.

6.3 TOTAL MERCURY IN WATER BY COLD VAPOR ATOMIC FLUORESCENCE (EPA METHOD 1631)

EPA Method 1631 is used routinely for the analysis of total mercury in water. This method uses a CVAF technique, based on the fluorescence of excited Hg atoms in an inert gas stream at 254 nm wavelength (Bloom and Creelius 1983). To determine total mercury, water samples are oxidized with bromine monochloride, which breaks down organo-mercury bonds. Mercuric ions in the oxidized sample are reduced to Hg with SnCl₂, and then are purged onto a gold trap as a means of pre-concentration and interference removal. Mercury vapor is thermally desorbed into the fluorescence pathway. Fluorescence (peak area) is proportional to the quantity of mercury collected, which is quantified using a standard curve as a function of the quantity of sample purged. The typical detection limit for total mercury is 0.2 ng/l as Hg or 0.2 parts per trillion.

6.4 TOTAL MERCURY IN TISSUE AND SEDIMENT BY COLD VAPOR ATOMIC ABSORPTION (EPA METHOD 7473)

The analysis of total mercury in tissue and sediment employs a CVAA technique based on the absorption of 254 nm radiation by excited Hg atoms in an inert gas stream. To determine total mercury, a known mass of each sample is combusted at 750°C. The evolved Hg ions are then swept into the absorption pathway. Absorption (peak area) is proportional to the quantity of mercury collected, which is quantified using a standard curve as a function of the quantity of sample purged. This method quantifies all mercury in the sediment including lithologic mercury. The typical detection limit for the method is 0.002 µg/g as Hg or two parts per billion.

6.5 BIOACCUMULATION TISSUE CHEMISTRY

Total mercury analysis of tissues will be performed to demonstrate the availability of sediment contaminants for accumulation by test organisms. Tissue composites from each replicate will be analyzed separately.

7 QUALITY ASSURANCE/QUALITY CONTROL

The Quality Assurance/Quality Control requirements for this project provide confidence in the data results through a system of quality control checks on data collection methods, laboratory analysis, data reporting, and appropriate corrective actions to achieve compliance with established performance and data quality criteria. This section presents the QA/QC procedures used to ensure that project data are defensible and usable for their intended purpose.

7.1 QA/QC OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective is to develop and implement procedures for field sampling, chemical and biological laboratory analyses, and reporting that provide data of a quality consistent with its intended use. This section defines the project specific goals for precision,

accuracy, sensitivity, completeness, representativeness, and comparability of field sampling and laboratory analyses.

7.1.1 Definitions

PRECISION

Precision is the measure of the reproducibility among individual measurements of the same property, usually under similar conditions. Precision is assessed by performing multiple analyses on a sample and is expressed as a relative percent difference (RPD) when duplicate analyses are performed and as a percent relative standard deviation (%RSD) when more than two analyses are performed on the same sample (i.e., triplicates). Precision is assessed by duplicate analyses of spiked samples for all parameters, except when reference materials are not available or spiking of the matrix is inappropriate; in these cases, precision is assessed by duplicate analysis of unspiked matrix (laboratory duplicate). Precision measurements can be affected by the nearness of a chemical concentration to the method detection limit, where the percent error (expressed as either %RSD or RPD) increases. The equations used to express precision are as follows:

$$RPD = \frac{\text{abs}(\text{measured value} - \text{measured duplicate value})}{(\text{measured value} + \text{measured duplicate value}) \div 2} \times 100$$

$$\%RSD = (SD/D_{\text{ave}}) \times 100$$

Where: SD= standard deviation
D= sample value
D_{ave} = average sample value
n = number of samples

ACCURACY

Accuracy is an expression of the degree to which a measured or computed value represents the true value. Accuracy may be expressed as the percent difference between two measured values, as a percentage of the true or reference value, or as a percent recovery in those analyses where reference materials are not available and spiked samples are analyzed. The equations used to express accuracy are as follows:

$$\text{Percent Difference} = \frac{\text{measured value} - \text{true value}}{\text{true value}} \times 100$$

For reference materials:

$$\text{Percent of true value} = \frac{\text{measured value}}{\text{true value}} \times 100$$

For spiked samples:

$$\text{Percent Recovery} = \frac{\text{sample spike result} - \text{unspiked sample result}}{\text{amount of spike added}} \times 100$$

REPRESENTATIVENESS

Representativeness expresses the degree to which data accurately and precisely represent an environmental condition. For this program, the substances selected for analysis have been identified as the potential hazardous substances related to the site.

