



Appendix H

Expanded Properties Sampling and Analysis Plan Addendum

Pines Area of Investigation

AOC II

Docket No. V-W-'04-C-784

May 5, 2015

Disclaimer

This document is a draft document prepared under a federal administrative order on consent. This document has not undergone formal review by U.S. Environmental Protection Agency (USEPA). The opinions, findings, and conclusions expressed are those of the author and not necessarily those of USEPA.

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Attachment 2 Operating Procedures

Field-Portable X-ray Fluorescence Analyzer Operating Procedure
Coal Combustion By-Product (CCB) Visual Inspection Procedures

List of Acronyms

bgs	Below Ground Surface
CCB	Coal Combustion By-product
CoC	Chain-of-Custody
EC	Emergency Coordinator
GPS	Global Positioning Satellite
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
ID	Identification
IDW	Investigation Derived Waste
MS/MSD	Matrix Spike/Matrix Spike Duplicate
PPE	Personal Protective Equipment
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SSC	Supplemental Soil Characterization
SSO	Site Safety and Health Officer
USEPA	U. S. Environmental Protection Agency

1 Introduction

This Sampling and Analysis Plan Addendum (SAP) is prepared as a supplement to the Supplemental Soil Characterization (SSC) Work Plan, November 2014. To meet the objectives stated in the SSC Work Plan, data collection tasks included gamma surveys, sampling to confirm the CCB quantification estimates made during the CCB visual inspections, and soil sampling with the latter posited for a statistically-defined subset of nine properties, chosen among a group of 45 properties identified as containing CCB surficial deposits during prior visual inspections. The confirmation sampling for the CCB visual inspections and gamma surveys were completed in June 2014. The soil property sampling was completed in November 2014. Based on the soil sampling results from the initial nine properties, the soil property sampling was expanded to the remaining 37 properties (of the 45) at which CCBs were identified during the visual inspections ("Identified Properties").

The sampling scope and methods presented in this SAP are generally the same as those used with respect to the initial nine properties (Appendix G of the SSC Work Plan, November 2014). This SAP includes procedural modifications, which will be implemented for the Identified Properties. Additionally, this SAP includes procedures for assessing and sampling properties within the Pines Area of Investigation other than the Identified Properties for which owners have requested sampling ("Additional Properties"). The Additional Properties also include 22 properties previously identified through prior visual inspections from public rights-of-way as possibly containing CCB surface deposits, but for which access to conduct on-site investigative work has not previously been granted.

2 Health and Safety

The project Health and Safety Plan (HASP) was updated to include the tasks outlined in the SSC Work Plan. The procedures outlined in the HASP will be followed by AECOM personnel and AECOM's subcontractors during all site work associated with the Identified Properties and the Additional Properties. Matt Laub is AECOM's designated Site Supervisor and Site Safety & Health Officer (SSO). Mr. Laub is also AECOM's designated Emergency Coordinator (EC), in the event of a site emergency. Mr. Laub, or his designee, will be present during all field work to fulfill his health and safety duties.

3 Pre-Field Activities

3.1. Access Agreements and Property Owner Notices

Updated access agreement requests were submitted in March 2015 to the owners of the 37 Identified Properties and 22 Additional Properties for which access was previously denied. Access agreement requests will be submitted to the owners of further Additional Properties, as requests to sample those properties are received (within a six-month period to be agreed upon with USEPA). Owners granting access will be notified of the proposed field sampling schedule via certified mail at least one week prior to the start of field activities.

3.2. Utility Clearance

Utility clearance activities will be conducted on any properties at which soil borings will be sampled to depths greater than 6 inches. Utility clearance activities will be conducted in accordance with AECOM Standard Operating Procedure (SOP) S3NA-417-PR Underground Utilities and Subsurface Installation

Clearance Process (see Appendix C of the SSC Work Plan). Clearance of utilities located in the work area is necessary prior to performing subsurface field activities. Public utilities may include municipal water, electricity, cable television, telephone, gas, and storm sewer. Private utilities may include electricity, propane, and septic. At least two full working days, but no more than 20 calendar days prior to the start of intrusive field work, the field staff will contact Indiana811:

Indiana811

1-800-382-5544

www.indiana811.org

The field staff will provide Indiana811 with the county and the township as well as a street address and cross street of the locations necessary for utility clearance. Where possible, the field staff will mark areas where field work may affect any subsurface utilities with white marking paint or white flags (where paint is not applicable) prior to calling Indiana811. Marking areas with white paint/flags will guide Indiana811 on where to concentrate their locating efforts.

Where subsurface work takes place on private property, a private utility locator will be retained, in addition to the Indiana811 service, to screen the proposed sampling locations for underground utilities. Field staff will work with the property owners to supplement the Indiana811 and private utility locate information, where possible.

Indiana811 underground facility members and the private utility locator will mark or otherwise identify facilities according to the following color codes in accordance with Damage to Underground Facilities, Indiana Code 8-1-26-18:

Utility	Marking Color
Electric power distribution and transmission	Safety Red
Municipal electric systems	Safety Red
Gas distribution and transmission	High Visibility Safety Yellow
Oil distribution and transmission	High Visibility Safety Yellow
Dangerous materials, product lines & steam lines	High Visibility Safety Yellow
Telephone and telegraph systems	Safety Alert Orange
Cable television	Safety Alert Orange
Police and fire communications	Safety Alert Orange
Water systems	Safety Precaution Blue
Sewer systems	Safety Green
Proposed construction	White

Additional information on contacting and providing the necessary information can be found on the Indiana811 website at www.indiana811.org.

4 Sampling Locations

Each Identified Property and Additional Property will be divided into four approximately equally-spaced quadrants (designated A through D). Additional “quadrants” will be added to the initial four quadrants to address three specific property conditions:

- A vegetable or flower garden
- An unpaved driveway where coal combustion by-products (CCBs) are present
- A child's play area, based on the presence of swing sets and/or other outdoor play equipment

For Identified Properties, five approximately equally-spaced sample locations will be identified within each defined quadrant. Preliminary quadrant layouts and sampling locations for each of the 37 Identified Properties are provided in Attachment 1.

For Additional Properties, one centralized boring location will be identified within each defined quadrant.

The quadrant layouts and sampling locations for each property will be verified with USEPA in the field. If any of the specific property conditions identified above are noted at the selected properties, particularly at those properties that were not inspected due to delays in receiving access agreements, additional quadrants will be added, as appropriate. Quadrant boundaries and discreet sampling locations will be logged with a global positioning satellite (GPS) unit.

5 Sampling Methods

5.1. Identified Properties

Samples will be collected using hand-cart mounted direct-push sampling equipment, a track-mounted direct-push sampling rig, and/or by using hand tools such as a hand auger. Direct-push drilling will be the primary sampling method and the hand-cart mounted unit will be used at most properties. The track-mounted rig may be used where property conditions are appropriate (e.g., at unoccupied properties). Hand auguring will be used where sampling locations are inaccessible for the drilling machine or in areas where sampling in close proximity to utilities will be necessary. Borings will be advanced to depths of 5 feet below ground surface (bgs). The sampling and logging procedures will be completed in accordance with SOP 7110Pines Surface Soil and/or SOP 7116Pines Subsurface Soil Sampling by Geoprobe Methods (Appendix C and Section 4.2.2.1 of the SSC Work Plan).

Samples collected at each of the five boring locations within a quadrant will be composited over specific depth intervals, such that one sample from each sampling depth from each quadrant will be obtained for possible laboratory analysis. The sample depths, as defined for the initial nine properties were:

- 0 to 6 inches bgs – referred to as “surface soil.”
- 6 to 18 inches bgs – referred to as “near-surface soil.”
- 18 inches to 60 inches bgs (1.5 feet to 5 feet) – referred to as “subsurface soil.”

Based on observations made at the initial nine properties subject to soil sampling under the SSC Work Plan, CCBs were generally observed to depths of no more than 20 to 30 inches bgs. To provide greater resolution in the vertical characterization of the remaining 37 Identified Properties, the “subsurface soil” interval will be further subdivided, as follows:

- 18 to 36 inches (1.5 to 3 feet)
- 36 to 60 inches (3 to 5 feet)

The 0-to-6-inch and 6-to-18-inch depth intervals from the five sampling locations in each quadrant will be composited for field screening and laboratory analysis. The remaining depth intervals (18 to 36 inches and 36 to 60 inches) from the five sampling locations in each quadrant will be visually characterized. Samples from the 18-to-36-inch interval will be analyzed if:

- CCBs are observed in a portion, but do not extend to the base of the 6-to-18-inch depth interval – the 18-36-inch interval will be composited for field screening and laboratory analysis.
- CCBs are observed in a portion, but do not extend to the base of the 18-to-36-inch depth interval – the portion of the 18-to-36-inch interval without CCBs will be collected and composited for field screening and laboratory analysis.

