



Comparing Field Portable X-Ray Fluorescence (XRF) To Laboratory Analysis Of Heavy Metals In Soil

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>Presented at the International Symposium of Field Screening Methods
for Hazardous Wastes and Toxic Chemicals

Las Vegas, Nevada, USA
January 29-31, 1997

ABSTRACT

Field portable x-ray fluorescence (XRF) continues to gain acceptance as a complement to traditional laboratory testing of metal contaminated soil. The quality of data produced by field XRF varies with site conditions, soil composition, and sample preparation. Quality assurance protocols for the field method usually require that a number of field samples be split and sent to a laboratory for confirmatory analysis. This confirmatory analysis can provide valuable information of the effectiveness of the field methodology.

We present field and confirmatory data from a variety of contaminated sites that show the effectiveness of field XRF under different site conditions, with different methods of sample preparation. In general, we find that field sample preparation (drying, grinding, sieving, homogenization) significantly improves data quality, compared to unprepared, in-situ measurement. The level of data quality provided by rapid, low-cost in-situ or abbreviated preparation methods can be predicted in the field by the comparison of representative field samples to fully prepared split samples, and can be proven by laboratory confirmation.

We find that the method with which one performs sample splitting for confirmatory analysis can greatly affect the correlation of the field results to the laboratory results. Unexpectedly poor correlation often arises from the introduction of error in the confirmatory sample splitting and sample handling procedures, and which may be misinterpreted as a deficiency of the field method. We discuss ways to avoid the introduction of such error. We also discuss how to use confirmatory analysis to determine the quality of field-obtained XRF data, and we discuss procedures for comparing the field XRF method to the laboratory method.

THE UTILITY OF FIELD METHODS

Field methods can offer significant advantages over laboratory methods, provided they are sufficiently accurate and well-documented to support field decision-making. Field analysis is frequently less expensive per sample than laboratory analysis because of less need for sample handling, transportation, and chain-of-custody documentation. In addition, the rapid analytical turn-around of a field method can provide timely support for field decision-making, and greatly reduce overall project cost. The lower cost-per-sample allows for denser, more complete sampling. And field methods offer the ability to rapidly delineate contaminated areas or "hot-spots", supporting interim control measures and guiding remediation.

Field portable x-ray fluorescence (XRF) is an exemplary field method, offering extremely rapid, cost-effective screening of heavy metals in soil by *in-situ* measurement. It is also versatile enough to provide *ex-situ*, prepared-sample analysis in the field with accuracy that can rival that of standard laboratory analysis. Even in cases where laboratory analysis is required, field XRF can be used to rapidly pre-screen samples (directly through the plastic sample bag), to obtain the optimal utility from the laboratory sampling effort. Since XRF is completely non-destructive, any sample collected and measured in the field can be retained for verification by a laboratory.

While field XRF cannot generally provide the low detection limits attained by laboratory methods, it can often provide detection limits well below regulatory levels. For example, field XRF can easily provide detection limits for lead-in-soil of less than 100 ppm, well below typical regulatory levels of 300 to 1500 ppm.^[1,2,3]

FIELD SAMPLE PREPARATION FOR XRF

The in-situ XRF measurement requires little or no sample preparation. Although the instrument can measure undisturbed soil directly, we recommend a minimal preparation protocol. First, the field operator should remove any debris, such as leaves, twigs, grass, and stones, from the measurement surface. Second, the operator should loosen the soil to a depth of 1.5 to 2.5 cm over an area of at least 10 cm diameter, and stir the loosened soil to achieve some homogenization. The loosened soil may be allowed to dry in the sun for a few hours before the measurement, to improve accuracy. Just before the measurement, the operator should mix and level the loose soil and pack it down gently. For improved accuracy, the operator may screen or comb the loose soil with a 2 mm mesh to remove stones, roots, broken glass, metal fragments, paint chips and other such objects.

Ex-situ measurement offers a variety of sample preparation strategies. A core sampling device may be used to collect the sample to a well-defined depth. A composite sample may be formed by combining soil from several spots in the sample area, mixing thoroughly before measuring. The sample may be dried by spreading it out on a paper and exposing it to sunlight and air, or by using a small field stove or oven. The dried sample can be screened with a 2 mm mesh to remove large objects, and placed in a sample bag, or prepared further. The ultimate field sample preparation for XRF is to grind and sieve the soil to reduce the particle size to less than 0.250 mm (or preferably to less than 0.125 mm), homogenize well, and then sub-sample 3 to 5 grams of the dry, well-ground soil and place in an XRF sample cup for analysis.

