



# STANDARD OPERATING PROCEDURES

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## OPERATION OF THE NITON XL722S FIELD PORTABLE X-RAY FLUORESCENCE INSTRUMENT

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\* These sections affected by Revision 1.0

SUPERCEDES: SOP #1700; Revision 0.0; 8/20/04; U.S. EPA Contract EP-C-04-032



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### 1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to outline the steps required for the start-up, check out, operation, calibration, and routine use of the NITON XL722S field-portable x-ray fluorescence (XRF) instrument. The data generated using this SOP meets the Screening Data objective for a quick, preliminary assessment of site contamination, and provides preliminary analyte identification and quantification. Screening data without associated confirmation data are generally not considered to be data of known quality. This SOP supplements the NITON 300series & 700series User's Guide (NITON 2002) that contains detailed information for optimizing instrument performance and for utilizing different applications. The principles of operation are detailed in Appendix A.

Solid and liquid samples may be analyzed with the NITON XL722S for elements potassium (K) through uranium (U) with proper X-ray source selection, application setup, measurement conditions, and instrument calibration. Typical environmental applications include:

- Heavy metals in soil (in-situ or samples collected from the surface or from bore hole drillings), sediments, and sludges;
- Heavy metal air particulates collected on membrane filters, either from personnel samplers or from high volume samplers; and
- Lead (Pb) in paint

The manufacturer recommends operating within a temperature range of 20 to 120 degrees Fahrenheit (°F) and a humidity range of 0 to 95 percent (%) relative humidity (RH).

### 2.0 METHOD SUMMARY

Two radioactive isotope sources, cadmium-109 (Cd-109) and americium-241 (Am-241), are used by the NITON XL722S XRF instrument for the production of primary X-rays. Each of these sources emit a specific set of primary X-rays which excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the appropriate source is selected according to its excitation efficiency for the element of interest. The NITON XL722S instrument can be configured with the appropriate sources depending on the applications provided with the unit.

A sample is positioned in front of the source-detector window and sample measurement is initiated. This exposes the sample to primary radiation from the source. Fluorescent and backscattered X-rays from the sample enter through the detector window and are counted by the high-performance, solid-state detector.

Elemental concentrations are computed based on ratios of analyte X-ray intensity to source backscatter. The raw ratios are corrected for spectral overlap and inter-element effects using correction coefficients and iteratively computed element concentrations. The NITON XL722S is factory calibrated, and the menu-driven software supports multiple calibrations called "applications." Each application is a complete analysis configuration including elements to be measured, interfering elements in the sample, and a set of calibration coefficients. The "Standard Soil Application" for the NITON XL722S may be used when: 1) the percentage of



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the elements of interest are less than (<) 1.0%, 2) the material is of a light matrix (for example, aluminum silicate), and 3) elements with atomic number greater than iron do not exceed several percent.

Measurement time is user controlled. Shorter measurement times (30 to 60 seconds [s]) are generally used for initial screening and hot spot delineation, while longer measurement times (60 to 300s) are typically used for higher precision and accuracy requirements.

### 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

Refer to Section 7.5, Sample Handling and Presentation.

### 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Total method error for XRF analysis is a combination of both instrument precision and user- or application-related error. Instrument precision is typically the least significant source of error in XRF analysis. User- or application-related error is generally more significant and will vary with each site and method used. The following examples of user or application-related errors are discussed below:

- **Sample Placement** - This is a potential source of error because the X-ray signal decreases as the distance from the radioactive source is increased. This error may be minimized by maintaining the same distance for each sample. Sample geometry with respect to the source/detector is also important. A tilted sample may cause analytical error. The NITON XL722S ratios analyte X-ray lines to source backscatter, which minimizes this type of error.
- **Sample Representivity** - To accurately characterize site conditions, samples collected must be representative of the site or area under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentration of the contaminant(s) of concern at a given time and location. Analytical results from representative samples reflect the variation in pollutant presence and concentration range throughout a site. Variables affecting sample representativeness include: (1) geologic variability, (2) contaminant concentration variability, (3) collection and preparation variability, and (4) analytical variability. Attempts should be made to minimize these sources of variability. For additional information on representative sampling, refer to the "Removal Program Representative Sampling Guidance, Volume 1 - Soil" (U.S. EPA/ERT 1991).
- **Reference Analysis** - Soil chemical and physical matrix effects may be corrected (to some extent) by adjusting XRF results (via regression) using site-specific soil samples which have been analyzed by Inductively-Coupled Plasma (ICP) or Atomic Absorption (AA) spectroscopy methods. A major source of error can result if these samples are not representative of the site and/or if the analytical error is large. Additionally, when comparing XRF results with reference analyses results, the efficiency of the sample digestion reference analysis should be considered. Some digestion methods may breakdown different sample matrices more efficiently than others.
- **Chemical Matrix Effects** - Chemical matrix effects result from differences in concentrations of interfering elements. These effects appear as either spectral interferences (peak overlaps) or as X-ray absorption/enhancement phenomena. Both effects are common in soils contaminated with heavy metals. For example, iron (Fe) tends to absorb copper (Cu) X-rays, reducing the intensity of Cu



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measured by the detector. This effect can be corrected mathematically through the use of inter-element correction coefficients.

- Physical Matrix Effects - Physical matrix effects are the result of variations in the physical character of the sample and include parameters such as particle size, uniformity, homogeneity, and surface condition. For example, consider a sample in which the analyte exists in the form of very fine particles within a matrix composed of much coarser material. If two separate aliquots of the sample are prepared in such a way that the matrix particles in one are much larger than in the other, then the relative volume of analyte occupied by the analyte-containing particles will be different in each. When measured, a larger amount of the analyte will be exposed to the source X-rays in the sample containing finer matrix particles, resulting in a higher intensity reading for that sample and, consequently, an apparently higher measured concentration for that element.
- Application Error - Generally, the error in the application calibration model is insignificant (relative to the other sources of error) **PROVIDED** the instrument's operating instructions are followed correctly. However, if the sample matrix varies significantly from the design of the application, the error may become significant (e.g., using the Bulk Sample [soils] application to analyze a 50% iron mine tailing sample).
- Moisture Content - Sample moisture content affects the analytical accuracy of soils or sludges. The overall error may be secondary when the moisture range is small (5 to 20%), or it may be a major source of error when measuring the surface of soils that are saturated with water.