If CCBs are observed to depths of 36 inches, those materials will be assumed to have the same characteristics as the overlying 6-to-18-inch interval and will not be submitted for field screening or laboratory analysis. Confirmatory sampling from depths greater than 36 inches will be conducted during the removal action, as necessary; and as such, samples from depths greater than 36 inches are not anticipated to require laboratory chemical analysis as part of the work outlined in this plan. However, samples from the 36-to-60-inch interval will be archived and may be retrieved for laboratory chemical analysis, if necessary.

For each depth interval, selected for field screening and laboratory analysis, the discrete samples from each of the five borings within a quadrant will be contained in a resealable, plastic bag or other appropriate container. The sample container will be labeled using permanent ink with the designated sample identification, as outlined in Section 7, below. Each “discrete” sample will be homogenized by manually breaking any soil “clumps” and mixing using plastic utensils or by manually mixing within a resealable, plastic bag or other non-metal container. Then, equal volumes (approximately one oz.) from each of the five discrete samples will be placed into a separate plastic, resealable bag or other non-metal container, the container will be labeled with the designated sample identification, and the sample will be thoroughly composited by manually mixing the combined sample.

The composited samples will then be field screened using a Field Portable X-Ray Fluorescence (FPXRF) analyzer in accordance with the procedure in Attachment 2. The FPXRF screening is being conducted to develop a data population which can be correlated with laboratory analytical results for possible use in developing a correlation basis for use during the removal actions. The FPXRF data will not be used in decision-making associated with the properties sampling work outlined in this SAP. Following FPXRF screening, the entire sample will be transferred to an appropriate, labeled sample container and submitted to the laboratory for chemical analysis.

Photographs will be taken of each quadrant to document conditions before and after the sampling/property restoration. Photographs will be taken of all soil samples as retrieved and also after homogenization in order to create a photographic record of the soil sampling. Additional photographs will be taken throughout the field program as appropriate. Photographs will be digital and the date, direction and subject will be recorded, as applicable. Photographs will be collected as described in Section 4.2.2 of the SSC Work Plan. The field logs will note the depth at which CCBs are no longer observed, or whether they appear to extend beyond the maximum depth of a sample location. Where sufficient accuracy may be achieved by field personnel, a GPS unit will be used to determine the locations of samples collected and quadrant boundaries.

5.2. Additional Properties

Additional Properties that have not previously been subject to on-site visual inspection will be visually inspected, in accordance with the Visual Inspection SOP (Attachment 2). Properties that have had visual inspections will undergo an abbreviated visual inspection, focusing on areas adjacent to rights of way, play areas, gardens, debris piles, and other areas indicated by the property owner. Based on the visual inspection results, samples will be collected for laboratory analysis, as follows:

- Scenario 1: If CCBs are not observed, one surface soil sample (0-6 inch bgs) will be collected from each designated quadrant using a hand auger. Samples will be preferentially collected from accessible, bare-soil areas in each quadrant, if present. The samples from each quadrant will be composited to form one sample per property, and the composite sample will be submitted for laboratory analysis. Additionally, if play areas or gardens are observed at a property, one discrete sample will be collected and submitted for laboratory analysis from each of these areas.
- Scenario 2: If CCBs are only observed along the right-of-way or appear to be on the property solely via tracking from the right-of-way (road), the property will be sampled as described for Scenario 1. Additionally, a "quadrant" will be established to encompass the area in which tracked/right-of-way CCBs are observed. In this quadrant, one sample will be collected from the 0-6 inch depth interval within the observed CCBs area and the sample will be submitted to laboratory analysis.
- Scenario 3: If CCBs are observed, and do not appear to be limited to the right-of-way or to have been solely tracked onto the property from the right-of-way, the Additional Property will be re-designated as an Identified Property and the sampling methodologies described in Section 5.1, above, will be applied.

Photographs will be taken of each quadrant to document conditions before and after the sampling/property restoration. Photographs will be taken of all soil samples as retrieved and also after homogenization in order to create a photographic record of the soil sampling. Additional photographs will be taken throughout the field program as appropriate. Photographs will be digital and the date, direction and subject will be recorded, as applicable. Photographs will be collected as described in Section 4.2.2 of the SSC Work Plan. The field logs will note the depth at which CCBs are no longer observed, or whether they appear to extend beyond the maximum depth of a sample location. Where sufficient accuracy may be achieved by field personnel, a GPS unit will be used to determine the locations of samples collected and quadrant boundaries.

6 Quality Control Samples

6.1. Identified Properties

Quality control samples will be collected and analyzed as specified in the QAPP Addendum, which is provided as Appendix B of the SSC Work Plan. Field duplicate and matrix spike/matrix spike duplicates (MS/MSDs) samples will be analyzed to assess the quality of data resulting from these sampling activities. For completeness and ease of implementation, quality control samples will be collected on a per-property basis, rather than on a total-sample-number basis, for the Identified Properties. It is estimated that 8 to 15 samples will be collected from most properties.

Field duplicates will be collected by alternately dividing portions of the sample material between two identical sets of containers for laboratory analysis. The samples will be labeled as two separate samples and carried through analysis and reporting. The field duplicate will be analyzed for the same parameters as its associated field sample. Field duplicate samples will be collected at a frequency of one per property. As described above, this equates to a duplicate sampling frequency of one duplicate per 8 to 15 field samples.

MS/MSD samples will be collected in the same manner as the original samples, with double sample volume (or the amount requested by the laboratory) collected to provide sufficient material for the analysis. Field duplicate samples will be collected at a frequency of one per property. As described above, this equates to a duplicate sampling frequency of one duplicate per 8 to 15 field samples.

Equipment rinsate blanks will be collected by pouring laboratory-grade, organic free water (provided by the laboratory) through non-dedicated sampling equipment (soil probe rod and/or hand-auger) and collecting the rinsate in the appropriate sample containers. Equipment rinsate blanks will be collected at a frequency of one per property per equipment used to sample the property. Equipment rinsate blanks will be collected after equipment decontamination and upon completion of sampling at each property, ensuring that equipment rinsate blanks are collected to represent each collection method (soil probe and/or hand auger) used at each property.

Additionally, replicate samples will be collected at 15% of the quadrants (1 per every 6 quadrants sampled). Replicate (triplicate) samples will be collected to develop estimates of variability associated with the sampling strategy. Replicate sampling will be accomplished by drilling two separate borings approximately one meter from each of the original boring locations in two cardinal directions. Samples will be collected, handled, and analyzed in the same manner as the original samples. The statistical variability among the results of the original and replicate samples will be evaluated to establish confidence limits associated with the composite sample data.

6.2. Additional Properties

Quality control samples for the Additional Properties will be collected at a rate of one per 20 samples analyzed or one per sampling week, whichever occurs first. Quality control samples will consist of field duplicates, MS/MSD samples, and equipment rinsate blanks.

7 Sample Identification

Immediately upon collection for FPXRF field screening and laboratory analysis, an adhesive sample label will be affixed to each container, including the unique sample identification (ID), as described below, the time and date of sample collection, the sampler's initials, parameters to be analyzed, and preservation, if applicable. The project name will not be shown on the label. The unique sample identification will be an alphanumeric code consisting of the elements described in the following sections.

7.1. Identified Properties

The unique sample identification will consist of following elements:

- Name of the property in five characters using the same IDs assigned during the gamma surveys (e.g., GS010). These location names will correspond to field logs, as well as sample locations posted on maps.
- Two letters signifying the quadrant identification. QA, QB, QC, QD for the minimum four quadrants. QG will signify garden quadrants, QP for play area quadrants, QZ for rights-of-way or driveways, and QX for all other locations, should additional locations be identified in conjunction with USEPA based on in-field observations. Location designations will be recorded in the field notes.
- Two letters signifying the sample depth interval:
 - SS for surface soil – 0 to 6-inch depth,
 - NS for near surface soil – 6 to 18-inch depth,
 - SB for subsurface soil – 18 to 36-inch depth, and
 - SC for subsurface soil – 36 to 60-inch depth.
- Sampling date consisting of the number corresponding to the month (2 digits), day (2 digits) and year (2 digits), for example, 111214 for samples collected on November 12, 2014.
- One letter denoting the type of sample. Codes for this field include: S – sample; D – field duplicate; B – rinsate blank; X – replicate offset 1; and Z – replicate offset 2.

No dashes will be used to separate fields. An example sample ID would be: GS010QCNS050115D, indicating a soil sample collected at Property GS010, quadrant C, at the near-surface depth interval, on May 1, 2015. The actual depth intervals will be recorded on field boring log forms. This example indicates the sample is a field duplicate as indicated by the “D” at the end of the sample ID.

Rinsate blank sample names will include the property ID, sampling date, B to indicate a blank sample, and number to indicate which equipment was rinsed to generate the blank sample, where 1 indicates the soil probe rod and 2 indicates the hand auger. For example: GS012050115B1 would indicate a rinsate blank sample collected following sampling at Property GS012 on May 1, 2015, where the sample was collected by rinsing the soil probe rod.

