Integrated Field Screening for Rapid Sediment Characterization

August 2004
COST & PERFORMANCE REPORT
ESTCP Project: CU-9717

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<td>acre(s)</td>
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<tr>
<td>ASTM</td>
<td>American Society for Standard Tests and Methods</td>
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<td>California Environmental Protection Agency</td>
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<td>Environmental Security Technology Certification Program</td>
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<td>ETV</td>
<td>Environmental Technology Verification</td>
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<tr>
<td>FP</td>
<td>fundamental parameter</td>
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<td>FPXRF</td>
<td>Field portable x-ray fluorescence</td>
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<td>NAS</td>
<td>Naval Air Station</td>
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<td>NAVFAC</td>
<td>Naval Facilities</td>
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<td>nm</td>
<td>nanometer</td>
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<td>PAH</td>
<td>polycyclic aromatic hydrocarbon</td>
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<td>PCB</td>
<td>polychlorinated biphenyl</td>
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<td>PHNC</td>
<td>Pearl Harbor Naval Center</td>
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<td>photomultiplier tube</td>
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<td>ppm</td>
<td>part per million</td>
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<td>Remediation Innovative Technology Seminar</td>
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<td>remedial project manager</td>
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<td>relative standard deviation</td>
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<td>San Diego–Space and Naval Warfare Systems Center, San Diego</td>
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<tr>
<td>TBT</td>
<td>tributyltin</td>
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<td>TIO</td>
<td>Technology Innovation Office</td>
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<td>TOC</td>
<td>total organic carbon</td>
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<tr>
<td>UV</td>
<td>ultraviolet</td>
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<td>ultraviolet fluorescence</td>
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ACKNOWLEDGEMENTS

This demonstration project was co-funded through the Naval Facilities (NAVFAC) Y0187 program run by Andy Del Collo. We would also like to acknowledge the participation of various NAVFAC regulatory programs and personnel at NAS Alameda and Pearl Harbor Naval Complex where these demonstrations took place. The screening analysts that aided in the project were Brian Ayers, Joel Guerrero, Gunther Rosen, and Jaelyn D. Chock of SSC San Diego. Jaelyn D. Chock also assisted in the preparation of this Cost and Performance Report.

Technical material contained in this report has been approved for public release.
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1.0 EXECUTIVE SUMMARY

1.1 BACKGROUND

The Environmental Security Technology Certification Program (ESTCP) has an established program to accelerate acceptance and application of innovative monitoring and site characterization technologies that improve the way the nation manages its environmental problems. Space and Naval Warfare Systems Center, San Diego (SSC San Diego), will demonstrate an integrated methodology to facilitate acceptance of three field screening techniques to delineate chemical concentrations and potential biological effects of sediment contaminants.

Defining the nature and extent of contamination in marine sediments can be a difficult problem. Detailed site investigations require extensive sampling and subsequent laboratory analyses for both metal and organic contaminant chemistries. Additional laboratory analyses including several different types of bioassays are conducted to determine any possible adverse biological effects to organisms exposed to the sediment. Samples are often collected without any a priori knowledge of the nature and extent of contamination. Due to the high cost of all these laboratory analyses, samples taken for analysis are often limited. Zones of contamination in marine sediments can be missed, or, if located, overestimated or underestimated. For more detailed spatial information on extent of contamination, sites of interest must often be sampled and analyzed in an iterative manner. This approach can be prohibitively costly, slow, and labor intensive.

An alternative to this approach is to combine standard laboratory analyses with field screening using various techniques to characterize both the contaminant chemistry and any possible biological effects. By using near real-time screening techniques during the sampling procedure, the full extent of contamination and possible biological effects can be rapidly mapped. This also allows more informed selection of a subset of the screened samples to continue on for laboratory analyses to fully describe the nature of contamination and biological effects. The use of geostatistical procedures can provide the basis for selecting sampling strategies and aid in selecting the number of samples to be screened and determining which samples will also continue onto laboratory analysis (Chapter 9, “Double Sampling,” in Gilbert, 1987).

Field-portable x-ray fluorescence (FPXRF) spectrometry will be used to screen for metals of interest. Ultraviolet fluorescence (UVF) will be used to screen for polycyclic aromatic hydrocarbons (PAH). As a measure of the biological effects, or bioavailability, bioluminescent-based bioassay QwikSed will be used (see Section 2.0, Technology Description, for a discussion of techniques).

1.2 OBJECTIVES OF THE DEMONSTRATION

The primary objectives of this demonstration are to evaluate the three field screening technologies in the following areas: (1) their performance compared to conventional sampling and analytical methods; (2) data quality; (3) the logistical and economic resources necessary to operate the technologies; and (4) the range of usefulness in which the technologies can be
operated and integrated into a screening procedure that allows more efficient assessment of sediment sites. Secondary objectives for this demonstration are to evaluate the technologies for their reliability, ruggedness, and ease of operation. The fourth primary objective is important because current regulatory projects often rely on “blind” sampling, with little or no knowledge of how much volume of sediment each laboratory measurement represents. Cheaper screening techniques will allow more knowledgeable sample selection for laboratory analysis and, therefore, better insight into how representative these samples are. As additional screening techniques are developed, they may be incorporated into existing screening procedures.

1.3 REGULATORY DRIVERS

This project will demonstrate how field screening data can provide the regulatory community with supplemental information on the extent of contamination with more extensive and higher density data that is obtained both faster and cheaper than current practices normally allow.

For the metals copper, zinc, and lead, total individual metal levels are the regulatory criteria against which the screening technique will be evaluated. For PAHs, individual PAHs and their sum total are often used as the regulatory driver. Because not all total levels of contaminants are bioavailable, the screening bioassay can be used to infer what fraction of total contaminant is actually bioavailable. The screening bioassay will be evaluated against standard laboratory bioassay endpoints such as percent survival.

1.4 DEMONSTRATION RESULTS

The chemical screening results indicate these techniques provide Level 2 semiquantitative screening data. This means that a limited number of confirmatory samples are required from the site to generate a calibration relationship. This relationship can be used to make the screening data comparable to confirmatory laboratory data. The biological screening results indicate Level 1 qualitative screening data. This means the screening bioassay may predict whether other confirmatory bioassays are above or below a certain benchmark level, but may not quantify the amount. The main factors affecting cost and performance of the screening technologies are the number of samples that will be screened and how many will be sent on for laboratory confirmation. As the number of samples to be screened increases, the per-sample cost will decrease. The number of samples that go to the confirmatory laboratory can also affect cost and performance. Between 10 and 50% of the screened samples are usually sent on for costly laboratory confirmation. Site-specific calibration relationships between screening and laboratory data will carry more confidence as the number of laboratory analyses increases, but this will come at a higher cost. All of these factors affect cost and performance, and professional judgment must be exercised to optimize the screening operation (see Chapter 9 in Gilbert, 1987).
2.0 TECHNOLOGY DESCRIPTION

2.1 TECHNOLOGY DEVELOPMENT AND APPLICATION

X-ray fluorescence (XRF) technologies operate on the concept of energy dispersive XRF spectrometry, a nondestructive qualitative and quantitative analytical technique. Most field-portable XRF units use sealed radioisotope sources to irradiate samples with x-rays. Laboratory-grade XRF technologies generally use an x-ray tube to irradiate the samples with x-rays, and both the field portable and laboratory-grade technologies produce x-rays of known energies. By exposing a sample to an x-ray excitation source having energy close to, but greater than, the binding energy of the inner shell electrons of the metals, an inner shell electron is discharged. Electrons cascading in from outer electron shells fill the electron vacancies that result. Electrons in outer shells have higher energy states than inner shell electrons; therefore, to fill the vacancies, the outer shell electrons give off energy in the x-ray spectrum as they cascade down into the inner shell vacancies. There are three electron shells generally involved in the emission of x-rays during the XRF analysis of environmental samples: K, M, and L shell electrons. The emission of x-rays is termed x-ray fluorescence. Each metal gives off x-rays of specific energy levels. The specific type or energy of the emitted x-ray is unique to a given metal and is called a “characteristic” x-ray. By measuring the different energies of x-rays emitted by a sample exposed to an x-ray source, it is possible to identify and quantify the metals composition of a sample (Bertin, 1975; Russ, 1984).