NOTE: Attempting an in-situ measurement on a saturated soil may damage the instrument.

- Cases of Severe X-ray Spectrum Overlaps - When present in the sample, certain X-ray lines from different elements can be very close in energy and therefore, can interfere by producing a severely overlapped spectrum.

Typical spectral overlaps are caused by the K-beta ( $K_{\beta}$ ) line of element Z-1 (or as with heavier elements, Z-2 or Z-3) overlapping with the K-alpha ( $K_{\alpha}$ ) line of element Z. This is the so-called  $K_{\alpha}/K_{\beta}$  interference. Since the  $K_{\alpha}:K_{\beta}$  intensity ratio for the given element usually varies from 5:1 to 7:1, the interfering element, Z-1, must be present in large concentrations in order to affect the measurement of analyte Z. For example, the presence of large Fe concentrations could affect the measurement of cobalt (Co). The Fe  $K_{\alpha}$  and  $K_{\beta}$  energies are 6.40 and 7.06 kiloelectron volts (KeV), respectively, and the Co  $K_{\alpha}$  energy is 6.93 KeV. The resolution of the detector is approximately 300 electron volts (eV). Therefore, large amounts of Fe in a sample will result in spectral overlap of the Fe  $K_{\beta}$  with the Co  $K_{\alpha}$  peak (Figure 1, Appendix B) and the resultant X-ray spectrum will include TOTAL counts for Fe plus Co lines.

Other interferences arise from K/L, K/M, and L/M line overlaps. While these are less common, the following are examples of severe overlap: Arsenic K-alpha ( $As K_{\alpha}$ )/Lead L-alpha ( $Pb L_{\alpha}$ ) and Titanium (Ti)  $K_{\alpha}$ /Barium (Ba)  $L_{\alpha}$

In the As/Pb case, Pb can be measured from the Pb L-beta ( $L_{\beta}$ ) line, and As from either the As  $K_{\alpha}$  or the As  $K_{\beta}$  line; this way the unwanted interference can be corrected. However, due to the limits of



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mathematical corrections, measurement sensitivity is reduced. Generally, as concentrations cannot be efficiently calculated in samples with Pb:As ratios of 10:1 or more. This may result in As being reported as a non-detect, regardless of what the actual concentration is.

The NITON XL722S uses overlap factors to correct for X-ray spectral overlaps for the elements of interest for a given application.

### 5.0 EQUIPMENT/APPARATUS

#### 5.1 Description of the NITON XL722S System

The NITON XL722S is a complete, hand-held, portable XRF analyzer that weighs less than three pounds and is powered from an eight-hour capacity battery. The instrument utilizes the method of Energy Dispersive X-Ray Fluorescence (EDXRF) spectroscopy to determine the elemental composition of soils, sludges, particulate, paint, and other waste materials.

The NITON XL722S analyzer includes two compact, sealed radiation sources: Cd-109 and Am-241. The user selects the source and the analyzer software reports concentrations based on stored information for each application. Measurement time is user determined. The NITON XL722S utilizes a high performance, electrically-cooled, solid-state detector optimized for L-shell and K-shell X-ray detection.

The unit provides internal non-volatile memory for storage of 1000 bulk and/or thin sample spectra and multi-element analysis reports, or up to 3000 paint-mode test results. A RS-232 serial port is provided for downloading results and spectra to a PC. The multi-element analysis reports and spectra can be displayed on the instrument's display screen. The replaceable and rechargeable Nickel Metal Hydride battery pack provides for field-portable operation.

The NITON XL722S is supplied with one or more applications. The "Bulk Sample" (soil samples) application is for analysis of up to 25 metals, where the balance of the sample (that portion not directly measured by the instrument) is essentially silica (SiO<sub>2</sub>). The "Thin Sample" application is for analysis of thin films such as air monitoring filters or wipes. The "Lead-Based Paint" application is for analyzing Pb in paint films. Contact NITON to develop new applications to meet user-specific requirements (e.g., adding elements to the "Soil Samples" application).

#### 5.2 Equipment and Apparatus List

##### 5.2.1 NITON XL722S Analyzer System

- Hand-held analyzer unit for data acquisition, processing, and display, including a high-performance, solid-state detector, two excitation sources (Cd-109, Am-241), data processing software, and control panel/results display
- RS-232C Serial Input/Output (I/O) Interface cable
- Battery charger



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- Two battery packs, rechargeable nickel metal hydride
- System carrying/shipping case and field carrying case/holster
- Soil sample analysis/preparation accessories in separate carrying/shipping case
- NITON XL722S User's Guide and NITON Xtras PC utilities software.

### 5.2.2 Optional Items

- 31-millimeter (mm) diameter sample cups
- XRF polypropylene film, 0.2 mil thick
- Windows 2000-based Personal Computer (PC)
- Spare battery packs and spare charger
- Sample preparation accessories (such as drying ovens, grinders, sieves, etc.) for mobile or stationery laboratory use

See the NITON XL722S Accessories List for additional options.

### 5.3 Peripheral Devices

The NITON XL722S may be used with a PC to download results/spectra, and for customized reports.

#### 5.3.1 Communication Cable Connection

Plug the round end of the RS-232 Serial I/O cable into the NITON XL722S connector (the connection left of the off/setup/on switch) and the nine-pin connector of the cable into the serial port of the PC.