MS/MSDs will have the same name as the original field sample and “MS/MSD” will be noted in the comments field of the chain-of-custody (CoC) form.

The sample ID code will be recorded on the label, in the field logbook/field forms, and on the CoC form. The sample ID will be carried through the analytical process to reporting. Sample chain-of-custody procedures and packaging and shipment of samples will be completed in accordance with SOP 1007Pines Chain-of-Custody Procedures and SOP 7510Pines Packaging and Shipment of Environmental Samples (Appendix C of the SSC Work Plan).

7.2. Additional Properties

The unique sample identification will consist of the same elements as identified for the Identified Properties, with the following differences:

- Properties for which no GS ID number was previously assigned (e.g., during the prior visual inspections) will be assigned a new GS ID number will be assigned, beginning with GS100..

- No quadrant designation will be used.

An example sample ID would be: GS100SS050115S, indicating a soil sample collected at Additional Property 001, at the surface soil depth interval, on May 1, 2015. Quality control samples (blanks and MS/MSDs) will follow the same nomenclature as indicated for the Identified Properties. The sample ID code will be recorded on the label, in the field logbook/field forms, and on the CoC form. The sample ID will be carried through the analytical process to reporting. Sample chain-of-custody procedures and packaging and shipment of samples will be completed in accordance with SOP 1007Pines Chain-of-Custody Procedures and SOP 7510Pines Packaging and Shipment of Environmental Samples (Appendix C of the SSC Work Plan).

8 Sample Custody

The field sampler(s) are responsible for the care and custody of the samples, including shipping to the laboratory. Field samplers will complete the CoC in accordance with AECOM SOP No. 1007Pines – Chain-of-Custody Procedures (Appendix C of the SSC Work Plan).

9 Sample Packaging and Shipping

Samples will be packaged for shipment to the laboratory under the CoC procedures described in AECOM SOP No. 1007Pines – Chain-of-Custody Procedures and AECOM SOP No. 7510Pines – Packaging and Shipment of Environmental Samples (Appendix C of the SSC Work Plan).

After sample containers are labeled and filled, samples may be placed in plastic zipper-lock bags to contain material in the event of container spillage during shipment. Containers will then be packaged in a cooler for shipment, using inert packing material (e.g., bubble wrap, rubber foam, or equivalent) to prevent breakage during shipment. The cooler will be packed with sufficient ice to maintain a temperature of $4^{\circ} \pm 2^{\circ}$ Celsius (C) for overnight delivery. A multi-form CoC form will be completed. An example CoC form is presented in AECOM SOP No. 1007Pines – Chain-of-Custody Procedures (see Appendix C of the SSC Work Plan). The original CoC will be placed in a zipper-lock bag that is taped to the lid inside each cooler of samples being submitted to the laboratory for analyses. The back copy of the CoC will be maintained with the field records. The cooler will be locked or sealed, and custody seals placed on the outside of the cooler in such a way that the cooler cannot be opened without breaking the seals.

Sampling, analytical holding times, and shipping and receiving of samples will require close attention and coordination between field staff and laboratory staff. During the time period between collection and shipment, samples will be stored in ice-filled coolers or refrigerated, if applicable, and maintained under sample custody. Samples will be shipped to the laboratory via commercial overnight courier (e.g., Federal Express).

10 Decontamination of Non-Dedicated Sampling Equipment

Decontamination of non-dedicated sampling equipment will be completed in accordance with SOP 7600Pines Decontamination of Field Equipment (Appendix C of the SSC Work Plan). All non-dedicated sampling equipment will be decontaminated prior to sampling. Decontamination of drilling/sampling equipment will take place between each quadrant. Dedicated (i.e., disposable)

sampling equipment will not be decontaminated. In general, equipment used will be decontaminated using the following procedure:

- Tap water rinse to remove gross contamination;
- Non-phosphate, non-borate detergent (e.g., DETERGENT8®) and water rinse;
- Tap water rinse;
- Distilled/deionized water rinse;
- 10% nitric acid rinse;
- Distilled/deionized water rinse; and
- Air dry.

If the decontaminated equipment will be stored until later use, it will be wrapped in aluminum foil to prevent recontamination. Water generated during decontamination of sampling equipment will be handled as described in Section 11, below.

11 Investigation Derived Waste (IDW) Management

Investigation-derived waste (IDW) for the field investigation program will be managed in accordance with applicable USEPA and IDEM regulations. It is anticipated that IDW materials will be generated during the field investigation. These materials may include:

- Decontamination fluids,
- Used PPE,
- Used sampling equipment, and
- Excess soil from soil borings.

These wastes will be handled in the following manner:

- Water used for decontamination will be disposed of at Yard 520.
- Excess soil from soil borings will be returned to respective boreholes within each quadrant as backfill.
- Used PPE, such as sampling gloves, paper towels, or other materials will be bagged and sealed prior to disposal as general refuse.
- Used disposable sampling equipment, such as trowels and empty bottles, will be disposed of with the PPE as general refuse.

12 Property Restoration

Property restoration will be performed upon completion of sampling activities. Boreholes will be backfilled with granular or chipped bentonite and the borehole will be topped with site soil or other material consistent with the surrounding ground surface. The ground surface will be smoothed of ruts and/or divots and returned to pre-sampling condition to the extent practical. If necessary, a seed mix

will be applied to areas where grass has been removed. Photographs will be taken of each quadrant to document conditions before and after the sampling/property restoration.

13 Laboratory Analysis

Soil samples will be analyzed for arsenic, thallium, and lead. Arsenic and thallium are selected as the indicator parameters, based on the results from the initial nine properties sampled. Lead is being analyzed solely for use in evaluating the accuracy with which the FPXRF programming resolves the arsenic-lead emission interference (see Attachment 2 for further discussion).

The samples will be submitted to the ALS Group (formerly Columbia Analytical Services, Inc.) for the arsenic, thallium, and lead analyses. Additional details on the analytical methods are provided in the project QAPP Addendum, which is provided as Appendix B of the SSC Work Plan.

14 Data Evaluation

Laboratory analytical data will be reviewed and validated in accordance with the project QAPP Addendum (Appendix B of the SSC Work Plan). The results will be provided to USEPA in a report, as outlined in the SSC Work Plan.

Attachment 1

Identified Properties

Maps with Preliminary Quadrants and Boring Locations

Property GS011 Quadrants and Preliminary Soil Sampling Locations
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[Submitted separately due to privacy concerns]

Attachment 2

Supplemental Operating Procedures

Field-Portable X-ray Fluorescence Analyzer Operating Procedure
Coal Combustion By-Product (CCB) Visual Inspection Procedures

HALEY & ALDRICH, INC. OPERATING PROCEDURE #3020

Field Screening for Metal Constituents using a Field Portable X-Ray Fluorescence (FPXRF) Analyzer

LEVEL II

PREPARATION AND APPROVALS

VERSION	AUTHORED/DATE	REVIEWED / DATE	REVIEWED / DATE	REVIEWED / DATE	APPROVED / DATE
1	D. Conley 31 March 2015				

Total Pages: 5

Privileged and Confidential Prepared at the Request of Counsel
DRAFT

Field Screening for Metal Constituents Using a Field Portable XRF (OP#3020)

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OPERATING PROCEDURE #3020

Field Screening for Metals Constituents using a Field Portable X-Ray Fluorescence (XRF) Analyzer

1. PURPOSE

This operating procedure (OP) presents the procedure for the use of a Field Portable X-ray Fluorescence (FPXRF) analyzer for the field screening of samples for the concentration of metal constituents. This screening procedure has been prepared with guidance provided by USEPA Method 6200- *Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment (Rev. 0 February 2007)*. This OP is designed to be a guideline to be used in conjunction with the instrument manufacturer's user manual for the operation of the instrument to support the development of a basis for correlation of the FPXRF readings obtained in the field to analytical laboratory results obtained using EPA promulgated methods (for example EPA 6020). The resulting correlation information can be used to support further investigation and remedial actions.

Prior to the operation of any FPXRF, all operators must receive instrument specific training in the safe handling and operation of this radiation generating device. In addition to the instrument specific training, the operator must be enrolled in a company sponsored Medical Monitoring Program, successfully completed an approved OSHA 40-hour HAZWOPER training program and 8-hour Refresher training as specified in the project health and safety plan.

During the operation of the FPXRF, all operators must wear a radiation dosimeter badge assigned for their sole use. Radiation dosimeter badges must be evaluated in accordance with applicable employer and regulatory requirements.

2. INSTRUMENT & SUPPLIES

2.1 Instrument Information

This procedure has been developed for use with an InnovX Systems Delta Model DP-4000 Field Portable X-Ray Fluorescence (FPXRF) Analyzer (DP-4000) or similar FPXRF that is a portable energy dispersive x-ray fluorescence spectrometer generating x-rays using an excited tube technology.

The DP-4000 is equipped with a broad spectrum detector system that can determine the concentration of numerous elemental constituents from trace levels (less than 10 parts per million) to percent (>10,000 ppm) concentrations in solid matrices such as soils, sediments and manufactured components.