Analyses for this demonstration will be performed using a TN Spectrace 9000® portable XRF spectrometer (TN Spectrace Instruments). The instrument contains three radioisotope sources—Fe-55, Cd-109, and Am-241—to provide the excitation x-rays. It has an electronically cooled solid-state mercury iodide detector for measuring the characteristic fluorescent x-rays. The instrument utilizes proprietary fundamental parameters (FP) algorithms, which eliminate the need for empirical calibration with site-specific standards.

Fluorescence can be used to measure the concentration of various organic analytes in addition to metals. Unlike metals, where high energy x-rays are required to generate fluorescence, PAHs require only ultraviolet (UV) light excitation to fluoresce visible light. Excitation light from a lamp is passed through an excitation filter that transmits light of the chosen wavelength range. The light passes through the sample, causing the sample to emit light (fluoresce) proportional to the concentration of the fluorescent molecule, PAH, in the sample. The emitted light is passed through another optical filter (emission filter) before reaching the detector (in this case, a photomultiplier tube). The excitation wavelength is chosen for strong absorption by the material under study and for minimal absorption by any interfering fluorescent materials that may be present. The photomultiplier and emission filters are also chosen so that they respond as much as possible to the light emitted by the material under study and as little as possible to the emission of any interfering fluorescent materials which may be present.

PAHs will be screened in this study by UVF on hexane solvent extracts of the sediments (Filkins, 1992; Owen et al., 1995). A Turner fluorometer (Turner Model AU-10 Digital Filter Fluorometer®) with a standard optical package (commonly used in routine water quality analyses) will be used to screen for total PAHs. This optical package from the manufacturer is
specifically designed for measurement of heavier weight PAH fluorescence with an excitation wavelength of 360 ± 10 nanometer (nm) provided by a quartz-halogen lamp. The detector system consists of a high gain, low noise photomultiplier tube with detection wavelength of 400-650 nm.

The QwikLite bioassay system was recently developed (Lapota et al., 1987) to measure the light output from bioluminescent dinoflagellates for assessment of toxic effects when exposed to many chemicals, individually or in compounds, effluents, and antifoulant coatings. Successful bioassays of this type have provided data on acute response as well as chronic effects (from 3 hours up to 11 days) on two species of dinoflagellate, *Pyrocystis lunula* and *Gonyaulax polyedra*. The basis of detection is to measure a light reduction from bioluminescent dinoflagellates following exposure to a toxicant. Bioluminescence is the production of light by living organisms due to an enzyme-catalyzed chemical reaction. Upon exposure to a toxicant, the dinoflagellates may shed an outer cell membrane called a theca and form a cyst. Consequently, light production decreases from the dinoflagellates. Encystment is a normal response by dinoflagellates to an unfavorable or stressful environment.

The QwikLite or QwikSed (the sediment version instrument of the QwikLite) bioassay system consists of a horizontally-mounted 2-inch diameter RCA 8575 photomultiplier tube (PMT) with an S-20 response used in the photon count mode. The QwikLite test chamber is constructed from black delron and is connected to the controller box via a combined power and signal cable. The top of the chamber is removable and houses a small adjustable stainless steel shaft terminating in a plastic propeller. The controller box has face displays for PMT and stirring motor voltages, PMT count LED, preset count time settings, manual and automatic switches to run the system, and backlit start, stop, and reset buttons. Neutral density optical filters can be easily changed (ND-1, ND-2, ND-3) to prevent PMT saturation. Dinoflagellate cells are cultured in optical grade spectrophotometric plastic cuvettes, which are placed individually into the test chamber.

### 2.2 PROCESS DESCRIPTION

FPXRF measures a wide range of elements (sulfur through uranium) simultaneously at concentrations between a part per million (ppm) and percent levels. FPXRF is chosen for its extraordinary sensitivity, high specificity, simplicity, and low cost. It is a widely accepted, powerful technique that is used for environmental, industrial, and biotechnology applications. FPXRF is a relatively simple analytical technique that involves minimum sample handling. FPXRF’s sensitivity and specificity reduce or eliminate the sample preparation procedures often required to concentrate analytes or remove interferences from samples before analysis. This reduction in or elimination of sample preparation time not only simplifies, but also expedites the analysis.

The principal limitations of this technique are that it is matrix sensitive, semiquantitative, and elemental, rather than species-of-molecule specific.

This UVF field screening method is used to rapidly determine the location and relative extent of PAH contamination in sediment. As with FPXRF, the method yields qualitative and semiquantitative results, making it appropriate for preliminary assessments of contaminant
distribution as in environmental field screening applications. The high sensitivity and ease of operation of a field fluorometer make fluorescence the method of choice for field screening. UVF uses solvent extractions of the bulk sediment to improve PAH detection levels even further, down in the low ppm range. Method sensitivity can vary depending on a number of factors including: sediment matrix, extraction solvent, excitation and emission wavelengths, and specific PAHs present.

Protection of aquatic species requires prevention of unacceptable effects on populations in natural habitats. Toxicity tests provide data for predicting changes in the viable numbers of individual species that may result from similar exposure in the natural habitat. Information may also be obtained on the effects of the material on the health of other species. Bioluminescent dinoflagellates represent an important eucaryotic group, which are widely distributed in the oceanic environment.

QwikSed bioassays can help piece together all the elements that determine whether or not a targeted area is in need of remediation or control. The use of bioluminescent dinoflagellates, as part of a broader-based biological and chemical testing program, can help identify a potential problem. By analyzing biological effects using QwikSed, unsuspected contaminants may be indicated.

Substantial savings in operational costs can be achieved by use of this system when compared to other standard bioassays. The QwikSed system can save money in conducting these toxicity tests when compared to conducting the more traditional tests using shrimp, fish, and amphipods. QwikSed requires less time to set up the bioassay and less time to conduct the test.

The QwikSed bioassay system has been shown to have equal sensitivity to other standard bioassays and can be used as a mapping tool for determining the extent of marine contamination of sediment pore waters in a fairly short period of time. Without this system, more costly and time consuming methods for toxicity determination of effluents and sediments will be necessary to determine compliance-related issues. Standard bioassays are time-consuming to implement (4 to 8 days of labor per test for an acute 4-day test) and expensive when compared to the proposed QwikSed system (6 to 7 hours of labor) for conducting an equivalent test.

2.3 PREVIOUS TESTING OF THE TECHNOLOGY

An ongoing validation process of the three field screening technologies has been used by SSC San Diego at multiple sites to provide a database for review by regulatory agencies in technology acceptance programs. The XRF, UVF, and QwikSed techniques have been either evaluated or demonstrated independently for different matrices by several technology certification programs including the following:

FPXRF—U.S. Environmental Protection Agency (EPA), Department of Defense (DoD), and Department of Energy - Consortium for Site Characterization Technology
UVF—California Environmental Protection Agency (Cal-EPA) Technology Certification Program

QwikSed—American Society for Standard Tests and Methods (ASTM)

The EPA site program compared several XRF units in their evaluation of soil screening methods. The Cal-EPA certification program also looked at soil screening methods, with UVF methods being one of several techniques addressed. ASTM methods have addressed different bioassay techniques, with QwikSed being one specific to the sediment matrix. All these comparisons are complete and results can be obtained from the specific programs.

2.4 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

There are far more sensitive and accurate methods for measuring contaminants in the laboratory than these screening tools. These laboratory methods, however, are slow, laborious and expensive. The field screening tools allow for the rapid mapping and ranking of contaminated sites. With the guidance of this low-cost tool, a high density of semiquantitative data can be generated on site in near real time. By pinpointing hotspots and ranking relative contamination levels, these data can guide further sampling, and an intelligent selection of meaningful, rather than random, samples for subsequent, more quantitative laboratory analysis.

