

The Application of X-Ray Fluorescence Technology for Field Screening of Metal Contamination in Soils

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Introduction

Environmental remediation often includes the removal of metal contamination from surfaces and removal of metal-contaminated soils, sludges and sediments. Generally, defining the scope of clean-up and developing a work plan and methods for decontaminating surfaces or defining the volume of soil and other material to be removed is dependent on the results of laboratory analysis of samples collected in the field. This analysis can be expensive and may take up to a week to complete, increasing the time required to complete a remediation project. X-ray fluorescence (XRF) technology can be used to screen for metal contamination on surfaces and in soil in the field, minimizing the need for laboratory analysis of samples.

A number of manufacturers have developed hand-held XRF instruments capable of field testing of surfaces, soils and other materials in the field. This equipment is portable, light-weight (Niton XL 700 weighs 1.4 kg) and has been validated as compliant with NIOSH and EPA methodologies.

The application of this equipment as a field screening tool is broad, including use in the mining industry to characterize mill heads and tailings and analysis of iron slag waste produced in steel manufacturing. Specific to the environmental remediation industry the range of potential applications of XRF technology is significant. Uses include:

- analysis of metals in soils, sludges and sediments for “Brownfield” investigations;
- site remediation and clearance testing;
- in-situ and ex-situ testing;
- environmental monitoring and characterization;
- analysis of lead and other metals in painted surfaces and other coatings; and
- characterization of metals contamination on building surfaces.

The principle behind this process is simple: when an x-ray emission from a radioactive source strikes a sample, the x-ray can either be absorbed by the atoms or scattered through the material. If absorbed the atoms become “excited” and will then give off a characteristic x-ray whose energy level is unique to the element impacted by the incident x-ray. The emission of these characteristic x-rays is called x-ray fluorescence, or XRF. Measurement of the discrete energy of the x-rays emitted is used to identify the metals species present. Measurement of the number of emitted x-rays provides a quantitative indication of the concentration of the metals present.

Each of the atomic elements present in a sample produces a unique set of *characteristic x-rays* that is a fingerprint for that specific element. XRF analyzers determine the chemistry of a sample by measuring the spectrum of the characteristic x-rays emitted by the different elements in the sample when it is illuminated by high energy photons (x-rays or gamma rays). A fluorescent x-ray is created when a photon of sufficient energy strikes an atom in the sample, dislodging an electron from one of the atom's inner orbital shells (lower quantum energy states). The atom regains stability, filling the vacancy left in the inner orbital shell with an electron from one of the atom's higher quantum energy orbital shells. The electron drops to the lower energy state by releasing a fluorescent x-ray, and the energy of this fluorescent x-ray (typically measured in electron volts, eV) is equal to the specific difference in energy between two quantum states of the dropping electron.

Because the quantum states of each electron orbital shell in each different type of atom (each of the atomic elements) are different, the energies of the fluorescent x-rays produced by different elements are also different. When a sample is measured via XRF, each element present in the sample emits its own unique fluorescent x-ray energy spectrum. By inducing and measuring a wide spectrum of the range of different characteristic fluorescent x-rays emitted by the different elements in the sample, XRF analyzers can rapidly determine the elements present in the sample and their relative concentrations, in other words, the elemental chemistry of the sample.

In typical samples containing many elements, the elements may range in concentrations from high percent levels down to parts per million (ppm). Up to 30 or more elements may be quantified simultaneously by measuring the characteristic fluorescence x-rays emitted by a sample. XRF analyzers quantify elements ranging from sulphur (element number 16 in the periodic table) through uranium (element number 92) to the heaviest transuranic elements, measuring fluorescent x-ray energies from two thousand electron volts (2 keV) up to 100 keV. In certain specialized laboratory applications, XRF analyzers can also measure x-rays of less than 1.5 keV energy in order to quantify elements as light as aluminum and silicon (elements 13 and 14).

Hand-held XRF analyzers are able to quickly, non-destructively determine the heavy elemental composition of:

- metal and precious metal samples;
- rocks and soil;
- slurries and liquid samples;
- painted surfaces including wood, concrete, plaster, drywall and other building materials;
- dust collected on wipe samples; and
- airborne heavy elements collected on filters.