The various stages of sample preparation require time and effort, but provide improved measurement accuracy. Core sampling improves the accuracy of the sample definition. Compositing increases the sample support, improving the sample's ability to represent a particular sample area, or "sampling unit". Drying the sample removes the diluting influence of moisture, and facilitates further sample preparation stages of grinding and sieving. Screening the sample with a 2 mm mesh removes the influence of large non-soil particles. Grinding facilitates thorough homogenization of the sample, reducing the effects of fundamental (particle) error and XRF particle-related bias. Sieving with grinding assures complete and accurate particle size reduction. Thorough homogenization assures accurate, unbiased sub-sampling. And the XRF sample cup assures consistent, accurate sample presentation to the XRF instrument. A companion paper^[4] discusses the importance of particle-related effects and their control in detail.

QUALITY ASSURANCE FOR THE LABORATORY AND FIELD METHODS

Quality assurance (QA) is a basic requirement of any analytical method. No measurement has value for decision-making unless its accuracy is known and understood. A quality assurance program should aim to assess the quality and accuracy of all stages of the measurement process, from sample selection and collection through sample handling and preparation and analysis. Significant levels of error can occur at all stages in the measurement process, and accuracy requires that errors at all stages be controlled. Laboratories concentrate a great deal of effort on their QA programs, which assess and control laboratory sample preparation and analytical error. At present, relatively little QA effort focuses on sample collection and sample handling. That is a pity, because much, if not most, of the overall measurement error occurs in the field, not the laboratory. If we do not assess the errors in the field stages, we cannot know the true accuracy of the laboratory-based measurement.

QA programs generally include calibration checks at several concentrations (typically at "background" or low-level, and at moderate to high level), and replicates (collocated or split samples) to assess variation. QA for a field method usually includes verification or confirmatory analysis of some samples, typically by laboratory. Laboratory confirmatory backup may be required for field methods used in decision-making, and assures that the field method is appropriate, effective, and of sufficiently accurate for its purpose. For in-situ XRF, the accuracy can vary significantly from site to site. Fully prepared ex-situ XRF offers the potential for field-based verification of the in-situ XRF method.

The laboratory confirmatory method should match the field method as well as possible. For example, since XRF is a total element method, the confirmatory method should also be a total element method. For lead, most laboratories use atomic absorption spectrometry (AAS) or inductively coupled plasma atomic emission spectrometry (ICP-AES). Both of these methods require that the soil sample be introduced to the instrument as a solution, so the lab must perform sample extraction or digestion. Laboratory analysis of total lead requires a strong acid total digestion to achieve complete dissolution of the sample. Weak acid extraction and leaching-based methods, such as the toxicity characteristic leaching procedure (TCLP), are not appropriate confirmatory methods for the total element XRF method. The most appropriate confirmatory method for XRF would completely digest siliceous minerals, as does EPA draft method 3052. However, total digestion is relatively difficult and expensive, and seldom used in environmental analysis. More commonly used strong acid-based extractions such as EPA methods 3050 and 3051 generally recover most of the heavy metal content, but they cannot recover metals locked within an undissolvable silicate.

ASSESSING TOTAL MEASUREMENT ERROR

Error includes the components of bias and precision (or variation). It is difficult to determine the true measurement bias, because we do not generally know the true concentration of the contaminant in the sampling unit. Instead, we must be satisfied to compare our measurement results against confirmatory results. We can assess the total measurement precision by replicate sampling the sampling unit, and observing the variation of the resulting measurements. To avoid spatial bias in our assessment, we avoid taking replicates from identical sampling locations. Ideally, we select replicate sampling locations randomly throughout the sampling unit. The sampling unit is the volume of soil a particular sample is intended to represent. For example, suppose the sampling unit is a plot running along a 10 meter long wall, from the wall to 2 meters from the wall, and from the surface to a depth of 2.5 cm. The total area of the sampling unit is then 20 square meters, and the total soil volume is 0.5 cubic meters. If the sampling protocol calls for a composite sample of 6 randomly located cores, then replicates should be sampled and composited exactly the same way: as 6 randomly located cores. The greater the number of replicate samples, the more accurately we can determine the total measurement precision. For routine work, it may be sufficient to take only two replicates (that is, one duplicate pair) per sampling unit. The precision may be expressed in terms of relative standard deviation (RSD), or coefficient of variation (COV), which is

simply the standard deviation of the set of replicate sample results divided by the mean of the set.