2.2 Equipment and Supplies

Equipment and supplies typically required to complete the tasks described in this Operating Procedure:

1. DP-4000 FPXRF Analyzer or equivalent.

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Field Screening for Metal Constituents Using a Field Portable XRF (OP#3020)

2. Portable Case with Back-up Battery and charging station
3. Alloy 316 Internal Calibration Coupon and Silica Blank
4. Pre-Cleaned 4.0 Ounce (Oz.) Glass Sample Jars with Teflon Liners
5. Plastic Bowl and disposable mixing spoons
6. Re-sealable Plastic Bags (1 Quart)
7. NIST Standard Reference Materials (SRM) Applicable Matrix/Analyte Concentrations
8. Paper Towels
9. Distilled Water
10. Replacement Mylar Windows
11. Field Logbook/Pre-printed Forms
12. Permanent Markers/Ball Point Pens
13. Calculator/Portable Laptop Computer

3. PROCEDURE

3.1 Sample Collection and Preparation

Samples collected to be representative of a depth interval (for example: 0-6 inches) will be transferred to a plastic bowl or a re-sealable plastic bag and homogenized using plastic utensils or manually mixing in the plastic bag. The sample will be transferred to and completely fill a pre-cleaned 4.0 ounce sample jar for analysis at an appropriate off-site laboratory. The sample jar will be labeled using a permanent marker (e.g. Sharpie) with the designated sample location and depth interval, sampling date/time, and sampler's identification. The naming convention for each sample location is provided in Section 7.1 of Appendix H Expanded Properties Sampling and Analysis Plan Addendum, Pines Area of Investigation, AOC II (AECOM 2015).

3.2 Instrument Calibration

The DP-4000 electronic internal calibration is performed using a clean silica blank sample and a standardization coupon comprised of Alloy 316 Stainless Steel provided by the manufacturer. The internal calibration must be performed at least once per day prior to instrument operation, and after every 10 hours of continuous operation. The procedure for electronic internal calibration of the DP-4000 is provided by Chapter C.4 Operations of the User Manual provided in Appendix B.

Prior to the analysis of samples, the internal instrument calibration should be confirmed using a certified standard reference material (SRM) provided by the National Institute of Standards and Technology (NIST) or certified vendor traceable to an NIST SRM. The SRM should be comprised of a similar matrix to be screened by the instrument (i.e. filter media or soil) with the metal constituents at known concentrations that are anticipated in the field samples/media.

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Field Screening for Metal Constituents Using a Field Portable XRF (OP#3020)

3.3 Sample Analysis

Testing Area Set-up

The testing area should be segregated from the general work area and should include proper signage and demarcation at a distance of at least 10 feet indicating that a radiation generating instrument is in use in the testing area. The instrument is equipped with a trigger system which turns off the excitation tube when not in operation. However, the instrument should never be pointed at any body part at any time for any reason. As such, samples to be screened should be placed away from the operator and the instrument should always be pointed away from the operator.

NOTE: NEVER HOLD SAMPLES IN YOUR HAND DURING SCREENING USING THE DP-4000 OR ANY FPXRF INSTRUMENT.

Sample Screening

Use the following steps to screen the pre-collected sample for metal constituents using the DP-4000 or similar FPXRF:

1. Remove the cover of the sample container and transfer the entire homogenized sample to a re-sealable plastic bag (<1.0 mil thick). (NOTE: DO NOT USE FREEZER BAGS)
2. Spread the sample within the bag to an even thickness of approximately 0.5 inches.
3. Place the DP-4000 analyzer sample window directly onto the plastic bag and hold in contact with the sample. (NOTE: Use of the Innov-X Systems work station or similar is recommended to hold the instrument securely in place.)
4. Engage the instrument trigger to open the sample window and expose the sample to the generated x-ray for a period of 30 seconds. (Note: sample acquisition time for samples and standards must be the same.)
5. The concentration of metal constituents detected by the DP-4000 or similar FPXRF will be stored to the instrument datalogger and identified with a unique reading number. The results will also be displayed on the instrument liquid crystal display (LCD) and should be recorded manually using a ball point pen in a dedicated Field Logbook or field form or entered into a laptop computer or tablet.
6. Replace instrument onto another location on the plastic bag and repeat screening until 5 readings are obtained for each sample.
7. Remove DP-4000 or similar FPXRF instrument and place in secure docking station.
8. Transfer the sample back to the sample container and re-seal the sample with the lined cover.
9. Discard the plastic bag to an appropriate container for proper disposal.

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10. After the screening of the last sample for the analytical batch, connect the DP-4000 or similar FPXRF to a laptop computer using the USB cord provided by the manufacturer and download the acquired results in a comma delimited spreadsheet format (.csv file) for evaluation and presentation purposes.

Quality Assurance and Quality Control

Quality control (QC) procedures should include:

1. Use of the clean silica blank and standardization coupon to perform internal instrument calibration every 10 hours of operation or per day.
2. Analysis of certified soil standards provided by the National Institute of Standards and Testing (NIST) before and after the analysis of a batch of field samples and,
3. Analysis of duplicate SRM samples to evaluate the reproducibility of the screening procedure. (NOTE: Frequency of duplicate SRM analysis should be consistent with the requirements of the project specific quality assurance project plan (QAPP)).

Precision will be measured in terms of the replicate percent difference (RPD) defined as the absolute value of the difference between two measurements divided by the mean of the two values multiplied by 100 (%) as presented by the following equation:

$$RPD = \frac{\left| \frac{R1 - R2}{(R1 + R2)} \right|}{2} \times 100\%$$

Acceptance criteria for duplicate analyses performed on solid matrices using the FPXRF should be less than 50% RPD for metal constituents present at 5 times (5x) the detection limit (DL) for the constituent and 100% for constituents at a concentration less than 5x the DL.

Accuracy is determined in terms of the percent difference (%D) of an analyte concentration detected by the DP-4000 or equivalent instrument from the known concentration within the SRM provided by NIST or equivalent source.

$$\% \text{ Difference} = \frac{\text{SRM Sample Measured} - \text{Known Value of SRM}}{\text{Known Value of SRM}} \times 100\%$$

The acceptance criteria for an SRM with a known concentration greater than 5x the DL should be +/- 10% of true value and +/- 30% for known concentrations less than 5x the DL.

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Instrument Detection Limit

The instrument detection limits for metal constituents using FPXRF analysis vary dependent upon the characteristics of the element. EPA Method 6200 presented in Appendix C provides the anticipated detection limits for some typical metal constituents as follows:

<u>Analyte</u>	<u>Lower Limit of Detection (mg/Kg)</u>
Antimony	40
Arsenic	40
Barium	20
Cadmium	100
Chromium	150
Cobalt	60
Lead	20
Nickel	50
Selenium	40
Thallium	20
Vanadium	50
Zinc	50

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APPENDIX A

REFERENCES

1. Method 6200 Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, SW-846, US EPA, February 2007, Rev. 0.
2. Delta Family Handheld XRF Analyzers: User Manual, InnovX Systems, PN 103201, Rev. A June 2010.
3. Method 6020A Inductively Coupled Plasma, SW-846, USEPA, February 2007, Rev. 1.
4. Sample Handling Strategies for Accurate Lead-in-Soil Measurements in the Field and Laboratory, NITON Corporation, Stephen Shefsky, Proceedings of the International Symposium of Field Screening Methods, Las Vegas NV, January 1997.

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APPENDIX B

InnovX Systems – Delta Family: Handheld XRF Analyzers – User Manual

User Manual

Delta™ Family: Handheld XRF Analyzers

PREMIUM



STANDARD



CLASSIC



General

This Manual is solely the property of Innov-X Systems, Inc. and, along with the Delta™ XRF analyzer to which it applies, is provided for the exclusive use of Innov-X clients under contractual agreement for Innov-X test and analysis equipment and services.

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Innov-X Systems, Inc. reserves the right to make changes in the product design without reservation and without notification to its users.

Revision History

Release Date for this document and its individual sections is June, 2010. This enters the Innov-X document control system as Revision A

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Delta Family End User Documentation Resources

During Delta's development and initial product shipments several End User documents have been created. They are listed in the table below.