Different metals in soil, dust, wipes, air filters and surface contamination (the method has been proven for 25 metals including: Pb, As, Cd, Cr, Hg, Ag, Ba, Se and others) can be measured quickly and non-destructively using an XRF. Using this technology a trained technician can complete over 100 tests in a single day. Specific methodology has been developed to ensure that the results of in-situ (or field) sampling are comparable to the results one would receive if samples were collected and sent to a laboratory for analysis. In general, more sample preparation (drying, milling and sieving), referred to as ex-situ sampling, will result in greater accuracy. Better measurements can be collected with drier, finer, and more homogeneous particles.

Field trials have been completed and the method has been validated for many applications including:

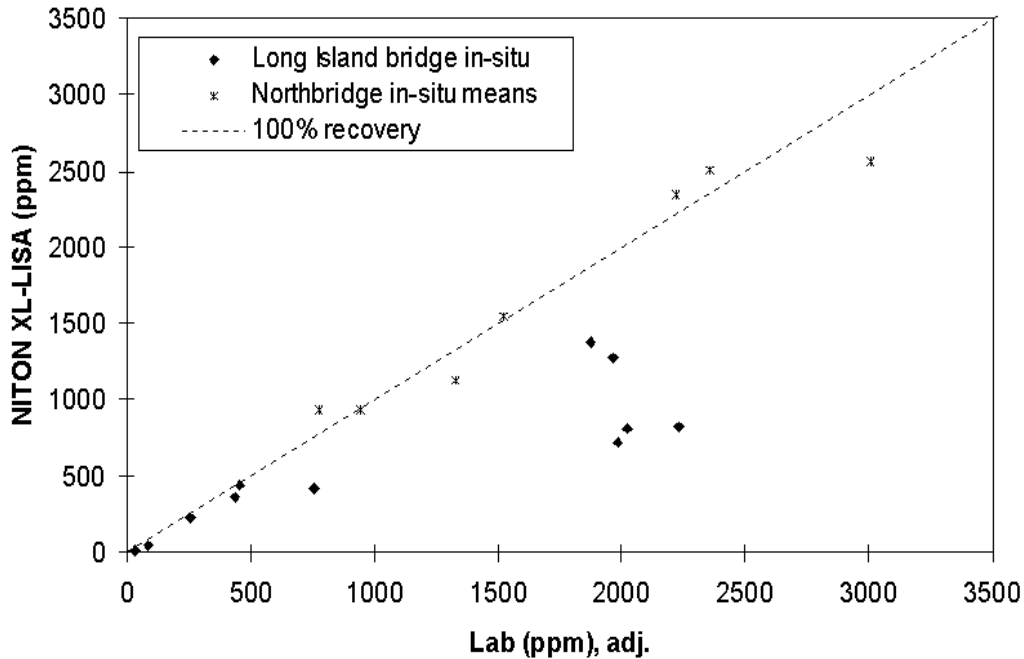
- lead in soil – remediation of a rifle and hand gun range, January 2000;
- arsenic in soil – evaluation of soils and sludges associated with a Canadian power plant, May 2000;
- lead clearance testing – surface testing to confirm completion of lead-based paint removal, lead-based paint abatement project, November 1999;
- lead in soil – assessment of lead in soils associated with a refurbishment of a bridge painted with lead-based paints, January 1997; and
- surface contamination evaluation – assessment of Uranium and Technetium contamination of concrete, soil and sludges during decommissioning of a nuclear power station, July 1996.

The results of the field trials confirmed a good correlation between the XRF and the laboratory. The US Environmental Protection Agency (EPA) has developed method 6200, “*Field Screening Using a Field-Portable XRF*”. The National Institute for Occupational Safety and Health has developed method 7702, “*Lead by Field-Portable XRF*”. The methods suggest that a minimum of 5% of samples tested by XRF be confirmed by an outside lab. The methods do not outline sample collection techniques. In general, more sample preparation (drying, milling and sieving) will result in greater accuracy. Better measurements can be collected with drier, finer, and more homogeneous particles.