Combining FPXRF, UVF, and QwikSed will make it possible to screen for multiple contaminants and their possible biological effects in a more cost-effective manner. Integrating these three screening techniques at a site will facilitate a more efficient and comprehensive mapping of the extent of contamination. Combining screening data with a selected number of laboratory analyses to fully characterize the nature of possible contamination will result in the most efficient analysis plan to characterize the nature and extent of contamination at a site.
3.0 DEMONSTRATION DESIGN

3.1 PERFORMANCE OBJECTIVES

The primary objectives of this demonstration are to evaluate the three field screening technologies in the following areas: (1) their performance compared to conventional sampling and analytical methods; (2) data quality; (3) the logistical and economic resources necessary to operate the technologies; and (4) the range of usefulness in which the technologies can be operated and integrated into a screening procedure that allows more efficient assessment of sediment sites. Secondary objectives for this demonstration are to evaluate the technologies for their reliability, ruggedness, and ease of operation. The fourth primary objective is important because current regulatory projects often rely on “blind” sampling, with little or no knowledge of how much volume of sediment each laboratory measurement represents. Cheaper screening techniques will allow more knowledgeable sample selection for laboratory analysis and therefore better insight into how representative these samples are. As additional screening techniques are developed, they may be incorporated into existing screening procedures.

3.2 SELECTION OF TEST SITES

SSC SAN DIEGO searched for suitable demonstration sites with sufficient contamination levels and ranges to demonstrate screening tool capabilities. It was determined that Naval Air Station (NAS) Alameda and the Pearl Harbor Naval Center contain several potential sites with metal and hydrocarbon contamination suitable for demonstrating the XRF, UVF, and QwikSed technologies. These sites were selected based on the following criteria:

- Demonstrations done at the same time as ongoing regulatory projects can offset some of the demonstration and validation costs of the screening project, including ship and sampling operations, and laboratory analyses.
- The updated results from screening methods can be used by the regulatory projects, and results will receive wide circulation among regulators and the public.
- The sediment contaminant levels identified during previous investigations ranged from below analytical laboratory detection limits to greater than significantly high levels [above those causing adverse biological effects (Long et al., 1995)]. The analytical results from the sites suggest that adequate levels of metals and PAHs exist to demonstrate the XRF, UVF, and QwikSed technologies.

NAS Alameda was chosen as the preliminary test site for demonstrating XRF, UVF, and QwikSed technologies. NAS Alameda is located on Alameda Island, at the western end of the City of Alameda in Alameda County, California. Alameda Island lies along the eastern side of San Francisco Bay adjacent to the City of Oakland. The rectangular-shaped base is approximately 2 miles long and 1 mile wide and occupies 2,634 acres. NAS Alameda includes 1,526 acres of land and 1,108 submarine acres. Most of the base is land that was created by fill. During the 1930s, most of the land at NAS Alameda was created by hydraulically filling existing...
tidelands, marshlands, and sloughs with material dredged from many areas, including the Oakland Inner Harbor.

The second demonstration was conducted in Pearl Harbor, Hawaii is a large, complex natural estuary and major feature on the south coast of Oahu in the Hawaiian Islands. Most of Pearl Harbor lies within the Pearl Harbor Naval Center (PHNC) in the southern portion of the Ewa plain, approximately 5.8 miles northwest of downtown Honolulu. Pearl Harbor contains 2,024 hectares (8 square miles [sq mi], 5,000 acres [ac]) of surface water area, and 58 kilometers (36 miles) of linear shoreline. Through the influence of drainage, the Pearl Harbor estuary is the receptacle for runoff from approximately 28,502 hectares (110 sq mi, 70,400 ac) of upland habitat making up the watershed for much of the southern portion of the island of Oahu.

During the last century, many human activities have been concentrated along the shoreline and within the upland drainage basins that empty into the harbor. These activities include the industrial and operational activities of the U.S. Navy; private industrial operations; municipal, commercial, and urban activities; and agriculture. These activities potentially release numerous types of chemical contaminants into the air, water, and soil along the shoreline and within the drainage basins that empty into Pearl Harbor. The approximately 2,024 hectares (5,000 ac) of soft (e.g., mud and sand) sediments that make up the bottom in Pearl Harbor are the ultimate sink or repository for these chemicals and the natural habitat for thousands of estuarine and marine species.

3.3 TEST SITE/FACILITY HISTORY/CHARACTERISTICS

The demonstration project will concentrate efforts in Seaplane Lagoon and deepwater piers on the south side of the lagoon. The lagoon has an area of 110 ac and is located at the southeastern corner of NAS Alameda. Sea walls surround most of the lagoon, inhibiting the natural flushing processes of bay tides. A breakwater extending from Pier 1 forms the southern wall of the lagoon. The entrance to the lagoon is through an 800-ft-long opening in the breakwater. The depth of the lagoon varies from small beach surfaces to a depth of 15 ft. Outside the Seaplane Lagoon are berths for deep draft ships (Piers 1, 2, and 3). These berths are protected by an outer breakwater and have periodic maintenance dredging. No regular dredging program has ever existed at the Seaplane Lagoon, and sediment accumulation is evident in many areas of the lagoon.

Industrial wastewater generated at NAS Alameda before 1974 was discharged directly to the storm drains. The storm drains, in turn, discharged to the Seaplane Lagoon and other offshore areas. The wastewater discharged in the lagoon from 1940 through 1975 was reported to contain heavy metals, solvents, paints, detergents, acids, caustics, mercury, oil, and grease (Ecology and Environment, Inc., 1983). Ship wastewater—which may have contained solvents, chromium, waste oil, and fuel—was also released into the lagoon (Ecology and Environment, Inc., 1983). Between 1972 and 1975, the industrial waste collection system was rerouted to discharge to the East Bay Municipal Utilities District (EBMUD) wastewater system. The Navy now conducts a storm water pollution prevention program to ensure that only rainwater is discharged through the storm drain system. A removal action to remove sediments from the drainage areas of the storm drains was performed in 1995 and the storm drain lines were steam cleaned in November 1996. Other chemicals may have entered the lagoon due to tidal action sweeping ship wastewater—
possibly containing solvents, chromium, waste oil, and fuel—from the berthing area into the lagoon. Continuing sources of chemicals may include sediment contamination caused by current berthing practices or historical activities at Piers 1, 2, and 3.

The present day PHNC is an outgrowth of more than 100 years of peacetime and wartime development that has resulted in dredging to construct a channel and berthing area of sufficient depth to allow passage of the “largest of ships” (Grovhoug, 1992) and construction of extensive shoreside facilities (e.g., ship mooring and repair facilities, fuel storage, handling, transfer, and recycling facilities as well as operations, maintenance, and support facilities) to meet changing needs of the U.S. Fleet. Military vessels using the harbor on a regular basis include U.S. Navy surface ships, submarines, and harbor craft; U.S. Army cargo transport vessels; U.S. Coast Guard buoy tenders and patrol vessels; and foreign naval vessels. Harbor navigation channels and mooring areas at piers and wharves supporting these vessels are maintained at water depths necessary for safe navigation through a program of routine maintenance dredging. New facilities are developed as needed and may involve in-water construction and project-specific dredging.

Grovhoug (1992) reviewed past environmental information on Pearl Harbor compiled from numerous studies conducted over several years. Most of these studies are project-specific and address environmental concerns at specific locations in the harbor. In general, these studies provide useful background information but are limited for purposes of a harborwide assessment because of their age (some are 20 years old), or they are fragmented over temporal or spatial scales.

An overview of the available data determined a few specific areas of interest to demonstrate the screening technologies. These included the Middle Loch and Bishop Point areas. From this overview, it appears as if the Middle Loch area is very fine-rich (75-90%) although it has low total organic carbon (TOC) values (1.98-3.83%). The Bishop Point area appears to be less fine-rich (41-56%), yet the TOC values are higher (4-6%) than the Middle Loch area. The Bishop Point area is a small pier area (~3 acres) and is rumored to be very heterogeneous, with coral hard bottom to soft mud conditions (Jeff Grovhoug personal communication). The pier area is in current use with ships always present. The Middle Loch area, on the other hand, is very large and more homogeneously fine-grained mud. This area is regularly dredged to maintain a draft of 20 ft and used to store a “mothballed” fleet of ships.

The contaminants of concern in these two areas differ. The metals levels are elevated in the Middle Loch area but they are very low at Bishop Point. However, the PAH levels at Bishop Point are elevated and range from ~20-40 ppm tPAH. PAHs do not appear to be elevated in the Middle Loch area. For the other contaminants of concern (pesticides, polychlorinated biphenyls [PCBs], tributyltin [TBT]), the data show these areas not very contaminated.