Total measurement variation may be substantially larger than you expect! It includes the variation in sample representation, sample collection, sample handling, sample preparation (including subsampling and homogenization), and analysis. Particle effects, including fundamental error, can generate serious variation in sampling and subsampling, depending on the particulate form of the contaminant. Soil contaminated by paint chips can exhibit severe particle effects, with relative errors easily exceeding 20 percent; this is discussed in detail in a companion paper.^[4] Another significant contributor to total error is the representativeness of the sample collected. The contaminant is not likely to be distributed evenly through the sampling unit. If we ignore spatial variation and let a single point represent a large area, we can expect relative errors of at least 20 percent. To reduce the effect of spatial variation, we must "increase sample support"; composite a sample from several points in the sampling unit. The total measurement variance, σ_{total}^2 , is given by the sum of the individual component variances:

$$\sigma_{total}^2 = \sigma_{sample\ representation}^2 + \sigma_{sample\ collection}^2 + \sigma_{sample\ handling}^2 + \sigma_{sample\ preparation}^2 + \sigma_{analysis}^2,$$

where the σ 's denote the errors introduced at each stage of the measurement process. The error due to the analytical stage itself, $\sigma_{analysis}$, may be a minor, even negligible contributor to the total error.

Suppose our field method has an analytical error of 10 percent, while the lab method has an analytical error of only 1 percent. You might expect that your choice of field or lab method will seriously affect the total measurement error. Not necessarily so. Suppose the total relative error using the super accurate (1%) lab analysis is 30 percent. Then the total relative error using the field method (precision 10%) ought to be

$$\sigma_{total} = \text{SQRT}((30\%)^2 - (1\%)^2 + (10\%)^2) = 31.6\%.$$

The difference in total error (31.6% versus 30%) is of little or no practical significance. In general, a component of error will affect total error only if it is large relative to the other components. If analytical error is much smaller than sampling and preparation-related errors (and it often is), then it will little affect total measurement error.

ASSESSING THE COMPONENTS OF MEASUREMENT ERROR

To assess the variation due to a particular stage of the measurement process, we prepare identical (replicate) splits and carry the replicates individually through the stage and the remainder of the measurement process. The variance due to a particular stage is calculated from the variance of the identical replicate results, minus the variances due to the remaining measurement stages.

The easiest measurement stage to assess for precision is the final, analytical stage. Analytical replicates entering analysis must be as identical as possible. For XRF this condition is particularly easy to satisfy: replicates can be repeat measurements of the same sample. For the laboratory, analytical replicates can be splits from a well-mixed digestate liquid.

Variance due to the final subsample and packing of the XRF sample cup can be assessed by preparing replicate sample cups from a well-mixed container of ground, sieved sample material. The variance due to subsampling and cupping is the variance of the replicates minus the variance of the analytical replicates.

COMPARING AND CORRELATING DIFFERENT ANALYTICAL METHODS

If you want to compare two different analytical methods, the most accurate assessment of their equivalence will derive from the analysis of identical sample material. The sample material should be split as late as possible in the measurement process to assure that the two analytical methods see similar material. Otherwise, variance will be introduced in intermediate stages that will ultimately degrade the accuracy of the assessment, even if such variance is carefully measured and subtracted from the total.

Suppose we wish to compare two atomic spectrometry methods, AAS and ICP-AES. The sample preparation is identical; both require sample digestion. So we split the sample *after* the digestion. The two methods will measure identical liquid digestate. Any difference between the two measurement results can then be attributed to the analytical stage, not to the sample collection, sample handling, or sample preparation. If we wish to assess variation in the sample preparation stage, that assessment should be performed separately.