<i>Delta Documentation Resources</i>		
<i>Innovx</i>	<i>Release</i>	
<i>Part #</i>	<i>Date</i>	<i>-----Title-----</i>
103202_RevA	July/2010	Delta User Interface Guide (UI version 2.5))
103201_RevR	June/2010	Delta Family User Manual (This document)
103076_RevA	3/2010	Delta Family Quick Start
101593_RevA	11/2007	Window Replacement: Hinged Plate HandHeld Analyzers
102922_RevA	2/2010	Delta Family User Manual (Canadian Edition)
103158_RevA	3/2010	HOW TO: Setup and Configure A-020-D Teststand/Workstation for Delta Analyzer
TBA	6/2010	HOW TO: Convert A-020-A or A-020-O Teststand/Workstation to Support a Delta Analyzer

Preface

This *Preface* provides the following information:

- “Manual Structure”
- “Document Conventions”

Manual Structure

This *User Manual* consists of eight chapters, ten appendices, this Preface, Table of Contents, and a Cover. Individual chapter material is summarized below:

C1. Introduction describes the basics of the system:

- The Innov-X Delta™ Family
- Visual tour of the instrument noting all the major components

C2. Safety Information describes general safety information:

- Priority Information
- General Precautions
- Electrical Precautions
- X-ray Safety
 - Safety Interlock Structure
 - Safe and Unsafe Usage Scenarios
 - Radiation Doses for Several Scenarios
 - Comparative Analysis of Typical Exposure
 - Common Questions and Answers
- Delta Radiation Profile
- Required Certification
- Analyzer Shut-down Procedures

C3. Safety Administration describes safety program information:

- Radiation Safety Training Recommendations
- Dosimeter badges
- A typical dosimeter monitoring program
- Dosimeter service contractors
- Registration requirements

C4. Operations describes operations and testing procedures:

- Configure and Use Delta Docking Station (DDS)
- Start-up Procedure
- Cal Check Issues
- Battery Issues
- Conducting and Ending Test Operations



NOTE

Information concerning the Delta Family's user interface is supplied in the companion document “*Delta User Interface Guide*” (PN103202_Rev2.5 June/2010). The goal is to provide revised Delta UI Guides when a substantial software change is released.

C5. Alloy Analysis Modes describes five specific modes and calibrations including:

- | | |
|--------------|----------------------------|
| — Alloy | — Pass/Fail |
| — Alloy Plus | — Precious Metal Additions |
| — FastID | |

C6. Mining Analysis Modes describes three specific modes and calibrations including:

- | | |
|-------------------|----------------|
| — Mining | — Car Catalyst |
| — Two Beam Mining | |

C7. Soil Analysis Modes describes two specific modes and two calibrations including:

- | | |
|-------------------|-----------------|
| — Soil | — Environmental |
| — Three Beam Soil | — Exploration |

- LEAP issues for Classic Delta (PIN detector)
- Check Standards
- Sample Preparation

C8. Consumer Goods Modes describes two specific modes including:

- | | |
|--------|---------------------|
| — RoHS | — Consumer Products |
|--------|---------------------|

- **RoHS Mode** provides a details from EU regulation directives which list the limits for RoHS elements and information for qualitative measurements.
- **Consumer Products Mode** is dedicated to testing for Lead (Pb)

A1. Overview: X-Ray Fluorescence (XRF) Spectrometry presents background information and general knowledge, including:

- Basic Theory and X-ray History
- Elemental Analysis
- EDXRF Spectrometers

A2. Soil Testing presents information on using the analyzer for soil analysis within certain accepted guidelines, including:

- Status for Field Portable XRF and Overview of Field Usage
- Quality Assurance
- Calibration for Innov-X Portable XRF
- Effects of Moisture on XRF Results
- Comparing XRF Results to Laboratory Results
- Common Interferences
- Sample Prep Procedures and Testing Protocols

A3. Specifications presents analyzer hardware and software specifications.

A4. Typical Delta Test Sequence

- Prerequisites noted by Mode
 - Grade Libraries
 - Check Standards
 - Sample Presentations
- Typical Test Sequence

A5. User Maintenance provides a key procedure/technique:

- Using the AC Power Adapter kit to replace a Li-ion battery
- Using the stand-alone battery charger
- Replacing a Prolene, Mylar, or Kapton Window

A6. Packing and Shipping gives the procedure for returning a unit to Innov-x.

- Warning Label for shipping products with Li-ion batteries

A7. Legal Information presents material, including:

- Analyzer Limited Warranty including:
 - Limitation of Liability
 - Warranty Period, Returns, and Repairs
 - Instructions for Contacting Innov-X
- End User Software License Agreement including:
 - Use, Restrictions, and Termination of Software
 - Liability Limitations

A8. Alloy Grade Libraries including the Alloy Factory Grade library for each Model, and a “Tramp” Library with seven base alloys.





Document Conventions

Messages

There are four messages used in this Manual:

WARNING, **CAUTION**, **NOTE**, and **GOTO**.

They are characterized by an icon and a message box topped with a colored banner. The message text is on a gray background. An example of each message is below:

	WARNING DEMANDS that you observe the actions given in the text. The WARNING message has a bold type style. <i>Remember:</i> The WARNING icon signifies information that denotes a potentially hazardous situation, which if not avoided, may result in serious injury or death.
	CAUTION SUGGESTS that you review the referenced details and heed the instructions offered. The CAUTION message has a regular type style with <i>emphasized keywords</i> .
	NOTE REQUESTS that you pay particular attention to a specified procedure or piece of information. Adds details that make it easier to use the system and this manual. The NOTE message has a regular type style.
	GO TO DIRECTS the user to another portion of this manual, or to other reference materials containing relevant data. The GOTO (or Pointer) message has a regular type style.

Type Styles

These conventions are used to present information:

Convention (Type Style)	Description
Bold	Indicates an action taken on a button or other item.
<i>Italic</i>	Menu commands, names of keys, buttons, tabs, or items from picklists. User-entered text. It is used for references to other documents, C(hapter) titles, and A(ppendix) titles (for example, "... see " C2. Safety Information "). Labels on unit's I/O panels; panel or window names of the UI (User Interface).
Courier type-face	Computer displayed text or filename.

Pagination

Page numbering in this Manual is consecutive with the Front Cover being assigned Page Number 1. This enables the PDF document file and any hard-copy print to map to the Page field information in the Adobe Reader.

C1. Introduction

C1 includes a:

- Description of the Innov-X *Delta*™ family of handheld XRF analyzers.
- Visual tour of the instrument(s) noting all the major features, and accessories.

Description of *Delta* System

What Is It?

The *Delta* is a handheld energy dispersive X-Ray fluorescence spectrometer, generally referred to as an XRF analyzer. A complete *Delta* package consists of:

- Handheld analyzer using an integrated group of instrument components that are sealed in an ergonomically designed, light-weight body. They include --
 - Controller
 - Color touchscreen (ergonomically mounted interactive display)
 - Membrane navigation keys
 - Choice of detectors (**PiN** or **SDD**) to meet wide-ranging application goals

Coordinated with these robust characteristics, the instrument's *key feature* is Innov-X's proprietary control, data acquisition, and analysis software with customer configured options.

Additional accessories (standard and optional) include:

- Li-Ion batteries (2) - {Standard}
- Delta Docking Station (DDS) Dedicated charging and calibration unit - {Standard}
- Rugged waterproof carry case - {Standard}
- Portable test stand to create a Delta workstation (A-020-D) {Optional}
- Soil foot (A-035) (Optional)
- Soil extension pole (990055) (Optional)
- Trimble Xplorer Package (Optional)

What Does It Do?

The expanded *Delta* family of handheld XRF instruments delivers fast and precise identification and analysis for elements from magnesium to uranium (Mg to U) depending on the selected model. A weatherproof/dustproof ultra rugged design including an **integral heat sink** permits users to conduct diverse analysis testing under severe operating conditions. An added convenience feature for field use is battery **"Hot Swapping."**

Applications

The analyzer gives accurate chemical analysis for commercial or industrial areas, such as:

- | | |
|------------------------------------|-------------------------------------|
| • Positive Material Identification | • Scrap Processing |
| • Mining and Exploration | • Environmental Testing |
| • Consumer Safety | • Light Element & Aluminum Analysis |

Delta Family: Types, Models, Modes and Calibrations



Delta Types and Models

Type	Modes	Models
Premium	Alloy	DP-2000
	Environmental	DP-4000
	Mining	DP-6000
	RoHS	DP-6500
Standard	Alloy	DS-2000
	Environmental	DS-4000
	Mining	DS-6000
	RoHS	DS-6500
Classic	Alloy	DC-2000
	Environmental	DC-4000
	Mining	DC-6000
	RoHS	DC-6500

Modes and Calibrations

ALLOY Analysis	Alloy	MINING	Mining Mode	LEAD PAINT	Lead in Paint (HUD)
	Alloy Plus		2 Beam Mining		Lead in Paint (Industrial)
	FastID & Pass/Fail		Car Catalyst		
	Precious Metals				
SOIL Analysis	Environmental	CONSUMER GOODS	RoHS	THIN	Filter Analysis
	Exploration		Consumer Products		Dust Wipe

Inspection

Inspection

Use this procedure:

1. Remove the carry case from the shipping cartons; save cartons.
2. Open the carry case
Remove the shipping documentation
3. Verify that all the parts and accessories are included.
Remember that the case has **TWO FOAM LAYERS**.
4. Verify that no visible damage occurred during shipping.



WARNING

If there is damage to any of the components, DO NOT attempt to use the instrument.