One program objective is the collection of samples that are representative of the matrix from which they are collected. Achievement of this goal relies on the use of standard and proven sampling procedures designed to obtain representative samples.

COMPARABILITY

The QA objective for comparability can be used to make valid comparisons with data that may be generated in the future. This objective involves the analysis of environmental samples collected during the sampling program in a manner that produces results comparable to results that would be obtained by another laboratory using the same procedures.

COMPLETENESS

Completeness is a measure of the amount of data that is determined to be valid in proportion to the amount of data collected. Completeness will be calculated as follows:

$$\text{Completeness} = \frac{\text{number of valid measurements}}{\text{total number of data points planned}} \times 100$$

The objective for data completeness for all measurement parameters will be 80 to 85 percent (EPA 1987). Data that are qualified as estimated because the quality control criteria were not met will be considered valid for the purpose of assessing completeness. Data that have been qualified as rejected will not be considered valid for the purpose of assessing completeness.

7.2 FIELD SAMPLING QA/QC

Field sampling data are assessed on comparability, representativeness, and completeness. Accuracy and precision of field data are achieved by use of standardized methods of locating sampling points such as differential Global Positioning Systems, with visual verification to known landmarks. Comparability and representativeness for field sampling are achieved by use of standardized sampling equipment appropriate for the sampling location. Completeness is measured as defined above.

Field logbooks provide documentation of all sample collection activities performed. As such, entries are described in as much detail as possible so that persons going to the project site could reconstruct a particular sampling event. Logbooks are assigned to field personnel and stored in the project file when not in use. Each logbook is identified by a project-specific number. The title page of each notebook will contain: (1) person or organization to which the book is assigned, (2) project name, and (3) start and end dates. At the beginning of each field day, the date, start time, weather, names of sampling and/or investigative personnel present, is entered and signed by the person making the entry.

Measurements made and information on samples collected are recorded in the logbook. All entries are made in ink and no erasures are made. If an incorrect entry is made, the

information is crossed out with a single strike mark. Wherever a sample is collected or a measurement is made, a detailed description of the location, with relevant information such that the sampling point can be relocated or mapped at a later time. Location information will include GPS coordinates; any appropriate reference points and distance measurements. Any photographs taken of the station are also documented. Equipment used to make field measurements are identified, along with the date of calibration.

A description of the equipment used to collect samples is entered, along with the date and time of collection, sample description, depth from which sample was collected, volume and number of containers. Sample identification numbers will be assigned during sample collection. Duplicate samples will receive a separate sample number and will be noted under the sample description.

Sample containers are *provided* by the analytical laboratory, who maintain documentation of the manufacturer, grade, lot number and/or other identifying information regarding preservatives added to sample containers. Chain-of-custody forms are maintained for each sample collected and the procedures used for chain of custodies were described in Section 4.3.

7.3 TOXICITY TESTING

The quality assurance objectives for toxicity testing conducted by the testing laboratory are provided in detail in the ITM (USEPA/USACE 1998). These objectives for accuracy and precision involve all aspects of the testing process, including the following:

- Water and sediment sampling and handling
- Source and condition of test organisms
- Condition of equipment
- Test conditions
- Instrument calibration
- Use of reference toxicants
- Record keeping
- Data evaluation

The sensitivity of the test organisms relative to established laboratory control charts will be evaluated using reference toxicant tests. The reference toxicant LC₅₀ or EC₅₀ should fall within two standard deviations of the historical laboratory mean. Water quality measurements will be monitored to ensure that they fall within prescribed limits and corrective actions will be taken if necessary. All limits established for this program meet or exceed those recommended by USEPA.

Finally, all data collected and produced will be recorded on approved data sheets, which will become part of the permanent data record of the program. If any aspect of a test deviates from protocol, the test will be evaluated to determine whether it is valid according to the regulatory agencies responsible for approval of the proposed permitting action.

Toxicity tests incorporate standard QA/QC procedures to ensure that the test results are valid. Standard QA/QC procedures include the use of negative and positive controls, the

use of testing replicates and the measurement of water quality parameters daily during testing.