Suppose instead we wish to compare AAS with prepared sample XRF. We split the sample after the final stage of XRF sample preparation. The dried, ground, sieved and mixed material will split accurately, giving uniform analyte concentration to each method. Alternately, instead of splitting, we can send the analyzed XRF cup to the lab, assuring that the sample is truly identical. Of course, we do not compare XRF directly to AAS, but to the combination of the digestion method and AAS analysis. We can attribute differences in the results to the digestion and to the analytical methods, but not to variation in the sample collection and handling stages.

But if we wish to compare in-situ XRF with lab AAS, we must split the sample early in the measurement process, since the in-situ method requires so little sample preparation. We should still strive to make the sample splits as identical as possible. The in-situ XRF method measures a small area of ground, only a few square centimeters. As nearly as possible, the sample taken to the lab for comparison to AAS should be removed from the same spot measured by the in-situ method.

When we observe differences between analytical methods, we must bear in mind that significant variation results from the sample collection, handling, and preparation stages. We should always consider the big picture: total measurement error. Unless two analytical methods differ by

a more than a few percent, the impact on total measurement error will probably be insignificant.

EXPERIMENTAL DETAILS

The data presented in this paper come from the study of samples from three lead-in-soil sources. The first source was a site around a highway bridge in Smithtown, Long Island, New York. The soil around the bridge had been contaminated by leaded paint that had come off the bridge through the aging and weathering of the painted surfaces, and through bridge maintenance activities. We observed visible paint chips in many of the samples. The second source was an archive of Massachusetts residential lead-in-soil samples collected by Environmental Science Laboratory, Inc. We believe most of the lead in these samples was derived from paint chips. The third source was a low-income residential tract in Northbridge, Massachusetts where lead-in-soil had been determined to be the cause of 6 childhood lead poisoning cases. The Massachusetts Department of Environmental Protection (MA-DEP) was overseeing remedial activities in the neighborhood at the time the measurements were performed. Since paint chips were not visible in the soil at this site, we believe that most of the lead was contained in finely-dispersed particles. Many of the samples measured high in zinc as well as lead, indicating that the likely contaminant origin was paint. The samples from the three sources represented a variety of soil types and textures, from sandy to loamy to clayey.

All XRF data were collected with a NITON XL equipped with a 10 mCi cadmium-109 radioisotope source, silicon PIN diode detector (750 eV resolution), and the Lead-In-Soil Analysis (LISA) software package. The LISA software reports concentrations in parts per million (ppm) for lead, arsenic, zinc, and copper in soil with matrix corrections determined by Compton normalization.[1] A newer model, the NITON 700, offers similar performance for lead, but with expanded multiple element capability.

In-situ XRF measurements were prepared minimally, by removing debris, loosening the soil, stirring the soil, and flattening the soil before measurement. The bridge samples of approximately 250 grams each were transported raw in heavy plastic bags and measured by the in-situ XRF method in the laboratory. In-situ measurements were 30 seconds in duration, adjusted for source decay. Ex-situ samples of approximately 100 grams each were field-prepared by air and/or sun drying, screening with a 2 mm sieve, then grinding and sieving to 0.250 mm or 0.125 mm. We measured prepared samples in Mylar window XRF cups for 120 seconds duration, adjusted for source decay.

Environmental Science Laboratory (Medway, MA), an ELPAT proficient and A2LA accredited laboratory, analyzed the Long Island bridge samples and the Massachusetts residential samples by flame-AAS. The MA-DEP Wall Experiment Station (Lawrence, MA) analyzed the Northbridge samples by ICP-AES. Both laboratories used microwave-assisted strong acid extractions, and achieved recoveries of 80 to 93 percent on reference materials (RMs). Since Wall Experiment Station reported results in mg/kg wet mass, we calculated mg/kg dry mass with water content determined by gravimetry. We adjusted both laboratory data sets by constant factors to give mean recoveries of 100 percent on RMs.

RESULTS AND DISCUSSION

The XRF method gave excellent performance on reference materials. (Graph 1) A set of 14 measurements on NIST Standard Reference Material (SRM) soils and Environmental Lead Proficient Analytical Testing Program (ELPAT) soils gave a linear regression slope of 0.996 and an R^2 of 0.996. For the 10 reference soils with more than 100 ppm lead, the mean recovery of the XRF was 0.992 and the standard deviation of the recovery was 0.058, for an RSD of 5.8 percent.