Immediately contact Innov-X Customer Support at:

- United States: 1-781-938-5005
- Europe: +31 (0)73 62 72 590
- Canada: 1-778-960-6279
- Australia: 02-9577-9500

Or call your local distributor.



GO TO

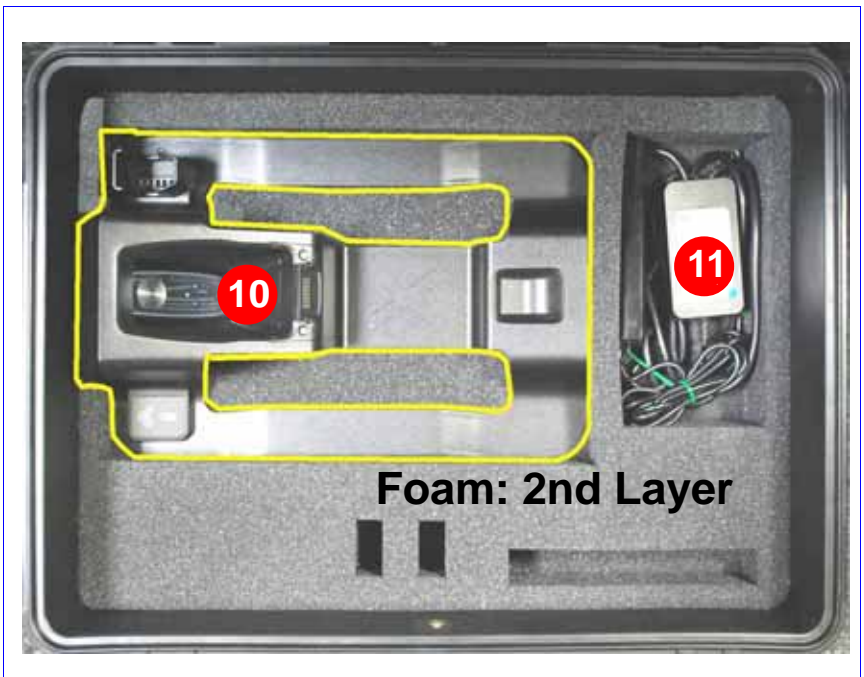
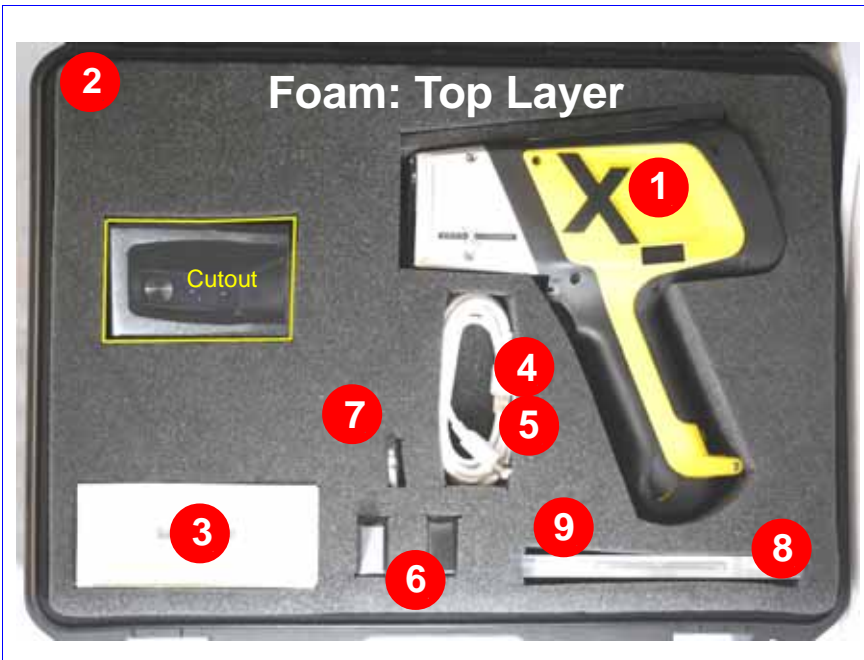
- See [C4. Operations, Page 45 & 50 Battery Issues](#) for battery charging information.
- See "[Delta User Interface Guide](#)" (PN 103202) for a complete description of the *Innov-X* application's User Interface.
- See [A7. Legal Information](#) for warranty, liability, and software licensing information.

Tour of the *Delta* Package

The figure below depicts *Delta's* major components as initially shipped to a customer. Note that the protective foam in the Carry Case has **TWO LAYERS**.

Component Key

—Foam: Top Layer—	
1	Delta Analyzer
2	Carry Case
3	Docking Station Charger
4	USB Cable #1
5	USB Cable #2
6	Li-ion Batteries (2)
7	Cal Check Coupon
8	Extra Windows (Bag of 10)
9	End/User Documentation
—Foam: 2nd Layer—	
10	Docking Station
11	AC Power Adapter (<u>Optional</u>)



Tour of Instrument

1. Handheld Analyzer

Component Key

Delta - All Models

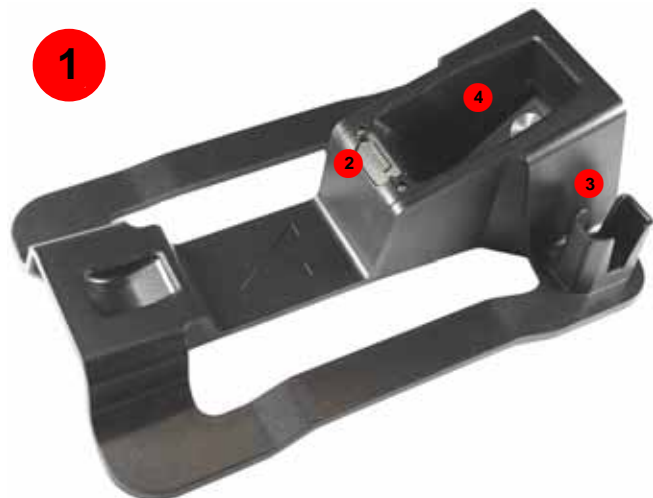
1	Delta Analyzer (Premium Model Shown)
2	Probe
3	Measurement Window (Prolene Film)
4	Hinged Window Plate
5	Docking Station Connector
6	Trigger
7	Handle - Non-Slip Rubber Grip
8	Battery Boot
9	Data Port w/ Rubber Cover
10	Heat Sink
11	I/O (Power) Switch w/ LED Indicator
12	X-ray Warning Light Array
13	Touchscreen for User Interface
14	Navigation Buttons



11 I/O Power Switch w/LED Indicator

12 X-ray Warning Light Array

2. Delta Docking Station (DDS)



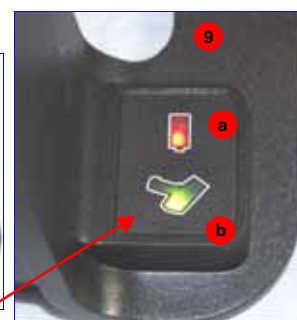
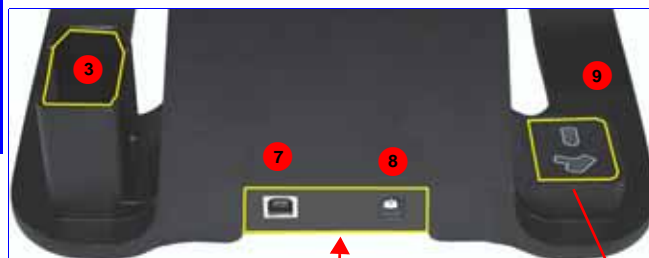
Component Key

Delta - All Model

1	Delta Docking Station (Empty)
2	Analyzer Signal/Control Connector
3	Spare Battery Charge Socket
4	CalCheck Test Cup (316 stainless steel coupon)
5	Docking Station (Loaded)
6	Second Battery in Socket
7	Data Port(s): — Docking Station ->Rear — Analyzer -> Left Side
8	Input Power (12 VDC)
9	Indicator Lights
a	Second Battery Charging
b	Analyzer Engaged



See C4/Pages 44-45
for more information



3. Accessories- List the Standard and Optional Accessories

Standard

- Batteries
- Delta Docking Station (DDS)
- DDS Power Adapter
- USB Cables 1 - USB mini to USB A
- USB Cable 2 - two part powered data cable
- Windows- Bags of Kapton and Prolene films
- Cal Check (Standardization) Coupon

Optional

- AC Power Adapter (Battery Replacement)
- A-020-D TestStand/Workstation for Delta