#### 5.3.2 NITON Xtras 5.9e Software

The PC must be running the NITON XTras v5.9e software to communicate with the NITON XL722S. The XTras software allows you to select various configurations for downloading, exporting, displaying, and reporting results/spectra. Refer to the NITON User's Guide and XTras Quickstart Guide for details.

## 6.0 REAGENTS

- SiO<sub>2</sub> check sample, used as a negative control or blank check
- National Institute of Standards and Technology (NIST) soil standard reference materials (SRMs) #2709, 2710, and 2711, target element response check used for accuracy. Refer to Appendix C for certified values and certificates of analysis.





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- Resource, Conservation, and Recovery Act (RCRA) check sample, 500 parts per million (ppm) nominal concentration (NOT Certified), used for precision
- Other NIST and/or precision standards depending on site requirements.

### 7.0 PROCEDURES

#### 7.1 Instrument Operation

##### 7.1.1 Startup

To remove the battery pack, loosen the two screws on the end of the unit below the off/setup/on switch and gently lift the battery pack away from the connector on the unit's base. Insert a freshly charged battery pack and tighten the set screws.

Depress and slide the off/setup/on switch to the "ON" position. Startup may be delayed due to the instrument's battery-saving feature. If the NITON does not turn on immediately, turn it off, wait a few seconds, and turn it on again. Each time the NITON is turned on, the Main Menu appears and the screen arrow points to **→Calibrate & Test ←**.

Allow the NITON to warm up for a minimum of 15 minutes before performing analysis.

##### 7.1.2 Precautions

The NITON XL722S should be handled in accordance with the following radiological control practices.

**Refer to the NITON User's Guide for detailed discussion of Radiation Safety practices.**

1. The NITON XL722S should always be in contact with the surface of the material being analyzed and the material should completely cover the aperture when the sources are exposed. Do not remove a sample or move the unit while the shutter is open.
2. When the sources are exposed, under no circumstances should the NITON XL722S be pointed at the operator or surrounding personnel.
3. Do not place any part of the operator's or co-worker's bodies in line of exposure when the sources are exposed or partially covered.
4. The shutter must be closed with the shutter safety lock engaged when not in use.
5. The manufacturer (NITON) must be notified immediately of any condition or concern relative to the NITON XL722S's structural integrity, source shielding, source switching condition, or operability.



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6. The appropriate state agency or the Nuclear Regulatory Commission (NRC) office must be notified immediately of any damage to the radioactive source, or any loss or theft of the device (see factory supplied data on radiological safety).
7. Labels or instructions on the NITON XL722S(s) must not be altered or removed.
8. The user must not attempt to open the unit.
9. The source(s) in the unit must be wipe-tested every six months as described in the NITON XL722S User's Guide. The leak test certificates must be kept on file, and a copy must accompany the instrument at all times.
10. The bulk test platform or equivalent sample stage provided by NITON LLC must be used whenever the NITON XL722S is used for measuring samples contained in cups.
11. The NITON XL722S should not be dropped or exposed to conditions of excessive shock or vibration.

Additional precautions include:

1. The NITON XL722S should always be stored in its waterproof, drop-proof carrying case.
2. The battery charging unit should only be used in dry conditions.
3. Battery packs should be changed only in dry conditions.

#### 7.2 Control Panel and Menu Software

This section outlines the control panel buttons and basic menu software. Detailed illustrations of the control panel and screen displays are in the NITON User's Guide.

##### 7.2.1 Control Panel Buttons

The NITON control panel consists of three buttons; Clear/Enter, right arrow (▶), and left arrow (◀). These buttons allow the operator to navigate all the NITON screens and menus. The amount of time that the button is held down also controls the function of the buttons. Pressing the Clear/Enter button briefly (less than 1 second) or pressing the right (▶) or left (◀) arrow buttons scrolls through the listed items shown on the screen. Holding down the Clear/Enter button for a longer period (more than 3 seconds) activates a different screen.

##### 7.2.2 The Setup Menu

The Setup Menu is used to check instrument specifications, to set date and time, to illuminate



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the screen continuously, or to select a different testing mode. Once set up, this screen will remain the same each time the NITON is turned on until it is reset. To activate, select Setup Menu from the Main Menu with the arrow buttons and press the Clear/Enter button to enter the Setup Menu. Refer to the NITON User's Guide for detailed instructions on using the Setup Menu.

### 7.2.3 Calibrate & Test

When →Calibrate & Test← appears on the display, press the Clear/Enter button to start the self-calibration process. This process calibrates detector energy gain/zero so that analyte X-rays are in their proper spectral location. Self-calibration takes about one to two minutes. When it is complete, the instrument will beep and the →Ready to Test← screen will appear. The self-calibration process should be performed every 2 to 4 hours during sample analysis to maintain proper detector calibration.

### 7.2.4 The Ready to Test Screen

This screen displays: the current date and time, the instrument serial number, the indication that the instrument is ready to test, the testing mode, the action level for "positive" or "negative" determination of lead in paint (lead based paint application only), the detector energy resolution, and the source strength.

**CAUTION: Check the date and time. If they are not correct, reset them before taking any measurements (see NITON User's Guide). Readings will not be accurate unless date and time are correct.**

### 7.2.5 The Measurement Screen

The highest concentration elements are displayed in ppm (with the two-sigma confidence intervals) on the first measurement screen. The test time is also displayed.

### 7.2.6 The Summary Screen

When the operator ends a reading, the Measurement Screen is replaced by the Summary Screen. Results are displayed for 14 elements on NITON XL700 series model. These are divided into two groups: detected elements, and elements that were not detected. Press the arrow buttons to scroll through the element list. An element is classified as detected when the measured concentration (ppm) is at least 1.5-times the confidence interval (i.e., 3-sigma).