The value of in-situ testing has been confirmed. The New York State Department of Transportation¹ conducted a study of the application of the Niton XRF to field testing of lead in air. The scope of work involved air testing for lead in support of the abrasive blasting of two lead-based paint coated bridge structures. Hourly tests were conducted and the results of the XRF were compared to results provided by an off-site laboratory by standard ICP methods. The correlation of the field and laboratory data is presented below.

¹ Zamurs, J. Bass, B. Williams, R. Fritsch, D. Sackett, and R. Heman; “Real-Time Measurement of Lead in Ambient Air During Bridge Paint Removal”, Transportation Research Record, 1998, No. 1641, P 29.

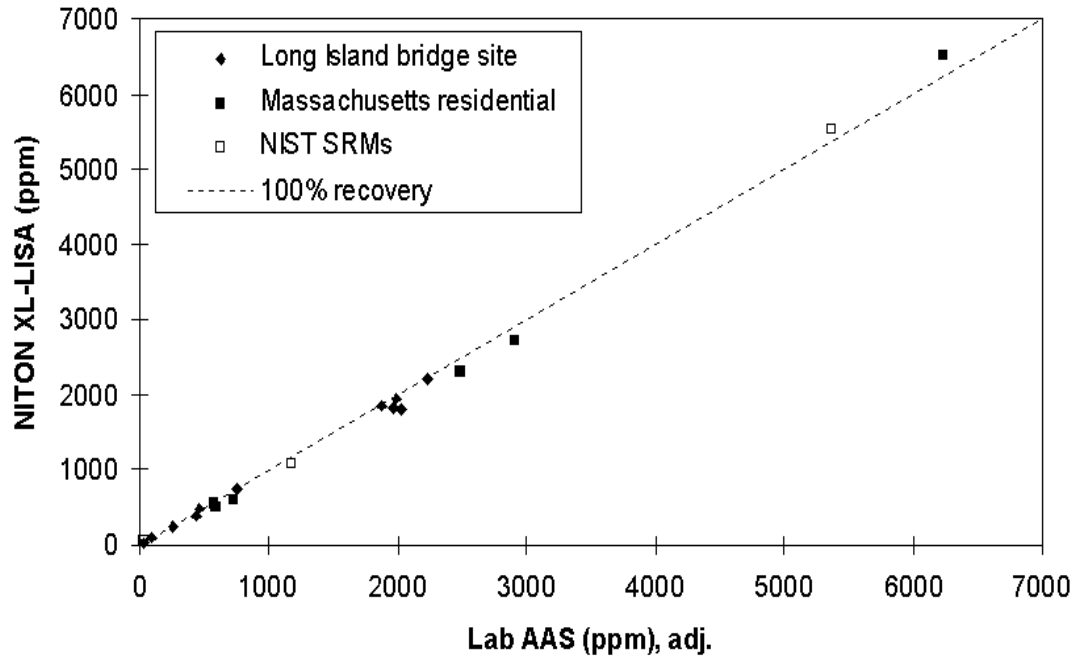
Comparison of in-situ XRF results with laboratory AAS and ICP-AES



The Northbridge site shows a strong correlation. The Long Island bridge site in-situ measurements exhibit strong negative bias, probably due to the concentration of lead in relatively large particles (paint chips). The correlation of in-situ test results to laboratory results has been shown to be related to the homogeneity of the field samples. In field homogenization improves the correlation.

A stronger correlation between ex-situ results and laboratory results has been consistently demonstrated. Ex-situ testing has the added benefit of improving the homogeneity of the samples prior to testing. Sieving and crushing of the samples is completed prior to analysis by XRF. The correlation of the ex-situ data from the New York State Department of Transportation (and data from two other studies) is presented below.

Comparison of fully prepared XRF (oven dried, screened, ground to 0.125 mm or less, and cupped) and laboratory AAS results



The XRF is an effective tool for the field evaluation of metals in soils, sludges, sediments, dust, air, and surface contamination. In-situ testing provides very rapid data that can be correlated to laboratory test methods. Ex-situ test methods have been shown to be very well correlated to laboratory results. This portable technology can be used for making real-time measurements and is a valuable tool for environmental scientists and remediation contractors in completing necessary field testing of metals in soils, sludges, sediments and other materials.