STANDARD Accessories

Batteries

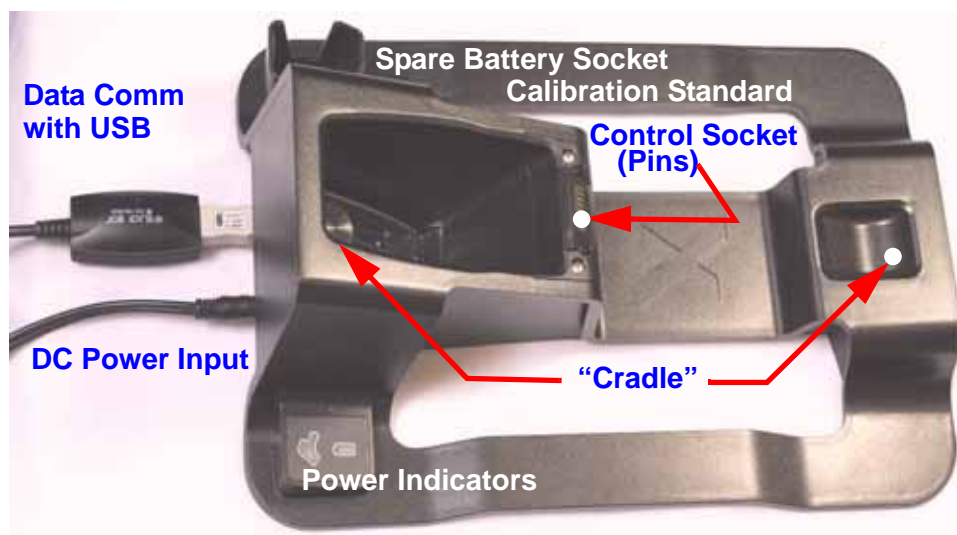
Two removable Li-ion batteries are standard accessories for the Delta.



Delta Docking Station (DDS)

This is key accessory. It provides three functions:

- Cal Check by one of two means - "On Demand" or Automatically
- Charge internal battery in handle
- Charge additional battery in auxiliary socket



See Chapter 4, Page 46
"Use DDS for Start Up & Initial Cal Check"

Power Adapter for DDS



See Chapter 4, Page 44
"Configure DDS"

I/O Cables

PN 101310: This *standard accessory* provides a means to transfer information into or out of the sealed analyzer. It is good practice to export the current day's testing results to your PC.

PN 101310
USB Data Cable
Mini USB B Connector
USB A Connector



PN 103209 and 103210: This is a two part assembly that supports communication between the Delta Docking Station and a PC.

Part One

PN 103209
USB Repeater Cable
USB A Female Connector
USB A Male Connector

Data Cable Assembly



Part Two

PN 103210
USB Adaptor
Male to Male
USB A Connector
USB B Connector

Cal Check (Standardization) Coupon

This part is used as a reference sample to provide a test standard for a Cal Check procedure if the Docking Stations is not available. The instrument indicates when a Cal_Check is necessary.



See Chapter 4, Page 51
"Cal Check Information"

Measurement Window Films

A bag of 10 window films are a standard accessory. The composition of the film is model and application dependent.

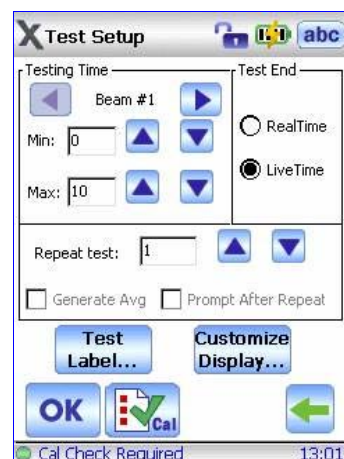
PN 103115 REV A
Kapton Windows
for Classic



PN 102999
Prolene windows for
Standard and Premium

Application Software

The *Delta* instrument is shipped with proprietary InnovX data acquisition and processing software and Windows Embedded CE® operating system. The User Interface employs an icon-based home page graphic style. Factory calibration has been completed on all purchased modes.



See the *User Interface Guide* (PN 103202) for a complete description of the Innov-X application's User Interface.

OPTIONAL Accessories

AC Power Adapter

PN 100043: This accessory enables the user to operate the instrument without the limitation of battery charge status. The unit comes with approximately ten feet of power cord that defines the effective range of use.



PC Software

This application package permits an operator to execute Innova S/W functions from a PC. With cable PN 101310, a user can connect from the Delta's mini-USB data port to a PC's USB port.

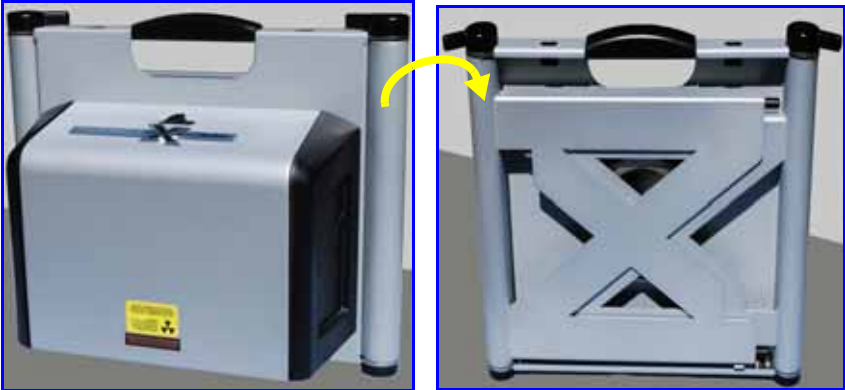
This package is optional for a handheld instrument and standard for A-020-D TestStand/Workstation. When used with the A-020-D the proper configuration cable is the powered USB assembly (PN 103209 - PN 103210)

XRF Workstation

The Delta XRF Workstation is comprised of two major components:

- A-020-D Test Stand, and
- Any Delta analyzer

In this configuration, the Delta is controlled by Innova Delta PC Software. The open-beam handheld instrument is converted to a closed-beam workstation.



The image shows two views of the XRF Workstation. On the left, the portable enclosure is shown. On the right, the test stand is shown. A yellow arrow points from the enclosure to the test stand.

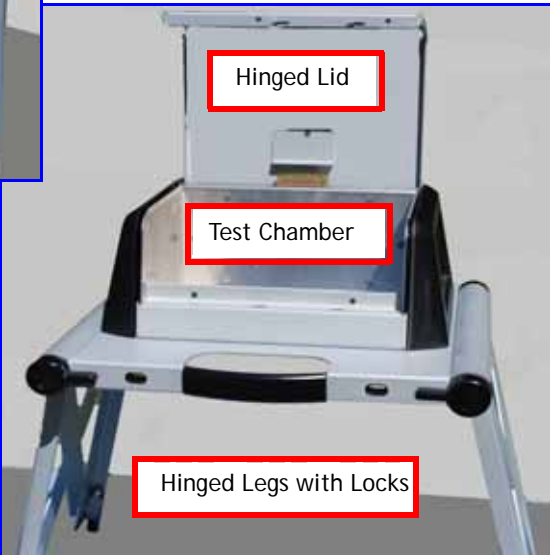
See PN 103158 document: *"HowTo-Setup & Configure Delta XRF Workstation"* for complete instructions.

Tour of XRF Workstation

The **XRF Workstation** offers the following features:

- Portable, light-weight, shielded enclosure
- A rugged and repeatable testing environment
- Easily erected in laboratory or at remote field site

In this configuration the Delta (an open-beam handheld instrument) is converted to a safe closed-beam system



The image shows the XRF Workstation with labels: **Hinged Lid**, **Test Chamber**, and **Hinged Legs with Locks**.

C2. Safety Information

C2 presents the following information:

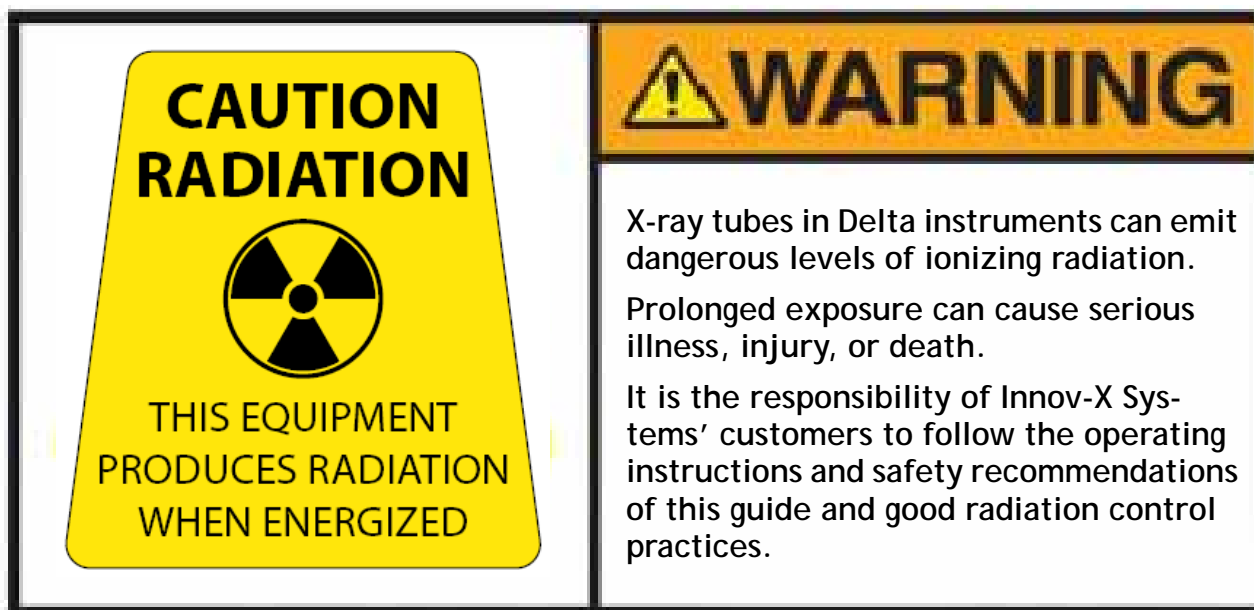
- Radiation Safety Information
- General Precautions
- Electrical Precautions
- X-Ray Safety
- Compliance
- Instrument Usage Scenarios
- Radiation Dosage for Several Scenarios
- Radiation Safety: Common Questions and Answers
- Analyzer Shut Down Procedure

Radiation Safety Information

Always make Operational Safety your HIGHEST PRIORITY.