Fully prepared XRF samples showed excellent correlation with laboratory AAS for material split after the final grinding, sieving, and homogenization. (Graph 2) A set of 20 fully prepared XRF samples (oven dried, ground to 0.125 mm), including 11 bridge site samples, 6 residential lead samples, and 3 NIST SRM soils, gave a linear regression slope of 1.004 and an R^2 of 0.995. For the 17 samples with lead concentrations above 100 ppm, the mean recovery of the XRF relative to AAS was 0.952 and the standard deviation of the recovery was 0.068, for an RSD of 7.1 percent. The subset of 11 bridge site samples gave a linear regression slope of 0.958 and an R^2 of 0.994. The subset of 6 Massachusetts residential samples gave a linear regression slope of 1.010 and an R^2 of 0.994. We were pleased to observe such strong correlation of widely different analytical methods, especially considering the possibility of less than total lead recovery by the laboratory extraction.

The Massachusetts residential samples yielded an unexpected observation of variation due to standard laboratory protocol for sample preparation. These archive samples had already been dried and ground to pass a 0.500 mm mesh, subsampled, digested and measured by AAS according to standard lab protocol. We ground the samples further, to pass a 0.125 mm mesh, in order to prepare for XRF analysis. We then submitted a 1.0 gram subsample of the finely ground material to the lab for a second analysis, and it is that data which gave an impressive R^2 of 0.994 against XRF. Interestingly, when we compared the XRF data to the original lab data for the same samples, the correlation was decidedly less impressive: R^2 was only 0.958. In fact, the XRF readings correlated with the final AAS readings far better than the original AAS readings did! (Table 1) We believe the better correlation was due to better control of fundamental (particle) error in the laboratory subsampling by the reduction of particle size from 0.500 mm to 0.125 mm. The recovery of the original AAS readings versus the final AAS readings ranged from 0.962 to 1.226, with an RSD of 11.3 percent. This finding supports the argument that fundamental error in subsampling can have a major impact on the precision of the laboratory sample preparation method. If you require better precision than the standard laboratory protocol delivers, consider preparing the sample yourself, by drying, grinding, sieving, mixing, and subsampling, before submitting it to the lab.

Seven field-prepared composite samples (mixed, air and sun dried, ground to 0.250 mm or less) from the Northbridge site, when correlated against the adjusted lab ICP-AES values, gave a linear regression slope of 1.004 and an R^2 of 0.982. (Graph 3 and Table 2) The mean recovery of these 7 samples was 0.997 and the standard deviation of the recovery was 0.066, for an RSD of 6.6 percent.

As expected, in-situ XRF samples did not correlate with the lab as well as did prepared samples, and the performance of the in-situ method varied by site. (Graph 4) The bridge site in-situ method results had a slope of 0.548 and an R^2 of 0.737; negative bias was pronounced on the highest concentration samples. The bridge site in-situ data were all single, uncomposited in-situ readings. The Northbridge in-situ samples, given as mean values of several spots in the sampling unit and compared against a composite sample sent to the lab gave a regression slope of 0.969 and an R^2 of 0.915. We attribute the better in-situ performance at the Northbridge site to a well-dispersed, small particle contaminant.

To examine the effect of spatial variation on single spot in-situ measurements, we recorded individual readings for each of 5 to 6 spots in each of 5 sampling units at the Northbridge site. The five sampling units varied in area from a drip line approximately 10 m long by 0.5 m wide (5 m²) to a rectangular yard of approximately 50 m² area. Compared with the lab analysis of the sampling unit composites, the set of 28 individual spot in-situ readings showed a mean recovery of 0.966 with a standard deviation of 0.320, giving an RSD of 33.1 percent. The means of the 5 to 6 spot in-situ readings per unit gave better correlation with the lab composites: mean recovery was 0.986 with a standard deviation of 0.150, giving an RSD of 15.2 percent. (Graph 5 and Table 2) By averaging 5 to 6 spot in-situ scattered through each sampling unit, we effectively "composite" a sample mathematically, improving sample support. We also retain the spatial data from the individual spot readings, giving us useful insight into site specific spatial variability and representativeness.