Detected elements are displayed as in the Measurement Screen. Non-detected elements are shown as "< xx", where xx is the three-sigma instrument detection limit for that sample. The instrument detection limit (3-sigma) for each element is calculated for each sample.

## 7.3 Preoperational Checks

### 7.3.1 Energy Calibration



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An energy calibration (Calibrate & Test) must be performed each time the analyzer is used or as required (e.g., after an instrument is shipped) to ensure proper energy calibration. The Calibrate & Test function is located in the Main Menu (see section 7.2.3). If the instrument does not calibrate properly, push the reset button and re-calibrate.

NOTE: When sudden large changes in ambient temperature occur, the instrument may require re-calibration.

### 7.3.2 Resolution Check

The resolution check examines the detector's ability to resolve X-ray energies (Figure 2, Appendix B). This must be performed once at the beginning of the day. Record/document the Energy Resolution in the Ready to Test screen (after Calibrate & Test). The value should not vary significantly from day to day and should typically be less than 400 eV. If the unit fails to meet this specification, call NITON for assistance. NOTE: The lower the number, the better the instrument will perform.

### 7.3.3 Blank (Zero) Sample Check

The blank (Zero) sample check is performed to monitor the instrument's zero drift in the selected application. The blank sample check only applies to the application (test mode) currently selected. This should be done once at the beginning of the day after Calibrate & Test, after selecting a test mode, and whenever the instrument exhibits a persistent drift on a blank or low-level sample.

Load the SiO<sub>2</sub> Blank (supplied with the NITON unit) in the NITON Bulk Sample Test Platform. Analyze for 60 seconds (source seconds) with each source in the unit. Review results. All elemental results should be reported as non-detected (<xx, where xx is the 3-sigma instrument detection limit). Repeat the measurement if the unit fails to meet these specifications. If several elements continue to be significantly out of these specifications, check the plastic window and the blank sample for contamination. Perform the blank (Zero) sample check again. Save the results/spectra for documentation.

### 7.3.4 Target Element Response Check

The purpose of the target element response check is to ensure that the instrument and the selected application are working properly prior to performing sample analysis. This check should be performed at the beginning of the day. Use NIST SRMs 2709, 2710, and 2711 standards provided with the NITON unit to check the Soil Samples application. These samples should be measured using the same source acquisition times that will be used for sample analysis. Save the sample check results/spectra for documentation.

## 7.4 Source Measuring Time

The source measuring time is user controlled. Generally, the element detection limit is reduced by 50 percent for every four-fold (x4) increase in source measuring time. Although counting statistics



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improve as measurement time increases, the practical upper limit for typical applications is about 300 seconds.

The NITON XL700 units measure time in “source seconds”. This includes an automatic correction for source decay so that 60 source seconds will have a constant precision regardless of source age. The correction extends measurement time to correct for source strength lost through the decay process.

A minimum measuring time of 60 source seconds for each source is recommended when using the Soil Samples application. Measuring times for a source that excites a target element can be increased if lower detection limits are required.

#### 7.5 Sample Handling and Presentation

When making XRF measurements, be sure to maintain constant measurement geometry in order to minimize variations in analysis results. Document any anomalies in measurement geometry, sample surface morphology, moisture content, sample grain size, and matrix (see Section 4.0).

##### 7.5.1 Soil Samples

Soil samples may be analyzed either in-situ or in XRF sample cups (after preparation). Appendix D summarizes sample preparation methods for field portable x-ray fluorescence (FPXRF) analysis. The Soil Samples application assumes the sample to be infinitely thick. For in-situ measurements this is the case, however, for sample cup measurements it is advisable to fill the cup nearly full and use the supplied paper disk and cotton ball to hold the sample firmly against the sample cup window. This ensures that the sample is as uniformly thick as possible from analysis to analysis. The NITON XL722S bulk sample test platform or equivalent bulk sample platform provided by NITON must be used when analyzing sample cups.

An area for in-situ analysis should be prepared by removing large rocks, vegetation, and debris. The soil surface should be flat and compact prior to analysis. The NITON XL722S should be placed in the in-situ adaptor and held firmly on the ground to maximize contact with the ground. The unit should not be moved during analysis. Analysis of water-saturated soils should be avoided.

Coarse-grained soil conditions or nuggets of contaminated material may preclude a truly representative sample and adversely affect the analysis results. Such samples should be prepared before analysis. Preparation consistency is important to minimize variation in analytical results.

This application is designed for soil with the assumption that the balance of the material is essentially silica. If samples with a much lighter (lower atomic number) balance are analyzed, the results may be elevated. Contact NITON for help with the analysis of different matrices.

##### 7.5.2 Thin (Filter) Samples



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The Thin Samples application is for analysis of thin samples such as particulates on filters or wipes. The detection limits are affected by the thickness of the substrate. Best results are obtained on the thinnest substrates. Always use the Dust wipe and Filter Test platform when measuring thin samples. This is not only for user safety, but also ensures a controlled environment to facilitate testing thin samples. Contaminated material captured on filters or wipes is not usually deposited uniformly. Therefore, to produce meaningful results, several readings must be taken for each thin sample measurement. The average or sum of these readings is the reported value for the measurement. Refer to the NITON User's Guide for details on analyzing Thin Samples.

#### 7.5.3 Lead in Paint

To analyze for lead in paint on a surface (e.g., wall, counter, etc.), the area selected for analysis should be smooth, representative and free of surface dirt. The NITON XL722S should be held firmly on the surface to maximize contact. The probe should not be moved during analysis. Refer to the NITON User's Guide for details on analyzing lead paint samples.

#### 7.6 Downloading Stored Results and Spectra

Results (analytical reports) and spectra which have been stored in the NITON XL722S internal memory should be downloaded and captured in disk files on a PC (see section 5.3). NITON LLC provides software (Xtras 5.9e) for this purpose. Additionally, results or spectra may be exported to text files for importing into a spreadsheet. Refer to the instructions provided with the programs for details on their operation.