3.4 PHYSICAL SET-UP AND OPERATION

The details of the methodology for the various screening techniques are adapted from standard protocols. These screening techniques have been adapted from EPA (PRC 1995; Filkins, 1992) or ASTM (Lapota et al., 1997) methods. Sediment samples were obtained by standard grabs or cores. Representative sample splits were separated for screening and laboratory analyses. Due to the different analysis times required by the various techniques, it is expected that results will
be available from FPXRF after several minutes, followed by UVF after half an hour, and finally by QwikSed after 4 to 24 hours.

With the differing analysis times, results were available from the various screening techniques at different times during the sampling process. Therefore, we depend more heavily on near real-time chemistry results to help guide subsequent sampling locations. The general procedure in mapping out contaminant plumes starts at suspected sources (e.g., industrial outfall pipes), and works outward to delineate the extent of contamination. If no contamination is detected at the source using one or several screened samples, there is no need to continue sampling away from the source. If contamination is detected at the source area, sampling continues outward to define the edge of the contaminant plume. Since the biological effects results from QwikSed would not be available until much later (4 to 24 hours), these data were not available for near real-time guidance during sampling. They are, however, used together with the chemistry screening data to select which samples continue to the laboratory for full characterization. Laboratory samples were selected to span the full range of results observed in all screening techniques. This allows calibration curves between screening and laboratory techniques to cover the entire range of observed results and therefore allows better predictions from the remaining screening results.

Site contractors (PRC and site subcontractors) conducted sampling and analysis for the regulatory project, so they handled all site setup and facilities. SSC San Diego provides FPXRF, UVF, and QwikSed equipment and operators who recover a sample split for screening analysis. Remaining samples continued on to the laboratory for confirmatory analyses.

3.5 SAMPLING/MONITORING PROCEDURES

During the demonstration at Alameda, 23 to 30 sediment samples were collected, depending on the screening technique. At the second demonstration in Pearl Harbor, 18 to 30 samples were collected. All samples were analyzed in the field at Pearl Harbor. Including both pre-demonstration and demonstration sampling, there were 91 samples for XRF, 67 samples for UVF, and 47 samples for QwikSed.

3.6 ANALYTICAL PROCEDURES

To assess the comparability of the data acquired by the FPXRF, UVF, and QwikSed screening technologies to data generated by conventional analytical methods, the screening data are compared to confirmatory analysis results. The overall objective of the sampling program is to collect FPXRF, UVF, QwikSed, and traditional analytical data in parallel to demonstrate the FPXRF, UVF, and QwikSed technologies’ capability to delineate the extent of sediment contamination.
4.0 PERFORMANCE ASSESSMENT

4.1 PERFORMANCE DATA

The main performance criteria used for comparing individual screening techniques to laboratory data are correlation coefficients ($r^2$), and relative standard deviations (RSD). Following the example of EPA procedures (PRC, 1995), screening data can be classed into three levels depending on these criteria. Level 3 definitive data ($r^2 = 0.85$ to 1.00, RSD < 10%) can be considered to substitute for laboratory data. Level 2 semiquantitative screening data ($r^2 = 0.6$ to 0.85, RSD = 10-20%) require a limited number of confirmatory samples (usually around 10%) for calibration to be considered quantitative. Level 1 qualitative screening data ($r^2 < 0.6$, RSD > 20%) detect the presence or absence of some parameter, but may not quantify concentration levels. Although most screening data is classed as Level 1, the goal of this project is to demonstrate the data will meet or exceed Level 2 requirements. It should be pointed out that with QwikSed, which is a screening bioassay, we will not be able to follow the exact EPA Level 1, 2, and 3 criteria defined for chemical screening techniques. Since there are no universally accepted criteria to evaluate the correlation of bioassay results similar to the above chemical criteria, we use a series of contingency tables.

Figure 1 and Figure 2 provide graphs of FPXRF data plotted versus certified laboratory data for sample splits. These data are also presented in tables and figures in the final report for this project. Figure 1 shows the data from the first demonstration at NAS Alameda, with data for Fe, Cu, Pb, and Zn provided for comparison. Correlation coefficients range from 0.71 to 0.87, with one outlier for Cu removed since it was significantly outside the range of the other data. The RSD data also fall within the Level 2 criteria above, so these data can be classified as semiquantitative as long as a certain percent of confirmatory samples are also run. Figure 2 shows these same data from the second demonstration at Pearl Harbor. Correlation coefficients at this site tended to be slightly lower, ranging from 0.46 to 0.78. As discussed in the final report and Section 4.3 of this report, matrix differences may explain some of the differences in performance between the sites. The RSD data were also slightly higher, with some RSDs above the 20% level. The overall evaluation of the data following the above criteria leads to the conclusion that at this particular site some of the data fall below the Level 2 criteria. This indicates that, depending on matrix characteristics, some FPXRF data may fall into Level 1 and therefore they may only be considered qualitative screening data. This restricts the ability of the data to be used with site-specific calibration curves (typically used with Level 2 data to convert field screening data onto comparable scale with conventional laboratory data) to produce quantitative data. Potential causes and solutions to this limitation will be further discussed in Section 4.3.
Figure 1. FPXRF Fe, Zn, Cu, and Pb Results Plotted Against Results from Standard Methods.

Inset of graph in Cu panel shows all samples, and blowup shows all samples in lower concentration range.
Circles represent samples collected from predemonstration at NAS Alameda, Southshore Pier area, and squares represent samples collected during demonstration at NAS Alameda, Seaplane Lagoon.
Circles represent samples collected for predemonstration from five sites at Pearl Harbor, squares represent demonstration samples collected from the Bishop Point site, and diamonds represent demonstration samples collected from the Middle Loch site.

Figure 2. FPXRF Fe, Zn, Cu, and Pb Results Plotted Against Results from Standard Methods.
Figure 3, Figure 4, and Figure 5 provide graphs of the UVF data plotted versus the certified laboratory results for PAHs on sample splits. These data are also presented in tables and graphs in the final report for this project. Figure 3 and Figure 4 show the data for the first demonstration at NAS Alameda, and Figure 5 shows the data for the second demonstration at Pearl Harbor. As discussed further in Section 4.3, multiple calibration curves are required at locations like NAS Alameda (Pier Area in Figure 3 and Seaplane Lagoon in Figure 4) because of different mixtures of PAHs leading to similar fluorescent intensity. As a measure of the accuracy of the techniques, the correlation coefficients range from 0.71 to 0.89 in the plots. To assess the precision, tables in the final report show RSDs are below 20%, with most below 10%. These screening data therefore also fall into the Level 2 criteria, being considered semiquantitative with a limited number of confirmatory samples required. These limited number of site-specific confirmatory samples can be used to generate similar calibration relationships to those shown in Figure 3, Figure 4, and Figure 5 so fluorescence intensity can be converted to a site specific total PAH level.

To assess correspondence of Qwiksed data to laboratory data, contingency tables will be used rather than correlation coefficients from x-y plots. Table 1, Table 2, and Table 3 contain NAS Alameda data, where two different laboratory bioassays were run. The first two tables compare Qwiksed to each laboratory bioassay, and the third table compares the two laboratory bioassays. Table 4 shows the Pearl Harbor results, where only one laboratory bioassay was run for comparison. The contingency tables show agreement (both assays above or both assays below benchmark level) ranged from 72% to 92% for these data. It is interesting to note that the least agreement was observed between the two laboratory bioassays. This points to the limitation in study design where the “real” value is assumed to be measured by some laboratory test. With bioassays, the different organisms show variable sensitivities to the site contaminant mixtures, so some degree of variability is expected between bioassay results. As a measure of precision, replicate measurements in the final report appendix show RSD range above 20%. Overall, the biological screen appears to be analogous to Level 1 qualitative data in the chemical classification scheme discussed above. It does not appear to support a quantitative value of another bioassay could be determined from the Qwiksed result, but it does support that the qualitative assessment of whether another bioassay will be above or below a regulatory benchmark.