There is no established accuracy or precision requirement for toxicity tests. Acceptable accuracy levels are generally assessed by the calibration of water quality instruments, the use of certified standards, and the establishment of acceptable water quality testing parameters. For example, water quality is monitored and, adjusted if necessary, throughout testing in at least one test replicate. Parameters that fall outside of acceptable test ranges may require corrective action. Deviations from water quality testing ranges do not necessarily fail the test; however, the potential impact on test exposures will be discussed.

Test organism behavior is visually monitored for each test chamber. The system is evaluated by conducting concurrent tests with negative control sediment. Adequate organism survival, as specified in the ITM (USEPA/USACE 1998), indicates a healthy testing population. Control survival is species and method specific but survival of <70% does not necessarily fail the test; however, it is an indication that the test system and test organisms should be further evaluated.

To ensure that each test chamber contains the appropriate number of test organisms, a second technician checks the number of organisms in each transfer cup prior to placement in the test chamber. Duplicate counts are performed at test termination. Random allocation of test organisms and testing chambers is conducted to remove any bias associated selectively picking the strongest organisms first or any bias associated with location of test chambers.

Representativeness is maintained during toxicity testing by ensuring that sediment is held in the dark at 4°C until needed for testing. Test sediment is homogenized prior to placement in test chambers. All test chambers and utensils are washed in warm soapy water, DI rinsed, acid-rinsed, and solvent rinsed. Water quality parameters are measured daily in at least one replicate per treatment. A calibration check is performed daily on all water quality instruments.

The QA objective for comparability can be used to make valid comparisons with data that may be generated in the future from the project site. This objective involves the analysis of environmental samples collected during the sampling program in a manner that produces results comparable to results that would be obtained by another laboratory using the same procedures. Comparability of the data can be assessed by the use of standard materials traceable to the National Institute of Standards and Technology (NIST), or approved suppliers, such as established vendors for the purchase of test organisms, the use of a positive control for toxicity tests, the use of standardized, regulatory approved procedures for sample collection and sample analysis, and analysis of quality control samples to validate the analytical results.

The test performance criteria will follow the guidance described in the ITM (USEPA/USACE1998) Section 6.1 – 6.3. The performance criteria for this project have been taken **directly** from the ITM manual and are provided for each test.

WATER- COLUMN TESTING PERFORMANCE CRITERIA (ITM ONLY):

- The 100% dredged material elutriate toxicity is not statistically higher the dilution water 0%, then the dredged material is not predicted to be acutely toxic to water-column organisms.

- The concentration of dissolved and suspended contaminants, after allowance for initial mixing, does not exceed 0.01 of the toxic concentration expressed as the EC or LC₅₀, beyond the boundaries of the mixing zone. Therefore the dredged material is predicted not to be acutely toxic to water column organisms. However, benthic impacts have to be considered. If information warrants, it is acceptable to determine water column effects at Tier III and benthic effects at another tier.
- The concentration of dissolved plus suspended contaminants, after allowance for mixing, exceeds 0.01 of the toxic (LC or EC₅₀) concentration beyond the boundaries of the mixing zone. Therefore, the dredged material is predicted to be acutely toxic to water column organisms.

Water-column tests are not routinely conducted as part of the Dredged Material Evaluation and Disposal Procedures (Users Manual), therefore interpretative criteria for the water-column test will follow guidance in ITM.

BENTHIC TOXICITY TESTING PERFORMANCE CRITERIA (ITM AND PSEP GUIDANCE)

ITM Performance Criteria for benthic tests are predicted to be acutely toxic to benthic organisms when mean test organism mortality:

- Is statistically greater than in the reference sediment **and**
- Exceeds mortality in the reference sediment by at least 10% (...20% value for lethality can be used for amphipods, *Ampelisca abdita*, *Rhepoxynius abronius*, or *Eohaustorius estuarius* (Swartz et al, 1985; Mearns et al., 1986; SAIC, 1992 a,b). \

Interpretative Criteria for the amphipod test based on the Dredged Material Evaluation and Disposal Procedures (Users Manual) (July 2008):

- Mean test mortality is greater than 20% (absolute) over the mean negative control response, and mean test mortality is greater than 10% (dispersive) or 30% (non-dispersive) over the mean reference sediment response and statistically significant compared to reference (alpha = 0.5) sediment is considered a hit

BIOACCUMULATION PERFORMANCE CRITERIA BASED ON TISSUE COMPARISONS (ITM, PSEP, AND PROJECT SPECIFIC GUIDANCE)

ITM performance guidance:

- Tissue concentrations of contaminants are not statistically less than the FDA levels. Therefore, the dredged material is predicted to result in benthic bioaccumulation of contaminants.
- Tissue concentrations of all contaminants are statistically less than FDA levels or there are no levels for the contaminants. In this case, the information is insufficient to reach a conclusion with respect to benthic bioaccumulation of contaminants. The dredged material needs to be further evaluated in Tier III as described in the subsequent bullets.