After capturing results to a file, print a copy and save both the disk files and the printout for future reference and documentation purposes.

#### 7.7 Instrument Maintenance

**NOTE: All service except exterior cleaning must be performed by NITON LLC. Do not attempt to make repairs yourself. Opening the case of the NITON XRF instrument will void the Warranty.**

##### 7.7.1 Exterior Cleaning

When the Kapton plastic window on the bottom of the instrument becomes dirty, the performance of the NITON unit will be affected. Clean the window gently with cotton swabs. Clean the instrument's metal case with a soft cloth. Never use water, detergents, or solvents. These may damage the instrument.

##### 7.7.2 Further Information and Troubleshooting



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Refer to the NITON XL722S User's Guide for additional detailed operational and/or maintenance and troubleshooting instructions. If no solution is found in the manual, contact NITON LLC for assistance.

An instrument log should be maintained to document specific corrective actions taken to alleviate any instrumental problems, or for recording any service that has been performed.

### 7.8 Reporting Results

All raw XRF data should be recorded including the individual results of multiple analyses of samples and sampling points. The average and concentration range of each multiple analysis should also be reported.

A "reported" value for each analysis or average of multiple analyses should be processed in the following manner.

1. Round the value to the same degree of significance contained in the calibration or check standard sample assay values (usually two). Round to two significant figures for sample results. DO NOT round results for standards used to determine method detection limit (MDL) or relative standard deviation (RSD) values (use raw data).
2. Report all values less than the reporting limit (RL) as not detected (U).
3. OPTIONAL: Flag and note all values greater than or equal to ( $\geq$ ) the MDL and less than the RL (usually with a "J" next to the reported value).
4. Report all values equal to or greater than the RL. The linear range for the NITON XL722S generally extends to 20,000 to 40,000 ppm. While the unit may be used for higher concentrations, these results should be verified by laboratory analyses.

### 8.0 CALCULATIONS

The NITON XL722S is a direct readout instrument that does not require any external calculations.

### 9.0 QUALITY ASSURANCE/QUALITY CONTROL

In addition to the specific quality assurance (QA) activities listed below, the following general QA procedures apply:

- All sample data, pre-operational and operational checks must be documented in the instrument run or analysis log;
- The instrument must be operated in accordance with this SOP and the manufacturer's recommendations; and
- Preventive maintenance is conducted at the intervals recommended by the manufacturer.



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Results may also be recorded electronically on a hard drive or floppy disk.

### 9.1 Calibration Check

The self-calibration or energy calibration (Calibrate and Test, Section 7.2.3) must be performed each time the instrument is used and may be performed every 2 to 4 hours during sample analysis to maintain proper detector calibration.

### 9.2 Resolution Check

A resolution check must be performed and documented at the beginning of each day or eight-hour shift.

### 9.3 Blank Check

The blank check must be performed and documented at the beginning of each day or eight-hour shift, after calibration, after selecting a test mode, or whenever the instrument drifts on the blank or a low-level sample.

### 9.4 Precision

The precision of the method is monitored by reading a low- or mid-level sample (e.g., SRM or RCRA sample) at the beginning and end of sample analysis and after approximately every tenth sample. Determining the precision around the site action level can be extremely important if the XRF results are to be used in an enforcement action. Therefore, selection of a sample with a target element concentration at or near the site action level or level of concern is recommended. The sample is analyzed by the instrument for the normal field analysis time, and the results are recorded. A minimum of seven measurements should be made during field activities. The standard deviation for each target element is calculated. The RSD of the sample mean can be used to calculate precision. The percent relative standard deviation (%RSD) should be within  $\pm 20$  percent (U.S. EPA/ERT 1991).

### 9.5 Method Detection Limit and Reporting Limit

The MDL and RL are dependent upon site conditions and data quality objectives and, therefore, are site/project specific. They must be calculated from the measurement of either a low or blank sample (or a SRM) at the start and end of sample analysis, and after approximately every tenth sample. Alternatively, the SiO<sub>2</sub> blank or "clean" sand may be used if a blank soil or sediment sample is unavailable.

Determine the MDL using the same application and measuring time used for routine samples. A minimum of seven measurements should be made during field activities. Calculate the sample standard deviation of the mean ( $\sigma$ ) for each target element, and round up to the next whole number prior to calculating the MDL and RL.

The MDL is defined as follows:





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$$MDL = t(n - 1, 99) * \sigma_s$$

where:

$t(n - 1, 99)$  = student's t-value for a 99% confidence level and a standard deviation estimate with  $n - 1$  degrees of freedom, and

$\sigma_s$  = sample standard deviation ( $n - 1$  degrees of freedom)

Since FPXRF is a screening technique, the definition of the RL is 1 to 5 times the MDL depending on element, soil type, site data quality objectives, and operator professional judgment.

Appendix E lists typical MDL values for the NITON XL722S FPXRF analyzer.

### 9.6 Accuracy

The results obtained using NIST SRMs #2709, 2710 or 2711 should fall within  $\pm 20\%$  of the true value for contaminant concentrations at least five times the XRF MDL. Certified concentrations are listed in Appendix C.

### 9.7 Confirmation Analysis

The comparability of the data, relative to a specific digestion method and elemental analysis procedure, is determined by submitting a sample analyzed by XRF methods (prepared sample cups may be submitted) for AA or ICP analysis at a laboratory.

The on-site analysis of soils by FPXRF instrumentation should be considered screening data only [data category (SD)]. Data derived from the instrument should be used with discretion. Confirmatory analyses on a subset of the screening samples (minimum 10%) can be used to determine if the XRF data meets the Screening Data with Definitive Confirmation (SD/DC) data objective. The confirmation samples should ideally be selected randomly from the sample set. A random selection of samples at or near the critical level may also be selected for confirmation in addition to the original random selection. The results of the laboratory analysis (dependent) and the XRF analysis (independent) are evaluated with a regression analysis. The coefficient of determination ( $r^2$ ) should be 0.7 or greater (U.S. EPA/ERT 1991).