4.2 PERFORMANCE CRITERIA

The primary and secondary performance objectives are stated in Section 3.1. The “performance compared to conventional analytical methods” and “data quality” can be assessed by looking at accuracy and precision as described in Section 4.1. The “logistical and economic resources necessary to operate the technologies” is discussed in Section 5. The “range of usefulness in which the technologies can be operated and integrated into a screening procedure” as well as the additional secondary criteria concerning “reliability, ruggedness, and ease of operation” is discussed in a qualitative manner in Section 4.3.
Figure 3. UVF Predemonstration and Demonstration Samples at Pier Area in Demo #1.

Figure 4. UVF Predemonstration and Demonstration Samples at Seaplane Lagoon in Demo #1.
Figure 5. UVF Predemonstration and Demonstration Samples at Demo #2.

Table 1. Comparison of Predemonstration Amphipod (Eohaustorius) Toxicity Data with QwikSed Demonstration Toxicity Data at NAS Alameda for 25 Samples.

<table>
<thead>
<tr>
<th>Amphipod</th>
<th>Qwiksed Results</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Toxic</td>
<td>Not Toxic</td>
</tr>
<tr>
<td>Toxic</td>
<td>5</td>
<td>1</td>
</tr>
<tr>
<td>Not Toxic</td>
<td>1</td>
<td>18</td>
</tr>
<tr>
<td>Total</td>
<td>6</td>
<td>19</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Both Toxic</th>
<th>Both Not Toxic</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Both Toxic</td>
<td>5/25</td>
<td>20%</td>
<td></td>
</tr>
<tr>
<td>Both Not Toxic</td>
<td>18/25</td>
<td>72%</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>23/25</td>
<td>92% agreement</td>
<td></td>
</tr>
</tbody>
</table>

Table 2. Comparison of the Sea Urchin Development Toxicity Data with QwikSed Toxicity Data at NAS Alameda for 25 Samples.

<table>
<thead>
<tr>
<th>Seaurchin</th>
<th>Qwiksed Results</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Toxic</td>
<td>Not Toxic</td>
</tr>
<tr>
<td>Toxic</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Not Toxic</td>
<td>4</td>
<td>17</td>
</tr>
<tr>
<td>Total</td>
<td>6</td>
<td>19</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Both Toxic</th>
<th>Both Not Toxic</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Both Toxic</td>
<td>2/25</td>
<td>8%</td>
<td></td>
</tr>
<tr>
<td>Both Not Toxic</td>
<td>17/25</td>
<td>68%</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>19/25</td>
<td>76% agreement</td>
<td></td>
</tr>
</tbody>
</table>
Table 3. Comparison of Predemonstration Amphipod Toxicity Data with Sea Urchin Development Toxicity Demonstration Data at NAS Alameda for 25 Samples.

<table>
<thead>
<tr>
<th>Seaurchin</th>
<th>Amphipod Results</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Toxic</td>
<td>Not Toxic</td>
</tr>
<tr>
<td>Toxic</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Not Toxic</td>
<td>5</td>
<td>16</td>
</tr>
<tr>
<td>Total</td>
<td>7</td>
<td>18</td>
</tr>
<tr>
<td>Both Toxic</td>
<td>2/25</td>
<td>8%</td>
</tr>
<tr>
<td>Both Not Toxic</td>
<td>16/25</td>
<td>64%</td>
</tr>
<tr>
<td>Total</td>
<td>18/25</td>
<td>72% agreement</td>
</tr>
</tbody>
</table>

Table 4. Comparison of the Sea Urchin Development Toxicity Data with QwikSed Toxicity Data at Pearl Harbor.

<table>
<thead>
<tr>
<th>Seaurchin</th>
<th>QwikSed Results</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Toxic</td>
<td>Not Toxic</td>
</tr>
<tr>
<td>Toxic</td>
<td>13</td>
<td>2</td>
</tr>
<tr>
<td>Not Toxic</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Total</td>
<td>14</td>
<td>4</td>
</tr>
<tr>
<td>Both Toxic</td>
<td>13/18</td>
<td>72%</td>
</tr>
<tr>
<td>Both Not Toxic</td>
<td>2/18</td>
<td>11%</td>
</tr>
<tr>
<td>Total</td>
<td>15/18</td>
<td>83% agreement</td>
</tr>
</tbody>
</table>

4.3 DATA ASSESSMENT

The discussion of accuracy and precision measures in Section 4.1 indicates the chemical screening techniques performance compared to conventional analytical methods and overall data quality are sufficient to classify them as Level 2 semiquantitative screening techniques (PRC, 1995). This indicates that a limited number of laboratory confirmatory analyses are required to generate a site-specific calibration curve to transform screening data onto a comparable scale with laboratory data. This is demonstrated in Figure 1 and Figure 2, where relationships in FPXRF data show their bias with varying slopes and nonzero intercepts. This is not unexpected given the differences in sample preparation (none [wet, underground samples]) for screening versus acid dissolution or leach for laboratory) and analysis. This is similar to what is seen in the UVF data in Figure 3, Figure 4, and Figure 5, but here site-specific calibration relationships are required to convert the fluorescence intensity into a comparable total PAH value. The matrix effects caused by variable mixtures of PAHs can lead to the need for multiple calibration curves depending on site PAH mixture, as is demonstrated by splitting the NAS Alameda data into Figure 3 and Figure 4. So when the NAS Alameda data as a whole showed poor correlation, the solution to the matrix problems was splitting the data depending on the source mixture of PAHs at the sites. Many individual alkylated PAHs show more fluorescence intensities than their parent PAH compounds, so dividing sites based on the type of PAHs present becomes important. Much better correlation coefficients are obtained and better site-specific calibrations are developed to predict PAHs from similar sets of screening fluorescence intensity data. The
scatter in the FPXRF data in Figure 2 may also be related to matrix effects, but different matrix effects and different techniques require different solutions. The Pearl Harbor sediment is derived from iron-rich volcanic deposits with varying amounts of calcareous (coral and shells) material. This is in contrast to the silica-rich deposits at NAS Alameda that are more common at continental sites. These solid matrix variations appear to have more effect on FPXRF performance since the analysis has no preparation and is conducted on the solid matrix of the sample. The UVF screening technique uses hexane solvent extraction as a preparation method so the variations in solid matrix do not appear to have much effect. As discussed in the final report, better results (higher correlation coefficients) may be possible with more sample preparation. If samples are dried and ground, much more homogeneous splits can be derived and variability due to sample heterogeneity can be reduced. Another discussion in the final report points out that since this project was started, FPXRF equipment is available with better source and detector capabilities so interferences from high iron levels are reduced. Overall the main demonstration point is that the chemical screening techniques are semiquantitative with the requirement of limited laboratory confirmatory analyses. Aside from these matrix limitations, the techniques were judged to show good performance for screening potential. They are fairly easy to operate with limited training required.

4.4 TECHNOLOGY COMPARISON

There are many additional field analytical screening techniques available as onsite mobile laboratories become more common. Many of these options are discussed in Section 5.3 of the project final report. For metals determinations, XRF still appears to be the best option. With better source and detector capabilities becoming available on field portable units, additional improvements in cost and performance can be expected. For organic contaminants such as PAHs, immunoassays are becoming another acceptable field option. The ability to run several immunoassays on the same solvent extract for different contaminant groups (PAHs, PCBs, and pesticides) shows one advantage immunoassays possess. For screening bioassays, there are fewer alternatives. Microtox, which uses bacterial bioluminescence, is one possible alternative. The main limitation for screening bioassays is finding a technique that matches the typical regulatory bioassays so that the screening technique adequately predicts the standard regulatory bioassay response over a range of contaminants. Since all organisms have different sensitivities, this becomes a limitation. Many screening bioassays also tend to have reduced sensitivity because exposure times are shortened to increase throughput.
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5.0 COST ASSESSMENT

5.1 COST REPORTING

In addition to the technical performance of the screening techniques, the cost of screening technique use plays a major role in determining whether screening will prove to be a useful addition to sediment assessments. As a general rule, screening techniques are inexpensive when compared to traditional standard laboratory techniques. This advantage needs to be weighed against potential limitations. To more fully evaluate the cost performance of the screening techniques, the following tables provide assumed costs for use under different scenarios. Because of the costs involved for mobilization and demobilization to deploy onsite, cost examples are given for deploying onsite and for having samples sent back to a centralized lab facility (similar to what would be done for standard analyses). Because there is economy of scale for most of these screening techniques, running larger sample sizes will generally prove more cost-effective. This is demonstrated in the examples with cost estimates for assessing both 30 and 100 samples at a time. Table 5, Table 6, and Table 7 contain costing examples for XRF, UVF, and QwikSed, respectively.