- Tissue contaminant concentrations following exposure to dredged material which are statistically less than FDA levels, or for which there are no such levels, are compared to tissue contaminant concentrations for organisms similarly exposed to reference sediment:
 - Tissue concentrations of contaminants of concern in organisms exposed to dredged material do not statistically exceed those of organisms exposed to the reference sediment; therefore, the dredged material is predicted not to result in benthic bioaccumulation of contaminants. However, benthic toxicity effects also have to be considered.
 - Tissue concentrations of contaminants of concern in organisms exposed to dredged material statistically exceed those of organisms exposed to reference material. In this case, the conclusion regarding benthic bioaccumulation of contaminants would be based upon technical evaluations that emphasize the various factors deemed appropriate in a particular region. Additional Tier IV may be required.
- Tissue concentrations are above FDA limits but are not statistically different from the reference (or disposal) site. This situation represents an exceptional case, which can only be dealt with at the regional level.

Interpretive guidance for the bioaccumulation test based on the Dredged Material Evaluation and Disposal Procedures (Users Manual) (July 2008):

- Numerical test interpretation guideline or target tissue levels (TTLs) were derived based on human health considerations. The TTLs are allowable tissue concentrations for the bioaccumulation contaminants of concern that were either derived from human-health risk assessments or from FDA action levels. The TTL for mercury is the FDA action level of 1.0 mg/kg wet weight. Interpretation of bioaccumulation results using the one-tailed one-sample t-test (alpha level = 0.05). For undetected chemicals, a concentration equal to one-half the detection limit is used.
 - If the mean tissue concentration of the contaminant of concern is greater than or equal to the TTL, then statistical testing is not required. The conclusion is that the DMMU is not acceptable for aquatic disposal.
 - If the mean tissue concentration of the contaminant of concern is less than the TTL, then a one-tailed, one-sample t-test is conducted and the DMMU is acceptable if the results are not statistically significant.

For an assessment of ecological effects, the results of the test sediment bioaccumulation responses will be compared with the bioaccumulation responses of the reference sediment. Significant bioaccumulation of chemicals of concern in test species relative to reference areas may demonstrate a potential for food-web effects.

- If the results of a statistical comparison show that the tissue concentration of the chemical of concern in test sediment is statistically higher (one-tailed, one-sample, t-test alpha level = 0.1) than the reference sediment, the DMMU will

need to be evaluated further to determine the potential ecological significance of the measure tissue resides.

In addition to the performance criteria provided in both the ITM and the PSEP, ADEC has requested that the bioaccumulation data be reviewed using an Alaska specific tissue concentration of total mercury of 0.32 ppm wet weight. This value was chosen based on region-specific information (State of Alaska Division of Public Health, 2007) and the fish consumption practices for Alaskans. The bioaccumulation data will also be reviewed and compared using this project specific total mercury value for tissues.

7.4 ANALYTICAL CHEMISTRY QA/QC

7.4.1 Data Quality Objectives

Table 10 lists specific data quality objectives for each group of analyses to be performed. The parameters used to assess data quality are precision, accuracy, representativeness, comparability, and completeness.

