

X-ray Fluorescence (XRF) Instruments Frequently Asked Questions (FAQ)

The following frequently asked questions (FAQs) address the proper use of field-portable X-ray fluorescence (FP-XRF) instruments to collect data at hazardous waste sites for use in the Integrated Exposure Uptake Biokinetic Model for Lead in Children (IEUBK model). Additional questions regarding the use of XRF instruments to collect data for use with the IEUBK model can be submitted to the TRW Technical Assistance Hotline either by telephone (1-866-282-8622) or e-mail. Please refer to the Lead Workgroups [Technical Assistance](#) page for further information.

What are the advantages of measuring lead concentration with a field-portable XRF (FP-XRF) instrument?

When lead is the main element of potential concern, analysis of samples by FP-XRF (and fixed-base XRF) instruments can offer considerable reductions in cost and time compared to standard CLP methods. Average sample throughput for *ex situ* (see third FAQ below for definition) analysis generally varies from 50 to 100 samples per day, depending on the number of analytes, the particular analyzer used, and the soil preparation protocol. *In situ* (see third FAQ below for definition) analysis allows a greater number of analyses at a given site because little or no sample preparation is performed; however, the loss of precision and accuracy in this mode of operation precludes quantitative site characterization. With FP-XRF instruments, measurements of soil lead concentration can be generated in real-time, allowing decision making in the field regarding the need for additional sampling or further remediation (provided that proper QC procedures are followed - see below). Another advantage of FP-XRF analysis over standard laboratory analysis is that the procedure does not generate investigation-derived waste, because it does not require solvent or acid extraction techniques that are employed by laboratory methods.

What media can be analyzed with an FP-XRF instrument?

XRF instruments are typically used to measure lead in soil, dust, and paint samples. Special techniques allow for measurements of air lead. XRF instruments are not typically used for measuring lead in water.

How is a FP-XRF instrument used to measure lead concentration in soil?

Some FP-XRF instruments can be placed directly on the soil surface for *in situ* measurements. The FP-XRF instrument measures the metal content of the sample over a surface area of approximately one square centimeter (1 cm²) to a depth of approximately 2 millimeters (2 mm), displaying lead concentration in parts per million (ppm). Other FP-XRF instruments require that soil samples are collected and placed in a sample cup that is then placed in a covered sample chamber for analysis (*ex situ* analysis). Most FP-XRF instruments can perform both *in situ* and *ex situ* analyses. Because of limitations on precision and accuracy, *in situ* analysis provides qualitative results. By contrast, *ex situ* analysis can provide semi-quantitative or quantitative results, depending upon the amount of preparation of the soil sample prior to analysis and the calibration standards used. Due to the inherent heterogeneity of soil, *ex situ* analysis is the preferred method

because the soil can be homogenized to provide a sample that is more representative of the concentration of lead at the location from which it was collected.

What is the quality of the data that are generated by an XRF instrument?

Historically, FP-XRF techniques were generally viewed as being suitable only for screening; however, recent advances in technology and instrumentation now permit this method to yield results that are fully comparable with CLP methods for soil and dust. The EPA Environmental Technology Verification (ETV) program evaluated 7 FP-XRF analyzers and found the precision-based detection limits ranged from 30 to 165 ppm in soil, and the relative standard deviations (RSDs) at 5 to 10 times the detection limit were less than 10% ([EPA, 1998 a-f](#)). The ETV program also found that measurements of soil lead concentrations made with FP-XRF analyzers compared well with those made by fixed-based reference laboratories using CLP methods; correlations between the FP-XRF measurements and reference laboratories ranged from 0.85 to 0.97. Once a relationship has been established between FP-XRF results and CLP lab data for a specific site, FP-XRF can provide inexpensive, real-time data concerning lead concentrations in soil. Other studies have also shown good correlation between lead levels measured by CLP and by XRF (including both field-portable and fixed-base measurements)¹.

Has EPA published standard operating procedures for the use of FP-XRF instruments?