5.2 COST ANALYSIS

These sample cost comparisons indicate that equipment costs can play a large role in determining per sample costs. For this reason, columns of cost data are provided for both a purchase and lease option. Under each cost scenario, four per sample costs are given with various options. Since the last two cost figures exclude the instrument costs, values in the purchase and lease columns are the same. After equipment costs, the number and cost of confirmatory (validation) samples is another large variable so various options are included that exclude these costs. The last cost option is probably most appropriate since it excludes both equipment and validation costs and provides an estimate of the per sample cost of the screening technique.

5.3 COST COMPARISONS

Table 5, Table 6, and Table 7 contain the approximate per sample cost currently charged by laboratories for the standard laboratory analyses. For standard sediment metals analyses of Cu, Pb, and Zn, the cost would be $150 to $300, depending on the laboratory. For PAHs, the per sample cost is approximately $500 and includes a breakdown of the individual PAHs as well as the total given by the screening technique. For bioassays, the cost is highly variable, depending on the particular bioassay. Sea Urchin larval development bioassays are approximately $500, while the amphipod bioassay may cost up to $1,500. For this comparison, an average of $1,000 is used. A rough estimate for combined laboratory costs is just under $2,000 per sample. This could be compared to a combined cost of these screening techniques, which would be just under $200 per sample (run in-house with 150 samples).

All three technologies are easily transferred and shippable. FPXRF, UVF, and QwikSed materials can be transferred as luggage aboard commercial flights. Total weight of each is between 150 and 300 lbs. Each technology is contained within a protective carrying case and
does not need special handling requirements. It may be more appropriate to ship centrifuge, test chamber, and miscellaneous laboratory supplies ahead of time.

Table 5. Relative FPXRF Analytical Costs.

<table>
<thead>
<tr>
<th></th>
<th>I. On Site FPXRF Costs (Continental US Example; e.g. Demo Site #1)</th>
<th>II. In House</th>
</tr>
</thead>
<tbody>
<tr>
<td># Samples</td>
<td>Purchase (Spectrace 9000)</td>
<td>Lease (Spectrace 9000)</td>
</tr>
<tr>
<td></td>
<td>n = 30 (includes QAQC)</td>
<td>58,000 (instrument)</td>
</tr>
<tr>
<td></td>
<td>Supplies</td>
<td>50 (supplies)</td>
</tr>
<tr>
<td></td>
<td>analysis time = 1 day</td>
<td>775 (labor@$86/hr for 9 hr)</td>
</tr>
<tr>
<td></td>
<td>Mob/demob = 1 day</td>
<td>775 (labor@$86/hr for 9 hr)</td>
</tr>
<tr>
<td></td>
<td>per diem = 2.5 days</td>
<td>188 (per diem@75/day)</td>
</tr>
<tr>
<td></td>
<td>travel (air)</td>
<td>250 (airfare, gov’t rate)</td>
</tr>
<tr>
<td></td>
<td>rental car = 2.5 days</td>
<td>75 (gov’t rate)</td>
</tr>
<tr>
<td></td>
<td>Validation Samples (20%)</td>
<td>900 (at $150/sample for n=6)</td>
</tr>
<tr>
<td></td>
<td>Cost</td>
<td>$2,034 per sample</td>
</tr>
<tr>
<td></td>
<td>Exclude Validation Cost</td>
<td>$2,003 per sample</td>
</tr>
<tr>
<td></td>
<td>Exclude Instrument Cost</td>
<td>$100 per sample</td>
</tr>
<tr>
<td></td>
<td>Exclude Instr. &amp; Val. Cost</td>
<td>$70 per sample</td>
</tr>
<tr>
<td></td>
<td>n = 150 (includes QAQC)</td>
<td>58,000 (instrument)</td>
</tr>
<tr>
<td></td>
<td>Supplies</td>
<td>250 (supplies)</td>
</tr>
<tr>
<td></td>
<td>analysis time = 5 days</td>
<td>3,870 (labor@$86/hr for 40 hr)</td>
</tr>
<tr>
<td></td>
<td>Mob/demob = 1 day</td>
<td>775 (labor@$86/hr for 9 hr)</td>
</tr>
<tr>
<td></td>
<td>per diem = 6.5 days</td>
<td>488 (per diem@75/day)</td>
</tr>
<tr>
<td></td>
<td>travel (air)</td>
<td>250 (airfare, gov’t rate)</td>
</tr>
<tr>
<td></td>
<td>rental car</td>
<td>195 (gov’t rate)</td>
</tr>
<tr>
<td></td>
<td>Validation Samples (20%)</td>
<td>4,500 (at $150/sample for n=30)</td>
</tr>
<tr>
<td></td>
<td>Cost</td>
<td>$455 per sample</td>
</tr>
<tr>
<td></td>
<td>Exclude Validation Cost</td>
<td>$426 per sample</td>
</tr>
<tr>
<td></td>
<td>Exclude Instrument Cost</td>
<td>$69 per sample</td>
</tr>
<tr>
<td></td>
<td>Exclude Instr. &amp; Val. Cost</td>
<td>$39 per sample</td>
</tr>
</tbody>
</table>

Instrument and Validation Cost

|                      |                      |                      |                      |
|                      |                      |                      |                      |

Note:

a: This purchase scenario is based on a unit purchase for a single project. The cost per sample for this scenario would decrease significantly if the purchased unit were used on multiple projects.

b: FPXRF Supplies: XRF sample cups, Mylar film, gloves, mixing rods, etc.

c: Sample shipment costs and data analysis/reporting costs are not included here
### Table 6. Relative UVF Analytical Costs.