Table 10. Data Quality Objectives for Mercury Analysis

QC Measurement	Frequency	Acceptable Limits	Corrective Action
Total Mercury in Sediment and Tissue			
Method blank	1 per ≤20 samples	< 5 times the MDL	Reanalyze. If confirmed and all samples are >10 times the blank, no corrective action is required. If samples are <10 times the blank, the batch must be reanalyzed
Certified/Standard Reference Samples	1 per ≤20 samples	80-120% of certified value	Reanalyze. Failure to meet the acceptable limits shall be reported in the Data Summary. Failure of multiple data quality objectives requires the samples to be reanalyzed.
Matrix Spike	1 per ≤20 samples	80 – 120% recovery	Reanalyze. Failure to meet the acceptable limits shall be reported in the Data Summary. Failure of multiple data quality objectives requires the samples to be reanalyzed.
Replicate Precision	1 per ≤20 samples	20% for analytes > 3 times the MDL. No more than 35% of all RPDs can be >25%	Reanalyze. Failure to meet the acceptable limits shall be reported in the Data Summary. Failure of multiple data quality objectives requires the samples to be reanalyzed.
Initial and Continuing Calibration Verification	Every 10 samples	10%/20% of initial calibration	Reanalyze. If subsequent ICV or CCV still fail, rerun the calibration curve and all samples analyzed after the last passing calibration check.
Total Mercury in Aqueous Samples			
Method blank	1 per ≤20 samples	< 5 times the MDL	If confirmed and all samples are >10 times the blank, no corrective action is required. If samples are <10 times the blank, the bath must be reanalyzed
Certified/Standard Reference Samples	1 per ≤20 samples	77-123 % of certified value	Failure to meet the acceptable limits shall be reported in the Data Summary. Failure of multiple data quality objectives requires the samples to be reanalyzed.

Table 10. Data Quality Objectives for Mercury Analysis (Continued)

QC Measurement	Frequency	Acceptable Limits	Corrective Action
Matrix Spike	1 per ≤20 samples	71- 125 % recovery	Failure to meet the acceptable limits shall be reported in the Data Summary. Failure of multiple data quality objectives requires the samples to be reanalyzed.
Replicate Precision	1 per ≤20 samples	21% for analytes > 3 times the MDL. No more than 35% of all RPDs can be >21%	Failure to meet the acceptable limits shall be reported in the Data Summary. Failure of multiple data quality objectives requires the samples to be reanalyzed.
Initial and Continuing Calibration Verification	Every 10 samples	<15% of initial calibration	If subsequent ICV or CCV still fail, rerun the calibration curve and all samples analyzed after the last passing calibration check.
Methyl Mercury in Sediment, and Aqueous Samples			
Method blank	1 per ≤20 samples	< 5 times the MDL	If confirmed and all samples are >10 times the blank, no corrective action is required. If samples are <10 times the blank, the bath must be reanalyzed
Certified/Standard Reference Samples	1 per ≤20 samples	66-123 % of certified value	Failure to meet the acceptable limits shall be reported in the Data Summary. Failure of multiple data quality objectives requires the samples to be reanalyzed.
Matrix Spike	1 per ≤20 samples	65- 135 % recovery	Failure to meet the acceptable limits shall be reported in the Data Summary. Failure of multiple data quality objectives requires the samples to be reanalyzed.
Replicate Precision	1 per ≤20 samples	35% for analytes > 5 times the MDL. No more than 35% of all RSDs can be >35%	Failure to meet the acceptable limits shall be reported in the Data Summary. Failure of multiple data quality objectives requires the samples to be reanalyzed.
Initial and Continuing Calibration Verification	Every 10 samples	<20% of initial calibration	If subsequent ICV or CCV still fail, rerun the calibration curve and all samples analyzed after the last passing calibration check.

The QA objective with respect to accuracy, precision, and sensitivity of laboratory data is to achieve the QC acceptance criteria of the testing protocols. In general, the accuracy and precision criteria are those stipulated by the most recent versions or modifications of USEPA SW-846.

To assess the quality of data resulting from the analytical chemistry program, the following QA/QC measures will be included in the sampling program:

- Procedural blanks will be performed to check for artifacts associated with sample extraction and analysis. Procedural blanks will be performed at a rate of one per 20 samples or each analytical batch.
- Sufficient sample volume will be supplied to the laboratory in order to perform matrix spike/matrix spike duplicate (MS/MSD). MS/MSD samples will evaluate analytical accuracy and precision. MS/MSD samples will be performed at a frequency of one per 20 (5%) investigative samples or each analytical batch.
- Laboratory duplicate samples will be performed to check precision of the analytical process. Lab duplicate samples will be conducted at a frequency of one per 20 (5%) investigative samples or one per analytical batch.

- A standard reference material will be conducted when appropriate to evaluate the analytical accuracy. An SRM sample will be conducted at a frequency of one per 20 samples (5%) or one per analytical batch.