Correcting the XRF results based on confirmatory analyses should only be undertaken after careful consideration. The confirmatory analysis (AA or ICP) is an estimate of the concentration of metal contamination and is dependent upon the digestion method and sampling methodology used. Since XRF is a total elemental technique, any comparison with referee results must account for the possibility of variable extraction efficiency, dependent upon the digestion method used and its ability to dissolve the waste or mineral form in question (see Appendix C, NIST Certificates of Analysis for SRMs 2709, 2710, 2711).

### 9.8 Matrix Considerations

Other types of quality assurance/quality control (QA/QC) verification should include verification that



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the instrument calibration is appropriate for the specific site to be assessed. This includes verification of potential multiple soil matrix types that may exist at a site. Matrix differences which affect the XRF measurement include large variations in calcium content, which may be encountered when going from siliceous to calcareous soils, as well as large variations in Fe content.

### 10.0 DATA VALIDATION

#### 10.1 Screening Data

The XRF analyst reviews the data prior to submittal to the client ensuring that the instrument has been operated in accordance with this SOP and manufacturer's recommendations and that all QA/QC checks have been performed. Screening data is evaluated for calibration and detection limits criterion only.

#### 10.2 Screening Data with Definitive Confirmation

Confirmation samples are recommended at a minimum rate of 10% and are required if SD/DC data objectives have been established for site activities. Ideally, the sample cup that was analyzed by XRF should be the same sample that is submitted for AA/ICP analysis. When confirming an in-situ analysis, collect a sample from a 12-inch by 12-inch area for both an XRF measurement and confirmation analysis.

The XRF and confirmatory AA/ICP results are analyzed with a regression analysis using a statistical program (such as SAS®) or a spreadsheet with the intercept calculated in the regression. The  $r^2$  between XRF and AA/ICP data must be  $\geq 0.7$  for the SD/DC data objective.

### 11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow Environmental Protection Agency (EPA), Occupational Safety and Health (OSHA), corporate and/or any other applicable health and safety practices. Be sure to read the precautions associated with this instrument in Section 7.1.2.

### 12.0 REFERENCES

NITON 300series & 700series User's Guide, Version 5.2, 1998. XTras QuickStart Guide for version 5.7, <http://www.niton.com>, 2002 (Available for download as a pdf file).

U.S. EPA/ERT, "Representative Sampling Guidance, Volume 1 - Soil," November, 1991 (OSWER Directive 9360.4-10).

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### 13.0 APPENDICES

A - Principles of Operation

B - Figures



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- C - Certified Standard Values and Certificates of Analysis
- D - Sample Preparation for FPXRF Analysis
- E - Typical MDL Values for the NITON XL722S FPXRF Analyzer

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APPENDIX A  
Principles of Operation  
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## OPERATION OF THE NITON XL722S FIELD PORTABLE X-RAY FLUORESCENCE INSTRUMENT

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### Principles of Operation

X-ray Fluorescence spectroscopy is a non-destructive, qualitative and quantitative analytical technique used to determine the chemical composition of samples. In a source-excited XRF analysis, primary X-rays emitted from a sealed radioisotope source are utilized to irradiate samples. During interaction with samples, source X-rays may either undergo scattering (dominating process) or absorption by sample atoms in a process known as the photoelectric effect (absorption coefficient). This phenomenon originates when incident radiation knocks out an electron from the innermost shell of an atom creating a vacancy. The atom is excited and releases its surplus energy almost instantly by filling the vacancy with an electron from one of the higher energy shells. This rearrangement of electrons is associated with the emission of X-rays characteristic (in terms of energy) of the given atom and this process is referred to as emission of fluorescent X-rays (fluorescent yield). The overall efficiency of the fluorescence process is referred to as excitation efficiency and is proportional to the product of the absorption coefficient and the fluorescent yield.

### Characteristic X-rays

The NITON XL722S uses characteristic X-ray lines originating from the innermost shells of the atoms: K, L, and occasionally M. The characteristic X-ray lines of the K series are the most energetic lines for any element and, therefore, are the preferred analytical lines. The K lines are always accompanied by the L and M lines of the same element. The energies of the L and M lines are much lower than the K lines and can usually be neglected for those elements for which the K lines are analytically useful. For heavy elements such as cerium (Ce) (atomic number,  $Z=58$ ) to uranium (U,  $Z=92$ ), the L lines are the preferred lines for analysis. The L-alpha ( $L_{\alpha}$ ) and L-beta ( $L_{\beta}$ ) lines have almost equal intensities, and the choice of one or the other depends on what interfering lines might be present. A source just energetic enough to excite the L lines will not excite the K lines of the same element. The M lines will appear together with the L lines. The NITON User's Guide contains information about the X-rays (K or L) and elements that are measured for each excitation source.

An X-ray source can excite characteristic X-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element (e.g., K absorption edge, L absorption edge, M absorption edge). The absorption edge energy is somewhat greater than the corresponding line energy. The K absorption edge energy is approximately the sum of the K, L, and M line energies, and the L absorption edge energy is approximately the sum of the L and M line energies of the particular element.

Energies of the characteristic fluorescent X-rays are converted (within the detector) into a train of electric pulses, the amplitudes of which are linearly proportional to the energy. An electronic multichannel analyzer measures the pulse amplitudes, which is the basis of a qualitative X-ray analysis. The number of counts at a given energy is representative of element concentration in a sample and is the basis for quantitative analysis.

### Scattered X-rays

The source radiation is scattered from the sample by two physical processes: coherent or elastic scattering (no energy loss), and Compton or inelastic scattering (small energy loss). Thus, source backscatter (background signal) consists of two components with X-ray lines close together. The higher energy line is equal to the source energy. Since the whole sample takes part in scattering, the scattered X-rays usually yield the most intense lines in the spectrum. Furthermore, the scattered X-rays have the highest energies in the spectrum and, therefore, contribute most of the total measured intensity signal.