#### I. On Site UVF Costs (Continental US Example; e.g. Demo Site #1)

<table>
<thead>
<tr>
<th># Samples</th>
<th>Purchasea (Turner Fluorometer)</th>
<th>Lease (Turner Fluorometer)</th>
<th>Cost Per Sample (Certified)</th>
</tr>
</thead>
<tbody>
<tr>
<td>n = 30 (includes QAQC)</td>
<td>9,500 (instrument)</td>
<td>1,200 (two weeks +S/H)</td>
<td></td>
</tr>
<tr>
<td>analysis time = 1 day</td>
<td>775 (labor@$86/hr for 9 hr)</td>
<td>775 (labor@$86/hr for 9 hr)</td>
<td></td>
</tr>
<tr>
<td>mob/demob = 1 day</td>
<td>775 (labor@$86/hr for 9 hr)</td>
<td>775 (labor@$86/hr for 9 hr)</td>
<td></td>
</tr>
<tr>
<td>per diem = 2.5 days</td>
<td>188 (per diem@$75/day)</td>
<td>188 (per diem@$75/day)</td>
<td></td>
</tr>
<tr>
<td>travel (air)</td>
<td>250 (airfare, gov’t rate)</td>
<td>250 (airfare, gov’t rate)</td>
<td></td>
</tr>
<tr>
<td>rental car = 2.5 days</td>
<td>75 (gov’t rate)</td>
<td>75 (gov’t rate)</td>
<td></td>
</tr>
<tr>
<td>Validation Samples (20%)</td>
<td>3,000 (at $500/sample for n=6)</td>
<td>3,000 (at $500/sample for n=6)</td>
<td></td>
</tr>
<tr>
<td>Cost</td>
<td>$487 per sample</td>
<td>$210 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Validation Cost</td>
<td>$387 per sample</td>
<td>$110 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Instrument Cost</td>
<td>$170 per sample</td>
<td>$170 per sample</td>
<td></td>
</tr>
<tr>
<td>n = 150 (includes QAQC)</td>
<td>9,500 (instrument)</td>
<td>1,200 (instrument)</td>
<td>$500 per sample</td>
</tr>
<tr>
<td>analysis time = 5 days</td>
<td>3,870 (labor@$86/hr for 40 hr)</td>
<td>3,870 (labor@$86/hr for 40 hr)</td>
<td></td>
</tr>
<tr>
<td>mob/demob = 1 day</td>
<td>775 (labor@$86/hr for 9 hr)</td>
<td>775 (labor@$86/hr for 9 hr)</td>
<td></td>
</tr>
<tr>
<td>per diem = 6.5 days</td>
<td>488 (per diem@$75/day)</td>
<td>488 (per diem@$75/day)</td>
<td></td>
</tr>
<tr>
<td>travel (air)</td>
<td>250 (airfare, gov’t rate)</td>
<td>250 (airfare, gov’t rate)</td>
<td></td>
</tr>
<tr>
<td>rental car</td>
<td>195 (gov’t rate)</td>
<td>195 (gov’t rate)</td>
<td></td>
</tr>
<tr>
<td>Validation Samples (20%)</td>
<td>15,000 (at $500/sample for n=30)</td>
<td>15,000 (at $500/sample for n=30)</td>
<td></td>
</tr>
<tr>
<td>Cost</td>
<td>$202 per sample</td>
<td>$147 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Validation Cost</td>
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<td>$47 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Instrument Cost</td>
<td>$139 per sample</td>
<td>$139 per sample</td>
<td></td>
</tr>
<tr>
<td>II. In House</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td># Samples</td>
<td>Purchasea (Turner Fluorometer)</td>
<td>Lease (Turner Fluorometer)</td>
<td>Cost Per Sample (Certified)</td>
</tr>
<tr>
<td>n = 30 (includes QAQC)</td>
<td>9,500 (instrument)</td>
<td>1,200 (two weeks +S/H)</td>
<td></td>
</tr>
<tr>
<td>analysis time = 1 day</td>
<td>775 (labor@$86/hr for 9 hr)</td>
<td>775 (labor@$86/hr for 9 hr)</td>
<td></td>
</tr>
<tr>
<td>Validation Samples (20%)</td>
<td>3,000 (at $500/sample for n=6)</td>
<td>3,000 (at $500/sample for n=6)</td>
<td></td>
</tr>
<tr>
<td>Cost</td>
<td>$444 per sample</td>
<td>$168 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Validation Cost</td>
<td>$344 per sample</td>
<td>$68 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Instrument Cost</td>
<td>$128 per sample</td>
<td>$128 per sample</td>
<td></td>
</tr>
<tr>
<td>n = 150 (includes QAQC)</td>
<td>9,500 (instrument)</td>
<td>1,200 (instrument)</td>
<td>$500 per sample</td>
</tr>
<tr>
<td>analysis time = 5 days</td>
<td>3,870 (labor@$86/hr for 40 hr)</td>
<td>3,870 (labor@$86/hr for 40 hr)</td>
<td></td>
</tr>
<tr>
<td>Validation Samples (20%)</td>
<td>15,000 (at $500/sample for n=30)</td>
<td>15,000 (at $500/sample for n=30)</td>
<td></td>
</tr>
<tr>
<td>Cost</td>
<td>$191 per sample</td>
<td>$135 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Validation Cost</td>
<td>$91 per sample</td>
<td>$35 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Instrument Cost</td>
<td>$127 per sample</td>
<td>$127 per sample</td>
<td></td>
</tr>
</tbody>
</table>

Note:
a: This purchase scenario is based on a unit purchase for a single project. The cost per sample for this scenario would decrease significantly if the purchased unit were costed over multiple projects.
b: UVF Supplies: Hexane solvent, glassware, gloves, mixing rods, etc.
c: Sample shipment costs and data analysis/reporting costs are not included here.
Table 7. Relative QwikSed Analytical Costs.

<table>
<thead>
<tr>
<th>I. On Site QwikSed Costs (Continental US Example; e.g., Demo Site #1) a</th>
<th># Samples</th>
<th>Purchase a (QwikSed Toxicity)</th>
<th>Lease (QwikSed)</th>
<th>Cost Per Sample (Certified)</th>
</tr>
</thead>
<tbody>
<tr>
<td>n = 30 (includes QAQC)</td>
<td>15,000 (instrument)</td>
<td>500 (two weeks +S/H)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>supplies b</td>
<td>50 (supplies)</td>
<td>50 (supplies)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>analysis time = 5 day</td>
<td>3,875 (labor @$86/hr for 9 hr)</td>
<td>3,875 (labor @$86/hr for 9 hr)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>mob/demob = 1 day</td>
<td>775 (labor @$86/hr for 9 hr)</td>
<td>775 (labor @$86/hr for 9 hr)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>per diem = 6.5 days</td>
<td>488 (per diem @$75/day)</td>
<td>488 (per diem @$75/day)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>travel (air)</td>
<td>250 (airfare, govt rate)</td>
<td>250 (airfare, govt rate)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rental car = 5 days</td>
<td>150 (govt rate)</td>
<td>150 (govt rate)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Validation Samples (20%)</td>
<td>6,000 (at $1000/sample for n=6)</td>
<td>6,000 (at $1000/sample for n=6)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cost</td>
<td>$886 per sample</td>
<td>$403 per sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exclude Validation Cost</td>
<td>$666 per sample</td>
<td>$203 per sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exclude Instrument Cost</td>
<td>$386 per sample</td>
<td>$386 per sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exclude Instr. &amp; Val. Cost</td>
<td>$186 per sample</td>
<td>$186 per sample</td>
<td>$1,000 per sample</td>
<td></td>
</tr>
<tr>
<td>n = 150 (includes QAQC)</td>
<td>15,000 (instrument)</td>
<td>1,000 (instrument)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>supplies b</td>
<td>250 (supplies)</td>
<td>250 (supplies)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>analysis time = 25 days</td>
<td>19,375 (labor @$86/hr for 40 hr)</td>
<td>19,375 (labor @$86/hr for 40 hr)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>mob/demob = 1 day</td>
<td>775 (labor @$86/hr for 9 hr)</td>
<td>775 (labor @$86/hr for 9 hr)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>per diem = 26.5 days</td>
<td>1,988 (per diem @$75/day)</td>
<td>1,988 (per diem @$75/day)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>travel (air)</td>
<td>250 (airfare, govt rate)</td>
<td>250 (airfare, govt rate)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>rental car</td>
<td>810 (govt rate)</td>
<td>810 (govt rate)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Validation Samples (20%)</td>
<td>30,000 (at $1000/sample for n=30)</td>
<td>30,000 (at $1000/sample for n=30)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cost</td>
<td>$456 per sample</td>
<td>$363 per sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exclude Validation Cost</td>
<td>$256 per sample</td>
<td>$163 per sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exclude Instrument Cost</td>
<td>$356 per sample</td>
<td>$356 per sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exclude Instr. &amp; Val. Cost</td>
<td>$156 per sample</td>
<td>$156 per sample</td>
<td>$1,000 per sample</td>
<td></td>
</tr>
</tbody>
</table>

II. In House

<table>
<thead>
<tr>
<th># Samples</th>
<th>Purchase a (QwikSed Toxicity)</th>
<th>Lease (QwikSed)</th>
<th>Cost Per Sample (Certified)</th>
</tr>
</thead>
<tbody>
<tr>
<td>n = 30 (includes QAQC)</td>
<td>15,000 (instrument)</td>
<td>500 (two weeks +S/H)</td>
<td></td>
</tr>
<tr>
<td>supplies b</td>
<td>50 (supplies)</td>
<td>50 (supplies)</td>
<td></td>
</tr>
<tr>
<td>analysis time = 5 day</td>
<td>3,875 (labor @$86/hr for 9 hr)</td>
<td>3,875 (labor @$86/hr for 9 hr)</td>
<td></td>
</tr>
<tr>
<td>Validation Samples (20%)</td>
<td>6,000 (at $1000/sample for n=6)</td>
<td>6,000 (at $1000/sample for n=6)</td>
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</tr>
<tr>
<td>Cost</td>
<td>$831 per sample</td>
<td>$348 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Validation Cost</td>
<td>$631 per sample</td>
<td>$138 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Instrument Cost</td>
<td>$331 per sample</td>
<td>$331 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Instr. &amp; Val. Cost</td>
<td>$131 per sample</td>
<td>$131 per sample</td>
<td>$1,000 per sample</td>
</tr>
<tr>
<td>n = 150 (includes QAQC)</td>
<td>15,000 (instrument)</td>
<td>1,000 (Four weeks +S/H)</td>
<td></td>
</tr>
<tr>
<td>supplies b</td>
<td>250 (supplies)</td>
<td>250 (supplies)</td>
<td></td>
</tr>
<tr>
<td>analysis time = 25 days</td>
<td>19,375 (labor @$86/hr for 40 hr)</td>
<td>19,375 (labor @$86/hr for 40 hr)</td>
<td></td>
</tr>
<tr>
<td>Validation Samples (20%)</td>
<td>30,000 (at $1000/sample for n=30)</td>
<td>30,000 (at $1000/sample for n=30)</td>
<td></td>
</tr>
<tr>
<td>Cost</td>
<td>$431 per sample</td>
<td>$338 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Validation Cost</td>
<td>$231 per sample</td>
<td>$138 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Instrument Cost</td>
<td>$331 per sample</td>
<td>$331 per sample</td>
<td></td>
</tr>
<tr>
<td>Exclude Instr. &amp; Val. Cost</td>
<td>$131 per sample</td>
<td>$131 per sample</td>
<td>$1,000 per sample</td>
</tr>
</tbody>
</table>

