TechData Sheet

Naval Facilities Engineering Command
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Rapid Characterization of Metals in Sediments Using X-Ray Fluorescence (XRF) Technology

Field-Portable XRF: A Rapid Sediment Characterization (RSC) Tool

Why Rapid Sediment Characterization? Marine sediments are more complex and heterogeneous than soil and groundwater media. In addition, sampling activities in sediments are logistically complex, since vessel deployment, sensitive environments, and ocean dynamics are usually involved. A more comprehensive characterization of sediment sites can be achieved with rapid, on-site analytical instruments and techniques. RSC tools are intended to highlight areas requiring more focused and thorough studies.

Issues. Metals such as copper, lead, and zinc are common contaminants found at Navy sediment sites. Site characterization usually involves extensive sampling and laboratory analyses to identify and characterize contaminants. Samples are often collected with little or no knowledge of the nature and extent of contamination. Due to the high cost of laboratory analyses, the number of samples taken is often limited. Therefore, zones of contamination can be missed, or, if located, overestimated or underestimated. Thus, sites of interest must often be re-sampled to provide more spatial information on the extent of contamination. This iterative process can take many months, and can involve remobilization of equipment and project downtime waiting for laboratory results. The Navy has avoided higher costs by using field screening techniques as a characterization strategy in soil and groundwater. Field screening technologies, such as x-ray fluorescence (XRF), provide semi-quantitative data at a low cost, relative to highly quantitative and expensive laboratory analyses. By incorporating field screening techniques into the site characterization process, contaminated areas can be quickly mapped, thereby, focusing on subsequent sampling. As a result, samples selected for expensive certified analysis are likely to resolve regulatory and health concerns.

Technology Description and EPA Method 6200. Commercially available, portable XRF analytical instruments can provide rapid, multi-element analysis of metals in sediment. Samples are exposed to x-ray energy, which liberates electrons in the inner shell of metal atoms. As the outer electrons cascade towards the inner shells to fill the vacancies, energy is released, or fluoresced (Figure 1). The fluorescing energy spectrum identifies the metals and its intensity is proportional to concentration.

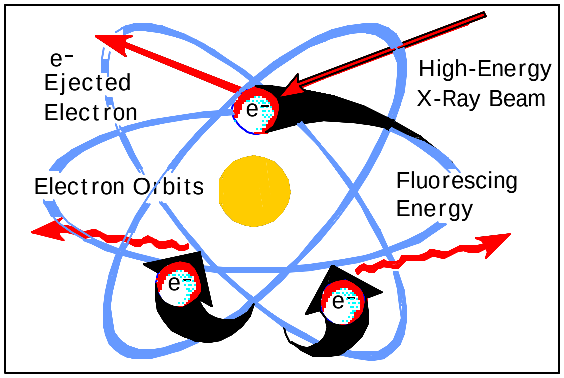

Figure 1. Physics of x-ray fluorescence technology.

This technology was successfully demonstrated under the U.S. Environmental Protection Agency's (EPA) Environmental Technology Verification Program and the Bay Area Defense Conversion Action Team at soil sites. EPA Method 6200 specifies analysis of metals by field portable XRF on dry soil and dry sediment. However, an Environmental Security Technology Certification Program (ESTCP) project showed good results from XRF analyses of unprepared, wet sediment.

What to Expect? XRF is not intended to replace the more rigorous laboratory-based analysis for regulatory purposes. Rather, XRF provides near real-time data to produce a contaminant distribution map while the crew is in the field, without relying on time consuming and costly laboratory analysis to complete the distribution picture. XRF analysis allows better delineation of contaminant distribution by providing higher data density in a time- and cost-effective manner.

Advantages

- ✓ Commercially available field portable equipment
- ✓ High throughput, about 40 samples analyzed per day
- ✓ Near real-time results, data in minutes
- ✓ Simultaneous, multi-element analysis
- ✓ Adequate analysis results on unadulterated sediment samples
- ✓ Low cost analysis allows more data on a fixed budget and higher statistical significance of the data
- ✓ Data are appropriate for mapping trends and designing smarter sampling strategies and plans
- ✓ Measures common sediment contaminants in parts per million range. Typical detection: copper (50-100 ppm), lead (25-50 ppm), zinc (50-100 ppm)

- ✓ No site specific calibration required

Limitations

- ✓ XRF data are not always accepted for regulatory compliance, but can be used to design smart sampling plans
- ✓ Instrument analyzes the sample's surface, XRF may not be appropriate on highly heterogeneous samples
- ✓ High detection levels for some metals, such as mercury, arsenic, cadmium and chromium, relative to regulatory limits

Case Study. The Space and Naval Warfare Systems Center (SSC), San Diego used commercially available XRF instruments to analyze wet, heterogeneous, unadulterated sediment. The samples were retrieved from the Seaplane Lagoon, at the former Naval Air Station Alameda, under an ESTCP project. In general, XRF produced fairly consistent results regardless of multiple variables such as moisture content, heterogeneity, contamination source, and sediment mineralogy. Figure 2 shows a comparison of XRF and certified laboratory analyses on split samples. The XRF was calibrated solely by internal software, and not calibrated to site-specific conditions.