7.5 RECORD KEEPING

Each sample shipped to the laboratory for analysis is given a unique identification number that is used by the analytical laboratory. The laboratory sample custodian records the client name, number of samples and date of receipt of samples in the Sample Control Logbook.

The laboratory is responsible for maintaining analytical logbooks and laboratory data as well as an updated sample inventory for submittal to NewFields upon request. Raw laboratory data produced from the analysis of samples submitted for this program are inventoried and archived by the laboratory for a period of five years. The laboratory will advise NewFields 60 days prior to expiration of the five years. NewFields will advise the laboratory regarding the need for additional storage prior to expiration of the 60 days.

7.6 SAMPLE STORAGE

After the sample custodian has completed the chain-of-custody forms and entries in the incoming sample log, all samples are stored in the appropriate cold storage locations. All samples are stored within an access controlled custody room and will be maintained at approximately 4°C until all analytical work is complete. If samples are to be archived for long-term storage, they will be maintained at approximately -20°C.

7.7 CALIBRATION PROCEDURES AND FREQUENCY

All laboratory instruments will be calibrated prior to use. The calibration procedures will follow standard manufacturer's instructions to assure that the equipment is functioning within acceptable tolerances established by the manufacturer requirements.

7.8 DATA REDUCTION, VALIDATION, ASSESSMENT, AND REPORTING

7.8.1 General

Each laboratory performs laboratory data reduction and in-house validation under the direction of the laboratory's QA/QC Officer. The laboratory QA/QC Officer is responsible for assessing data quality and advising NewFields of any qualifications, based on the QC criteria outlined in appropriate methods, which would caution the data user of potential unreliability. Laboratory data reduction, validation, and reporting will be conducted as follows:

- If necessary, any sample not meeting minimal QC acceptance criteria specified by the laboratory protocol will be re-analyzed.
- Toxicity test interpretation consists of endpoint comparisons of test sediments to the measurements observed in the controls and if appropriate reference sediments on an absolute percentage basis, as well as statistical comparison between the test and reference endpoints, where appropriate. Test interpretation follows the guidelines established in the ITM (USEPA/USACE 1998). Summary statistics such as means and standard errors for response variables will be generated for each sample.

- Data generated are checked by the responsible laboratory technician and are reviewed independently by another analyst.
- The laboratory supervisor reviews the results and the quality control acceptance criteria specified in the referenced analytical methods.
- Upon completion of all reviews and acceptance of the data by the laboratory manager, a computerized report is generated and sent to the laboratory QA/QC Officer.
- The laboratory QA/QC Officer completes a thorough inspection of all data and in conjunction with the laboratory manager makes any necessary revisions to the report.
- Upon acceptance of the preliminary reports by the laboratory QA/QC Officer, final reports are generated and signed by the laboratory manager.

NewFields' QA/QC Officer conducts a review of laboratory data reduction and reporting. The data reviewed include all laboratory results, field blank data, field duplicate data, and recovery data for surrogate compounds and QC analyses. The material is checked for legibility, completeness, correctness, and the presence of required dates, initials, and signatures. The results of these checks are summarized and reported to NewFields' Project Manager, noting any discrepancies and their effect upon the reliability of the data. All information generated from QA/QC checks will be discussed in the final report.

The laboratory conducting the toxicity tests is responsible for internal checks on data reporting and corrects errors identified during the quality assurance review. The laboratory is required to report results that include all information recommended by the test protocols for quality assurance review, as follows:

- Test methods used for toxicity testing and statistical analyses
- Source of control sediment and method for collection, handling, shipping, storage, and disposal of sediment.
- Source of testing water including a description of any pretreatment, and results demonstrating survival and growth of test organism in test water
- Source, history, and age of test organisms and if appropriate culturing information, or collection information. Records should include information regarding taxonomic identification of test organisms.
- Source of food composition and procedures used to prepare and dispense food to test chambers.
- Description of experimental design including test setup, test monitoring, and test termination. Water quality and observation records should be summarized and included in report.
- Methods used for physical and chemical characterization of test sediment and surface waters.
- A table of biological data for each sample, including negative and positive control information.
- Methods used for statistical analysis.

- A description of any deviations from the methodology or problems with the process and procedures of analyses.
- Original data sheets for water quality, survival, growth, reburial, abnormalities, reference toxicant, and statistics as applicable by test protocol.
- Chain-of-custody records.
- References and literature.