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APPENDIX B  
Figures  
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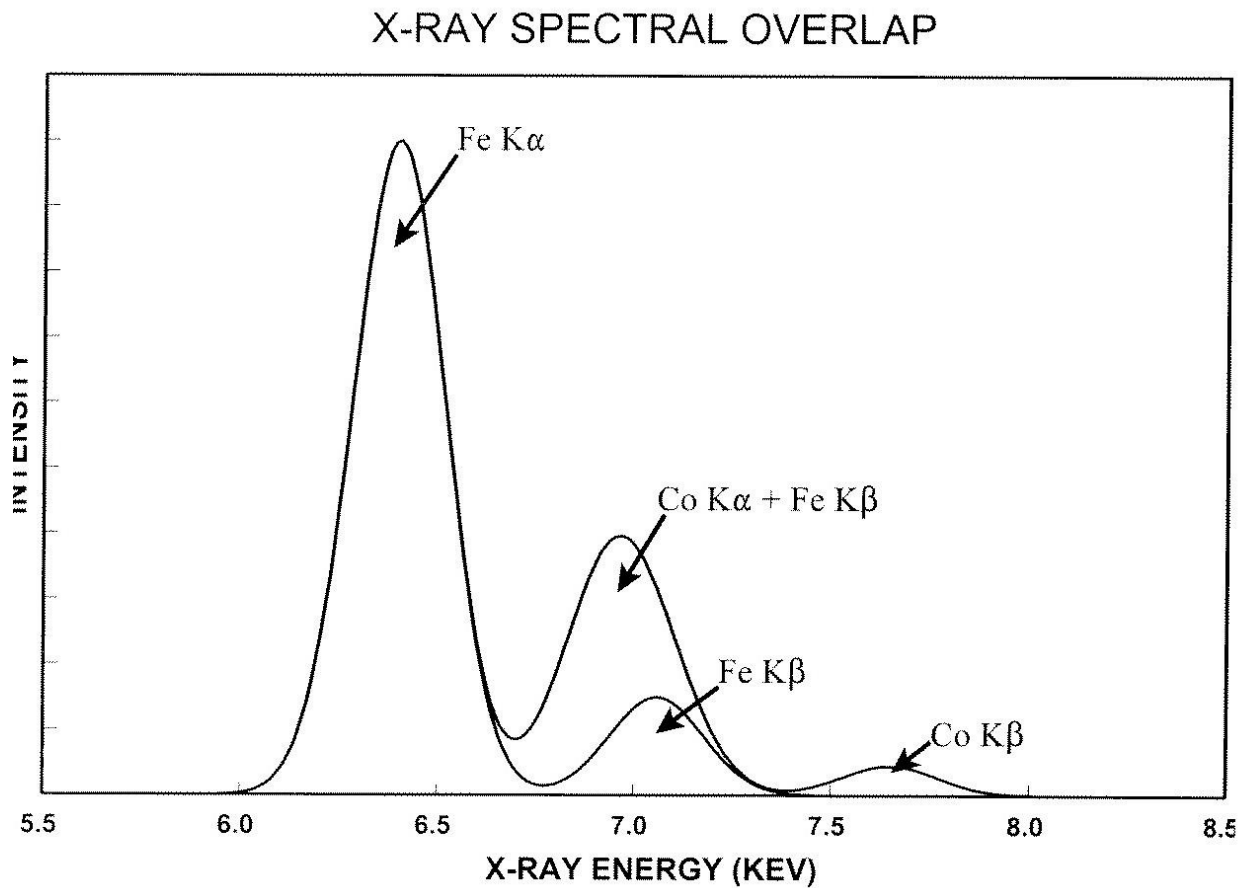


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FIGURE 1. X-Ray Spectral Plot Showing Overlap of Iron  $K_{\beta}$  X-Rays in the Cobalt  $K_{\alpha}$  Measurement Region.