CONCLUSIONS AND RECOMMENDATIONS

Field prepared XRF can correlate extremely well with laboratory atomic spectrometry. The total measurement quality depends as much on sample support, collection, handling, and preparation procedures as it does on choice of analytical method. We can determine total measurement precision through replicate sampling within the sampling unit. We can better control overall measurement quality by paying close attention to sampling, handling, and preparation protocols. We can compare different analytical methods most effectively by splitting the sample as late as possible in the measurement process to eliminate variation caused by sample handling and preparation.

In-situ XRF provides rapid, low-cost measurement of heavy metals in soil with a minimum of sample preparation. While the in-situ XRF method is not generally as accurate as the ex-situ prepared sample method, it allows for more thorough sampling of an area to map out contamination patterns and assess spatial variation. The accuracy of the in-situ method depends on site-specific conditions of contaminant particle size and distribution; the accuracy can be assessed in the field by comparison to the prepared sample XRF method.

ACKNOWLEDGMENTS

The author acknowledges contributions to this work by Robert Juliano (Environmental Science Laboratory, Inc.), Mark Begley (Massachusetts Department of Environmental Protection), Michael Kienbusch (Galson Corporation), and Donald Sackett (NITON Corporation).

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Table 1: Massachusetts residential lead-in-soil samples measured by AAS before and after XRF sample preparation. The original samples had been dried and ground to 0.500 mm before subsampling for the microwave-assisted strong acid digestion and AAS analysis. Afterward, the dried ground samples were further ground to 0.125 mm, mixed, subsampled for XRF analysis, and then subsampled for a final microwave digestion and AAS analysis. Correlation coefficient (R^2) between the XRF and AAS values improved from 0.958 with the original AAS results to 0.994 with the final AAS results.

Sample	AAS-original (ppm)	XL-LISA (ppm)	AAS-final (ppm)
9502445	3251	2715	2652
9502446	508	549	524
9502557	605	500	535
9502448	2230	2310	2271
9502449	5487	6512	5704

Table 2: Northbridge samples first measured in-situ (composites averaged), then manually composited (where noted), field prepared and measured by XRF, then split and measured by laboratory ICP-AES. Agreement of in-situ XRF with field prepared XRF predicts agreement with the lab.

Sample	Composite sample?	In-situ XRF mean (ppm)	Field prep'd XRF (ppm)	Lab ICP-AES (ppm), adj.
Dripline C	Y	2561	3155	3004
Dripline 1	N	2512	2325	2354
Yard C	Y	1546	1637	1520
Yard 1	N	2347	2096	2217
Cover area C	Y	1132	1174	1328
Play area C	Y	942	796	774
Doghouse C	Y	932	944	940
Doghouse 1*	N	3632*	N/A*	15213*

*This sample was not dried and ground for field prepared XRF analysis before the laboratory split. Post-split preparation of the sample in a laboratory environment yielded an XRF reading of 12100 ppm. The sample came from a highly localized hot-spot which would not have been discovered without field XRF. Five in-situ method measurements of this non-composite sample ranged from 2976 ppm to 5885 ppm, indicating highly inhomogenous composition. The moisture content was 24.1 percent.

Graph 1: Performance of field portable XRF for lead-in-soil reference materials: NIST SRM numbers 2709, 2710, and 2711, and ELPAT soil samples from rounds 016 and 017.



[{GRAPH 1}](#)

Graph 2: Comparison of fully prepared XRF (oven dried, screened, ground to 0.125 mm or less, and cupped) and laboratory AAS results on Long Island bridge site soil samples, Massachusetts residential lead-in-soil samples and NIST Standard Reference Materials.



[{GRAPH 2}](#)

Graph 3: Comparison of field prepared XRF (field dried, screened, ground to 0.250 mm or less, and cupped) and laboratory ICP-AES for Northbridge lead-in-soil samples.



[{GRAPH 3}](#)

Graph 4: Comparison of in-situ XRF results with laboratory AAS and ICP-AES. 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).



[{GRAPH 4}](#)

Graph 5: Comparison of individual spot in-situ readings and averaged in-situ readings with laboratory ICP-AES measurement of composite samples. Samples from Northbridge site.

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