Note:

a. This purchase scenario is based on a unit purchase for a single project. The cost per sample for this scenario would decrease significantly if the purchased unit were costed over multiple projects.
b. QwikSed Supplies
c. Sample shipment costs and data analysis/reporting costs are not included here.
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6.0 IMPLEMENTATION ISSUES

6.1 COST OBSERVATIONS

Collection of data necessary to support decisions at Navy marine sites in a cost-effective manner is often hindered by the complexity and heterogeneity of marine ecosystems. Detailed site investigations require extensive sampling and subsequent laboratory analyses for both metal and organic contaminants. Samples are often collected without any a priori knowledge of the nature and extent of contamination. Because of the high cost of laboratory analyses, the number of samples taken is often cost-limited. Thus, zones of contamination can be missed, or, if located, overestimated or underestimated. For more detailed spatial information on the extent of contamination, sites of interest must often be sampled and analyzed in an iterative manner. Chemical assays are often combined with additional laboratory analyses, including one or several bioassays to determine whether there are adverse biological effects of these contaminants in various media (e.g., sediment, elutriate, water column). This approach can be prohibitively costly, slow, and labor-intensive. When used appropriately, rapid sediment characterization tools can streamline many aspects of the site assessment process, delineating areas of concern, filling information gaps, and assuring that expensive, certified analyses have the highest possible impact.

6.2 PERFORMANCE OBSERVATIONS

The objective was to show these screening techniques had high enough data quality to be regarded as semiquantitative measurements comparable to laboratory methods. The discussion in Section 4 on performance assessment shows that, for the most part, these techniques are rated as Level 2 screening techniques, semiquantitative with a limited number of confirmatory analyses required. For the screening bioassay QwikSed, however, the results are not always directly comparable to these chemical criteria so it is not possible to rate its performance as Level 2.

6.3 SCALE-UP

There are no scale-up issues since the full-scale screening techniques were used for the demonstrations. There may be some additional economy of scale when larger numbers of samples are run in a production line fashion.

6.4 OTHER SIGNIFICANT OBSERVATIONS

The Technology Innovation Office (TIO) at EPA is currently designing guidance on the use of screening technologies for site assessments. As more guidance from headquarters is established, more implementation will be seen at the various regions. Additionally, technology improvements will continue to make field analytical techniques more applicable to site assessments. Improvements in laboratory techniques were driven by the need to reduce laboratory uncertainty in analyses, and now the uncertainty in “representativeness” of each field sample will continue to drive the development of screening techniques. It is anticipated that future assessments will combine laboratory and field analytics in a cost-effective program to characterize sites.
6.5 LESSONS LEARNED

Much of the cost in demonstrations of innovative technologies is in analytical laboratory costs. Each of these screening techniques required laboratory validation data as part of the demonstration. By partnering with ongoing NAVFAC regulatory projects, many of these laboratory costs were paid by the regulatory project since these laboratory measurements were a required element of their project. Unfortunately, the timetable for the ESTCP project then becomes dependent on the regulatory project, which is often delayed for numerous reasons.

Although the regulator community was initially suspicious of screening techniques (due to concerns of adequate detection limits, matrix effects, fears we were going to replace all laboratory data, etc.), once their concerns were addressed they actually became strong advocates of screening techniques. If any innovative techniques are to be successfully employed, successfully addressing regulator concerns is an important component of the process.

6.6 END-USER ISSUES

Within the Department of the Navy alone, there are an estimated 110 facilities with sediment contaminant sites with assessment needs which carry estimated costs of more than $500 million (NAVFAC NORM database). These figures are expected to be even greater for the DoD as a whole. Given the assumptions in Chapter 6 on cost implementation, analytical costs could be expected to be reduced by a conservative 50% if screening techniques were integrated into existing laboratory-based assessment programs.

The transition plan for screening techniques within the Navy is already in progress. The jointly funded ESTCP-NAVFAC demonstrations reported in this report provide the basis for case studies to show screening utility. Additional case studies are available from other sites, including EPA Superfund Innovative Technology Evaluation (SITE) and Environmental Technology Verification (ETV) programs. A series of Remediation Innovative Technology Seminar (RITS) classes during October 2000 at eight NAVFAC sites around the country were used to transition information to remedial project managers (RPMs) from Navy sites. NAVFAC has contracted Battelle (Columbus, Ohio) to run these classes and put together a screening guide for RPM use. This guide will provide RPMs with a short review of screening techniques, standard operating procedures (SOPs) for various screening techniques, and statements of work (SOWs) with generic contract language to facilitate screening use at Navy sites. Since most environmental work at Navy sites is performed by contractor, the transition of these screening techniques mostly occurs via contractor utilization. RPMs must be given the authority to allow screening technologies to be employed by the Department of Navy contractors, including policy that screening should be included as needed in an efficient, cost-effective assessment.

6.7 APPROACH TO REGULATORY COMPLIANCE AND ACCEPTANCE

This demonstration project was designed as part of an ongoing regulatory project to encourage interaction and involvement with regulators. By collecting field screening and standard laboratory data on the same samples during a regulatory project, acceptance of the screening tools will be promoted. During the regulatory process, public participation is allowed through meetings where project status and results are discussed.
In addition to these ESTCP demonstrations, EPA has had screening techniques demonstrated in several programs, including the SITE and ETV programs. Because of the involvement of and interactions with regulators during all these demonstrations, screening techniques are becoming more accepted at sediment assessments in the same manner they are in soil sites. Additionally, EPA is including more screening methods in their standard SW-846 manual of accepted analytical techniques. The proof can be found at websites such as http://clu-in.org/charl.htm, where information and discussions about regulatory acceptance of innovative techniques such as screening are present.
7.0 REFERENCES


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## APPENDIX A

### POINTS OF CONTACT

<table>
<thead>
<tr>
<th>Point of Contact</th>
<th>Organization</th>
<th>Phone/Fax/E-mail</th>
<th>Role in Project</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jim Leather</td>
<td>Space and Naval Warfare Systems Center San Diego 236 53475 Strothe Road San Diego, CA 92152-6310</td>
<td>(619) 553-6240 (619) 553-6305 <a href="mailto:leather@spawar.navy.mil">leather@spawar.navy.mil</a></td>
<td>Overall PI; UVF</td>
</tr>
<tr>
<td>Victoria Kirtay</td>
<td>Space and Naval Warfare Systems Center San Diego 236 53475 Strothe Road San Diego, CA 92152-6310</td>
<td>(619) 553-1395 (619) 553-6305 <a href="mailto:kirtay@spawar.navy.mil">kirtay@spawar.navy.mil</a></td>
<td>XRF</td>
</tr>
<tr>
<td>David Lapota</td>
<td>Space and Naval Warfare Systems Center San Diego 236 53475 Strothe Road San Diego, CA 92152-6310</td>
<td>(619) 553-2798 (619) 553-6305 <a href="mailto:lapota@spawar.navy.mil">lapota@spawar.navy.mil</a></td>
<td>QwikSed</td>
</tr>
</tbody>
</table>