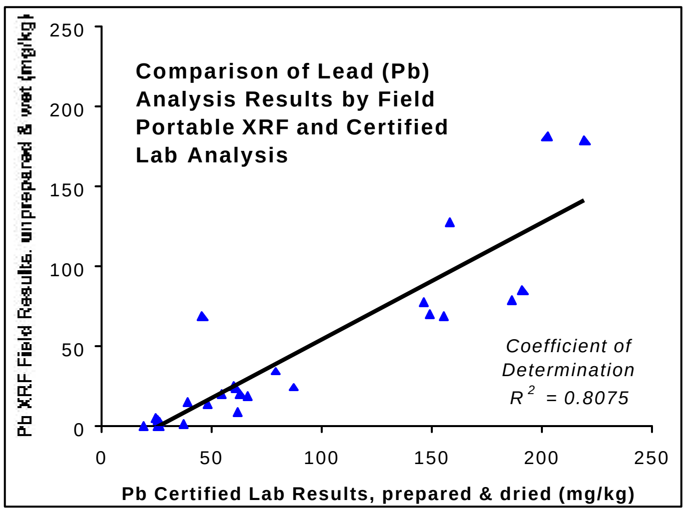

Figure 2. Close correlation in the comparison of XRF and lab results on split samples.

At this site, XRF proved to be a valuable tool for mapping the contaminant distribution. Figure 3 shows discrete sample results based on XRF data, and Figure 4 shows results based on certified laboratory data. Note the distribution characteristics are very similar and suggest the same trends. The XRF generated an accurate map, which can be used to develop intelligent sampling strategies to address regulatory and health concerns at a relatively low cost.

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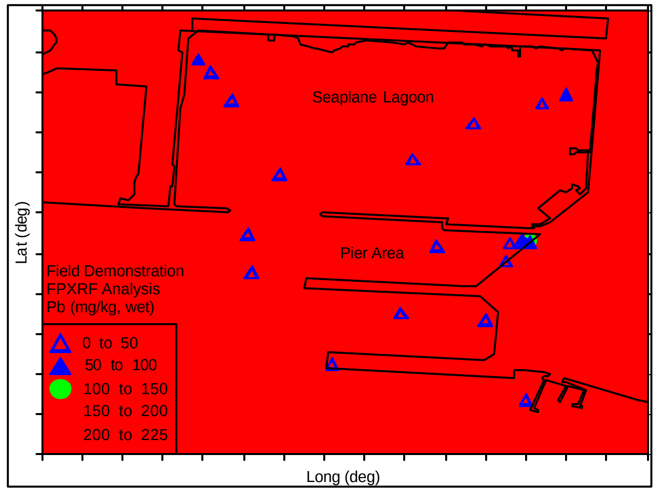

Figure 3. Contaminant distribution model based on XRF data shows good correlation with data in Figure 4.

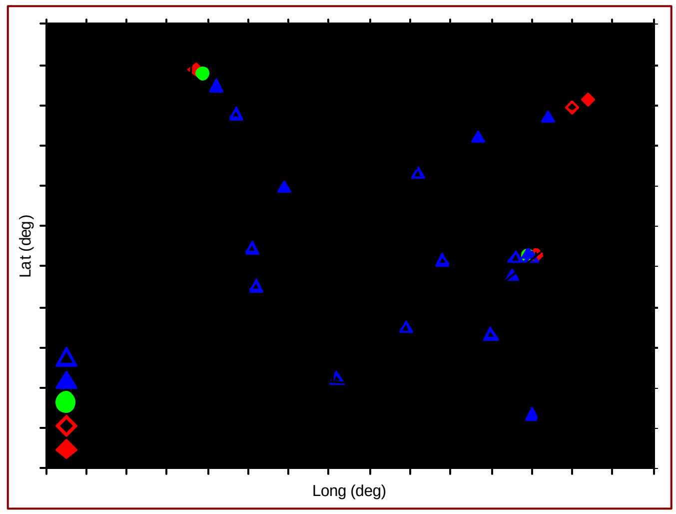

Figure 4. Contaminant distribution model based on lab data.

Cost Avoidance. Using XRF technology to map metal distributions and choosing 25% of those samples for confirmatory laboratory analysis could reduce the overall analytical costs by 50% as compared to the cost of analyzing all of the samples at a laboratory. Such a strategy could yield comparable end results. Another tangible benefit to this RSC technique is better delineation of an area with more data. The estimated sediment volume requiring costly remediation will likely be less than predicted by the traditional sampling strategies, which tend to be more conservative because the volume is estimated with fewer data points. A cost comparison for a site requiring 400 sample points is shown in Table 1.

Table 1. Cost benefit Comparison for 400 Samples

Sampling Method	XRF $90/sample	Laboratory $350/sample	**Total Cost**
RSC w/ X-Ray Fluorescence	400 samples	100 samples	**$71k**
Traditional Characterization	0 samples	400 samples	**$140k**