7.9 SYSTEM AND PERFORMANCE AUDITS

Internal system audits are performed *if necessary* throughout the duration of this program. The objectives of the system and performance audits are to ensure that the quality assurance program is implemented according to the specified requirements, to assess the effectiveness of the quality assurance program, to identify non-conformances, and to verify that any identified deficiencies are corrected. The Project Manager or QA/QC officer may require at his/her discretion any of the following audits be conducted. Audits will be performed if the Project Manager or QA/QC officer has reason to suspect that this plan is not being followed. If any significant deviations from the QAPP are documented, corrective action measures are immediately implemented and documented as detailed in Section 7.14.

7.10 LABORATORY SYSTEM AUDIT

A laboratory systems audit may be conducted by the NewFields QA/QC Officer or qualified designee during analysis of initial sample shipments sent to the laboratory. If a laboratory systems audit is conducted, the NewFields QA/QC Officer, in conjunction with the Project Manager representing the subcontracted laboratory, will ensure that documentation is available to verify that instrumentation required by the project designated method is used in the analysis of samples, and that the instruments were functioning properly. Prior to the laboratory information systems audit, NewFields will review the analytical methods proposed for use and the laboratory Standard Operating Procedures prepared from these methods. The Laboratory Project Manager or his/her designee would make changes as necessary following the initial laboratory systems audits and confirm orally within five working days and in writing within ten working days to the NewFields Project Manager and/or NewFields QA/QC Officer or designee that the laboratory meets all requirements of the measurement system.

A toxicity testing laboratory audit may be conducted by NewFields QA/QC Officer or qualified designee during testing phase of the program. The NewFields QA/QC Officer, in conjunction with the Project Manager will ensure that documentation is available to verify that established methods are being followed. Specific areas of evaluation may include organism culturing and holding, proper care, and maintenance of instrumentation, and a review of testing procedures.

7.11 OFFICE SYSTEM AUDIT

Office system audits are conducted as part of the overall NewFields Quality Assurance Program. The office audit consists of reviewing the project file and verifying that data collected is being presented, reviewed, and filed in accordance with this QAPP and the NewFields QA Manual. The NewFields Project QA officer is responsible for conducting office system audits as part of his/her regular duties. He/she will notify the NewFields Project Manager in writing of the audit findings within ten working days of the audit. The

NewFields Project Manager will implement corrective actions if required, based on the results of the office systems audit.

7.12 PERFORMANCE AUDITS

Performance audits are usually conducted after data production systems are operational and are generating data. Performance audits consist of two types: internal and external. The performance audit is a quantitative evaluation of the measurement systems used for a given sampling program. It requires testing the analytical measurement systems with samples of known composition or behavior to evaluate accuracy and precision. Internal performance audits may be carried out by or under the auspices of the laboratory QA/QC officer without the knowledge of the analyst. The type and frequency of internal audits conducted by the analytical laboratory are detailed in their SOPs (presented separately).

7.13 PREVENTATIVE MAINTENANCE

As part of their QA/QC Program, a routine preventive maintenance program is conducted by the analytical laboratory to minimize the occurrence of instrument failure and other system malfunctions. The laboratory has an internal group which performs routine scheduled maintenance and to repair or coordinate with the vendor the repair of all instruments. The laboratory also has instruments that will serve as backup to those instruments used for this project.

Field and laboratory instruments will be checked and calibrated prior to use and batteries will be charged daily, where applicable. Calibration and maintenance information will be recorded in the instrument logbook. Manufacturer's operating manuals will be kept on-site for instruction on calibration and maintenance. For field instrumentation, rental units will be obtained should instrument failure occur.

7.14 CORRECTIVE ACTION

Corrective or preventive action is required when existing or potential conditions are identified that may have an adverse impact on data quantity or quality. The ultimate responsibility for maintaining quality throughout the project rests with the Project Manager and the QA/QC Officer. The routine operation of the quality assurance program, however, falls upon the technical staff, and the subcontracted laboratory's Quality Assurance Officers and Project Managers. Any member of the project staff who identifies a condition adversely affecting quality will initiate corrective action by notifying the Project Manager or QA/QC Officer verbally or in writing. A written communication identifying the condition and an explanation of how it may affect data quality or quantity is preferable for initiating the corrective action process. Corrective actions will also be initiated based on system or performance audits.