Methods for XRF analysis are described in SW-846 (EPA, 1998g), and a FP-XRF protocol is available from the EPA Region 1 [Web site](#). However, it is important that a site-specific QC plan be established to ensure that the data quality objectives (DQOs) for the site are achieved. The Superfund Lead- Contaminated Residential Sites Handbook ([EPA, 2003](#)) and the Abandoned Mine Site Characterization and Cleanup Handbook (EPA, 2000a) also provide suggestions on the proper use of FP-XRF. For example, the Superfund Lead-Contaminated Residential Sites Handbook ([EPA, 2003](#)) suggests 20 percent of the samples be analyzed by both the FP-XRF instrument and a CLP laboratory to develop a site-specific statistical relationship between the two methods. Once the accuracy and precision of the FP-XRF results have been determined (and assuming they satisfy the DQO requirements of the project), the number of laboratory confirmatory samples could be reduced (*e.g.*, to 5 percent). Inherent in this comparison is the determination of the type of sample preparation (*e.g.*, drying and sieving) that is required to ensure that the relationship is consistent ([EPA, 2003](#)). Confirmatory analysis should be focused on samples where the FP-XRF instrument indicates the lead concentration is near the soil cleanup level, if a cleanup level has been determined.

What factors can compromise the quality of FP-XRF data?

There are a number of factors, known as interferences, that can affect the detection limits and precision of FP-XRF instruments. Some interferences can be inherent in the method of analysis, whereas others are the result of the instrument's setup, such as calibration methods. Other interferences may arise from outside sources, such as the sample matrix. The following is a brief overview of some factors that can affect the quality of FP-XRF data:

Sample matrix effects include particle size, uniformity, homogeneity, and condition of the surface. The ETV reports ([EPA, 1998 a-f](#)) indicate the heterogeneity of the sample generally has the greatest effect

¹Validation references listed in the reference list are marked with an asterisk (*).

on comparability with confirmatory samples. Every effort should be made to homogenize soil samples thoroughly before analysis. One way to reduce particle size effects is to sieve all soil samples. Guidance on sieving samples for lead analysis is available in the TRW short sheet on soil sampling and analysis ([EPA, 2000b](#)).

Moisture content above 20 percent can interfere with the analysis, since moisture alters the soil matrix for which the field-portable XRF has been calibrated. This problem can be minimized by drying, preferably in a convection or toaster oven. Drying by microwave can increase variability between the results and can cause arcing if fragments of metal are present in the sample. At some sites, oven drying is an important part of the sample preparation protocol for quantitative analysis to ensure sample homogeneity. The effect of moisture on XRF results is not universal, and the requirement for drying should be determined at each site.

Inconsistent positioning of samples in front of the probe window is a potential source of error because the X-ray signal decreases as the distance from the radioactive source increases. Maintaining a consistent distance between the window and the sample minimizes this problem. For best results, the window of the probe should be in direct contact with the sample.

Chemical matrix effects also can occur as X-ray absorption and enhancement phenomena. The presence of certain metals can interfere with the analysis of certain other metals. For example, iron tends to absorb copper X-rays, whereas chromium levels will be enhanced in the presence of iron. These effects can be corrected mathematically through the field-portable XRF instrument's software. Vendors can typically provide the necessary information during the planning stage to anticipate these interferences.

Instrument resolution limitations may cause problems in analyzing some elements. If the energy difference between the characteristic X-rays of two elements (as measured in eV) is less than the resolution of the detector in eVs, then the detector will not be able to resolve the peaks. In other words, if two peaks are 240 eVs apart, but the resolution of the detector is 270 eV, the detector will have difficulty in differentiating those peaks. A common example is the overlap of the arsenic K peak with the lead L peak. With the use of mathematical corrections that subtract the lead interference, lead can be measured from the lead L peak and arsenic can be measured from the arsenic K peak. However, concentrations of arsenic cannot be calculated efficiently for samples that have lead-to-arsenic ratios of 10 to 1 or more, because the lead peak will overwhelm the arsenic peak completely. Additional information concerning interference from arsenic is available from U.S. EPA Region 8 (U.S. EPA, 2001b) and the ETV FP-XRF reports ([EPA, 1998 a-f](#)).

Where can I find additional information regarding the use of FP-XRF analyzers?

In addition to the sources cited above, other sources of information include *Lead-Safe Yards Developing and Implementing a Monitoring, Assessment, and Outreach Program for Your Community* (EPA, 2001a) and an EPA document that compares FP-XRF instruments (EPA, 2001b). The EPA Technology Innovation Program's [Web site](#) provides documents that describe the use of FP-XRF analyzers in the TRIAD approach to site assessment.

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