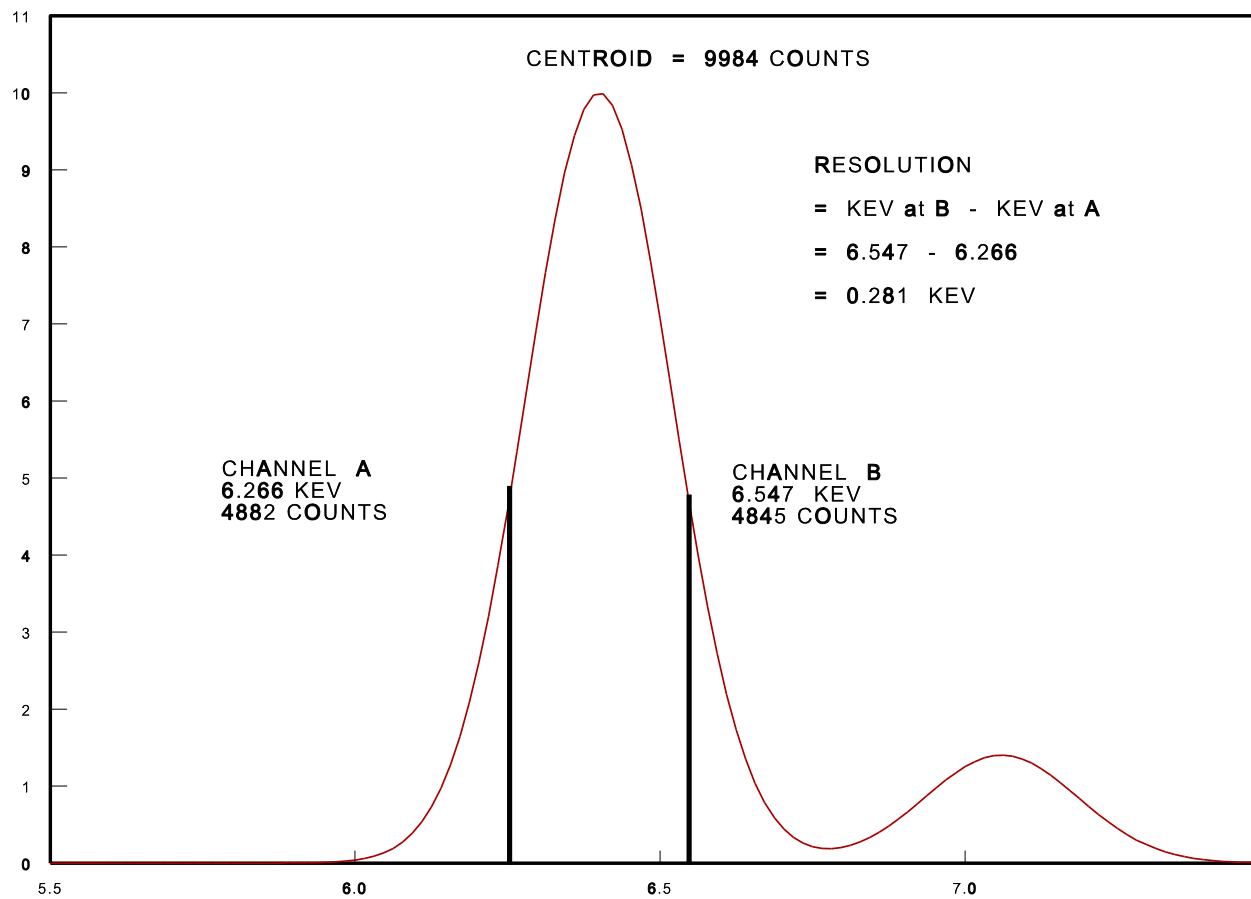


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FIGURE 2. Iron X-Ray Spectrum Illustrating Detector Resolution





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APPENDIX C  
Certified SRM Values and Certificates of Analysis  
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Certified SRM Values\*

Element	#2709 (Low Standard)	#2710 (Medium Standard)	#2711 (High Standard)
Antimony	7.9 ± 0.6	19.4 ± 1.8	38.4 ± 3.0
Arsenic	17.7 ± 0.8	105 ± 8	626.0 ± 38.0
Barium	968 ± 40	726 ± 38	707.0 ± 51.0
Cadmium	0.38 ± 0.01	41.7 ± 0.25	21.8 ± 0.2
Chromium	130 ± 4	NA	NA
Cobalt	13.4 ± 0.7	NA	NA
Copper	34.6 ± 0.7	114 ± 2	2950 ± 130
Lead	18.9 ± 0.5	1162 ± 31	5532 ± 80
Manganese	538 ± 17	638 ± 28	NA
Mercury	1.40 ± 0.08	6.25 ± 0.19	32.6 ± 1.8
Nickel	88 ± 5	20.6 ± 1.1	14.3 ± 1.0
Selenium	1.57 ± 0.08	1.52 ± 0.14	NA
Silver	0.41 ± 0.03	4.63 ± 0.39	35.3 ± 1.5
Strontium	231 ± 2	245.3 ± 0.7	NA
Thallium	0.74 ± 0.05	2.47 ± 0.15	NA
Vanadium	112 ± 5	81.6 ± 2.9	76.6 ± 2.3
Zinc	106 ± 3	350.4 ± 4.8	6952 ± 91

\* - NIST Certificates of Analysis available on NIST web site <https://srms.nist.gov>

NA - Not Applicable, SRM - Standard Reference Material



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APPENDIX D  
Sample Preparation for FPXRF Analysis  
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#### SUMMARY OF SAMPLE PREPARATION FOR FPXRF ANALYSIS

##### A. In-Situ vs Prepared Samples

The choice to use in-situ or prepared samples for Field Portable X-Ray Fluorescence (FPXRF) analysis of metals in soil/sediment depends on FPXRF detection limit capabilities, sample wetness, sample homogeneity, and site data quality objectives. In-situ analysis is faster and, therefore, suitable for screening purposes and to guide excavation activities. If the soil/sediment is wet (greater than 20 percent moisture) or the concentration approaches the FPXRF detection limit (e.g., 3 - 5 times the detection limit), the samples should be prepared prior to analysis. Procedures for in-situ and prepared samples are summarized below.

##### B. In-situ Analysis

Prepare an area (12 x 12 inches) for in-situ analysis:

1. Remove large rocks, vegetation, and debris.
2. Ensure that the soil surface is flat and compact prior to analysis.
3. Place the FPXRF unit firmly in contact with the soil surface.
4. Initiate analysis; DO NOT move the unit during analysis.

NOTE: Avoid analysis of water saturated soils/sediments.

##### C. Prepared Samples

Samples are generally received in labeled plastic bags or glass jars and should be prepared as follows:

1. Thoroughly mix each sample.
2. Remove stones, vegetation, and other debris.
3. Place 10 - 20 grams of sample into a labeled aluminum weighing dish.
4. Dry the samples in a convection oven for 1 - 2 hours or until dry.
5. Allow the sample to cool and pass it through a clean 10-mesh stainless steel sieve.
6. Fill a labeled polyethylene X-ray sample cup to the snap ring with a portion of the sample that passed through the sieve.
7. Seal the XRF cup with 0.2-mil (5 micrometer) thick polypropylene X-ray window film.
8. Pack the sample evenly against the window film by tapping the XRF cup against a tabletop or other clean, flat surface.
9. Place the sample cup in the test stand (window side down), close the safety shield, and initiate the analysis.



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APPENDIX E  
Typical MDL Values for the NITON XL722S FPXRF Analyzer  
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