Any non-conformances with the established quality control procedures are identified and controlled. No additional work that is dependent on the non-conforming activity is performed until the identified non-conformance is corrected.

7.15 FIELD CORRECTIVE ACTION

The Project Manager reviews the procedures implemented in the field for consistency with the established protocols. Sample collection, preservation, and labeling, etc. are checked for completeness. Where procedures are not strictly in compliance with the established

protocol, deviations will be documented. Corrective actions, if required, will be defined by the Project Manager and documented as appropriate. Upon implementation of the corrective action, the Project Manager will provide the QA/QC Officer with a written memo documenting field implementation. The memo will become part of the project file.

7.16 LABORATORY CORRECTIVE ACTION

The laboratory's QA/QC Officer and NewFields's Data Validator(s) will review the laboratory data generated to ensure that all quality control procedures have been performed as specified in the protocol. Where QC criteria fall outside of the acceptable ranges, deficiencies will be reported to the NewFields Project Manager and QA/QC Officer. Corrective actions will be defined by the Project Manager in coordination with the Laboratory Project Manager and documented as appropriate. The laboratory has responsibility for Laboratory Corrective Action in accordance with the procedures identified in the Methods.

Corrective actions may be necessary if:

- QC data (i.e., spike recoveries, duplicate results, calibrations, instrument tunes, etc.) are outside the warning or acceptable windows for precision.
- Blanks contain contaminant concentrations above the required quantitation limit of any target compound.
- There are unusual changes in detection limits (i.e., if detection limits are substantially higher or lower than what is expected for a given parameter within a given matrix).
- Deficiencies detected during internal or external audits, or from the results of performance evaluation samples.
- Water quality test parameters consistently fall outside of established ranges
- Negative or positive control results vary significantly from established guidance

8 DATA REVIEW, MANAGEMENT, AND ANALYSIS

8.1 DATA REVIEW

All data will be reviewed and verified by participating team laboratories to determine whether all data quality objectives have been met, and whether appropriate corrective actions have occurred. NewFields' QA Officer (Lucinda Word) or her delegate will be responsible for the final review of all data generated.

8.2 DATA MANAGEMENT

All laboratories will supply analytical results in both hard copy and electronic formats. Laboratories will have the responsibility of ensuring that both forms are accurate.

After completion of the sediment data review by participating team laboratories, hard copy results will be placed in the project file at NewFields and the results in electronic format will be imported into NewFields' archive system.

8.3 DATA ANALYSIS

Data analysis will consist of tabulation and comparison with the reference site. Biological results will be compared to appropriate laboratory controls and reference results where applicable as designated in the ITM (USEPA/USACE 1998).

9 REPORTING

9.1 DRAFT AND FINAL REPORTS

After all results are received, statistical analyses completed, and all evaluations made, NewFields will prepare draft and final reports. These will include summaries of all activities associated with collecting, compositing, transporting, and chemically and biologically analyzing sediment samples. The chemical and biological data reports will be included as appendices. As a minimum, the following will be included in the final report:

- Summary of all field activities, including a description of any deviations from the approved SAP/QAPP
- Descriptions of each sample and all original core logs
- Plan view of the project showing the actual sampling locations
- Data results
- In addition to hard copies of field data, laboratory analysis results, and associated QA/QC data, electronic copies for all data will be stored at NewFields
- Discussion of the suitability of the proposed dredge material for unconfined open water disposal

9.2 QA/QC AND LABORATORY DATA REPORT

Analytical laboratories will provide a QA/QC narrative that describes the results of the standard QA/QC protocols that accompany analysis of field samples. All hard copies of results will be maintained in the project file at NewFields in Port Gamble and included in the final report. In addition, back-up copies of results generated by each laboratory will be maintained at their respective facilities. At a minimum, the laboratory reports will contain results of the laboratory analysis, QA/QC results, all protocols, and any deviations from the project SAP, and a case narrative of COC details.

10 SCHEDULE

Scheduling of proposed activities will be dependent on final approval of the SAP. Once initiated, field-sampling activities are anticipated to take approximately 1 week. Upon completion of the field sampling effort, chemical analysis of dredged material will be completed in approximately four weeks. All bioassays (solid phase and bioaccumulation) will be initiated within the 8-week holding time, as required in the ITM (USEPA/USACE 1998). Once all data have been collected and undergone QA/QC review, a draft report will be prepared and submitted to PND for review.

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