The *Delta* Handheld XRF Analyzer is a secure and dependable instrument when used according to Innov-X's recommended testing techniques and safety procedures. However, this instrument produces ionizing radiation; only individuals trained in correct operating techniques and authorized to use X-ray producing devices should be permitted to use it. The radiation detected at any outside surface (excluding the Prolene, Mylar, or Kapton window area) is below that required for an unrestricted area.

- Heed all warning labels and messages
- Observe the safety interlock features



Radiation Safety Program


Innov-X strongly recommends that organizations using *Delta* analyzers implement a formal *Radiation Safety Program* that includes:

- Dose monitoring of critical personnel.
- Monitoring of area radiation levels.
- Information specific to the site and application of the XRF system.
- An annual review (and update, if necessary).

“C3. *Safety Administration*” provides a more comprehensive safety discussion for operators and managers.

X-Ray Safety

X-ray safety is a priority at any time and in any testing situation.

	WARNING
	<ul style="list-style-type: none"> • Innov-X analyzers must be used by trained and authorized operators, according to proper safety procedures. Improper usage may circumvent safety protections and could potentially cause harm to the user. • Heed all warning labels and messages. • DO NOT USE the instrument if there is any chance that it is damaged or might leak radiation. In such a case, arrange for qualified personnel to perform a radiation safety test and repair any analyzer damage.

General Precautions

Apply these general safety guidelines when managing or operating the *Delta* instrument:

- Retain and follow all product safety and operating instructions.
- Comply with all warnings on the product and in the operating instructions.

Comply with the precautions listed in this section to reduce the risk to:

- Users
 - Physical injury
 - Electric shock
 - Radiation exposure
- Equipment damage
 - Measurement window
 - Overheated electronics and other internal components

Service Considerations

Except as expressly noted here, do not service any Innov-X product yourself. Opening or removing the external housings may expose you to electric shock and the instrument to mechanical damage. It also voids the warranty.



CAUTION

If service is required, it must be performed by Innov-X or its authorized service representatives. Failure to observe this can result in loss of warranty. The **ONLY EXCEPTION** is replacing a damaged measurement window (see [“A5. Window Replacement”](#)).

Damage Requiring Service

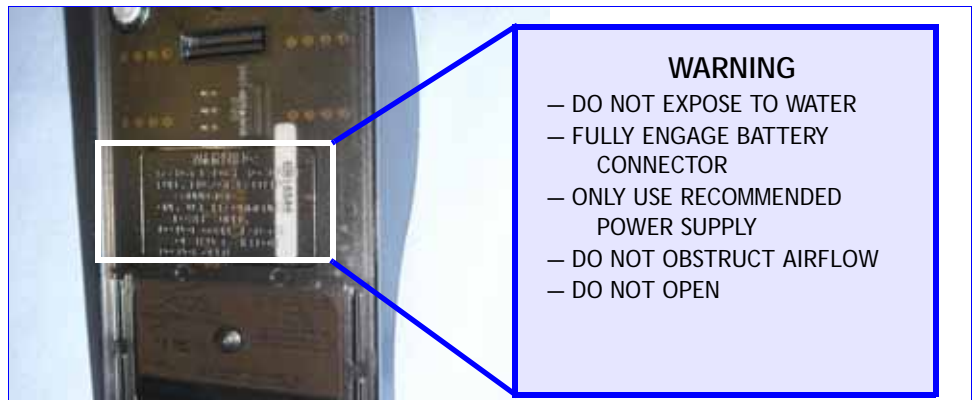
Types of problems or conditions that require service are (*but not limited to*):

- Power cords are damaged.
- Excessive or corrosive liquids spilled on the instrument or accessories.
- Instrument impacted, dropped, or physically damaged.
- Noticeable signs of overheating.
- Instrument or docking station does not perform normally when you follow the usual operating instructions.

Electrical Precautions

Guidelines for safe electrical operation of a *Delta instrument*:

- Use the correct battery or AC power adapter.
 - Install the battery or AC power adapter carefully, don't damage connections.
- Use the correct external AC power sources for the Delta Docking Station (DDS) (battery charging and Cal Checking) and the AC power adapter:
 - Ensure that the voltage is appropriate (100V-240 V/ 50-60 Hz) for operating either accessory.
 - See [“A3. Specifications”](#) for electrical specifications.
- Do not overload an electrical outlet, power strip, or convenience receptacle.
- Do not exceed 80% of the branch circuit rating.
- Comply with the warning messages on the under side of the Battery Charger.
 - Similar precautions should be observed for the Delta Docking Station (DDS).



Cables and Cords

The *Delta* instrument and docking station is delivered with:

- AC power adapter (1) for Docking Station (standard)
- AC power adapter (2) as battery replacement for instrument (optional)

Each device has a standard IEC 3 conductor power cord which includes a safety grounding plug.

- If necessary, have an authorized individual replace these plugs to conform to local conventions.

Two data cables are supplied:

- Data cable (1) with connectors — USB A to USB B
- Data cable (2) with connectors — USB A to mini USB B

Cable Guidelines

Use these guidelines to ensure safety and proper equipment performance:

- The power cords **MUST** be connected to a properly grounded and easily accessible power outlet.
- Use a surge protector device, if possible.
- Do not defeat or bypass the ground conductor.
- Do not pull on cords or cables. Grasp the plug housing when removing the cord from the electrical outlet.
- Install all cords in accordance with applicable regulations.
- If you substitute a USB cable, ensure that the length doesn't exceed 10 feet.

Delta Docking Station (DDS) and Li ion Battery Packs

Plug the Delta Docking Station (and optional battery charger, if utilized) into a grounded electrical outlet that is easily accessible at all times.

- To handle battery packs properly do not:
 - Disassemble
 - Crush
 - Puncture
 - Short external contacts
 - Dispose of in fire or water
 - Expose to temperatures higher than 60 °C (140 °F).



GO TO

See **“C4. Battery Issues”** for instructions concerning Batteries, the Battery Charger, and the AC Power Adapter.



WARNING

**Danger of explosion if battery is incorrectly substituted.
Replace only with Innov-X specified batteries.**

**Used batteries may be returned to Innov-X Systems for disposal.
If returning batteries, or equipment with batteries installed, the shipping container must display a special caution label.
See **“A6. Packing and Shipping”** for label details.**

Indicator and Warning Lights

Power Switch w/ Integral Indicator Light

The Delta power switch is located at the upper rear of the unit.



POWER ON

- Press the I/O switch to turn on the power.
 - A green LED indicator comes on.
- This switch **DOES NOT** turn on the x-ray tube.
 - No tube power supplied until the Innov-X software is launched and a test is initiated.



POWER OFF

- Press and hold switch for >3 seconds.
 - Unit powers off. (See page 36 for more Exit options)

X-Ray Indicator Light Array

An indicator light array (six red LEDs) alerts the operator when the tube is receiving power, and when x-rays are emitted from the analyzer through the measurement window.



- When the unit is initially powered ON, the Indicator array remains Off.
- As test is conducted, array is in a **flashing** state.
- At the test's conclusion, the array stays **on continuously** until the beginning of the next test.

X-Ray Indicator ON (*Blinking*)

When the indicator array is flashing, this signifies:

- X-ray tube is powered to full operational level
- Internal filter wheel is in operational position
- Analyzer is emitting x-ray radiation through the analysis window.

In this condition, the analyzer must be pointed at a test sample.

X-Ray Indicator ON Continuously (*Not Blinking*)

When the indicator array is on continuously, this signifies:

- X-ray tube's current is set to 0.0
- X-ray tube is producing a minimum level of x-rays
- Internal filter wheel is closed so there is no radiation exposure to you or bystanders.

The instrument is *safe* to be carried or set down in this condition.

Back of Analyzer



In addition to the I/O switch and the X-Ray indicator array, the back of the Delta analyzer has:

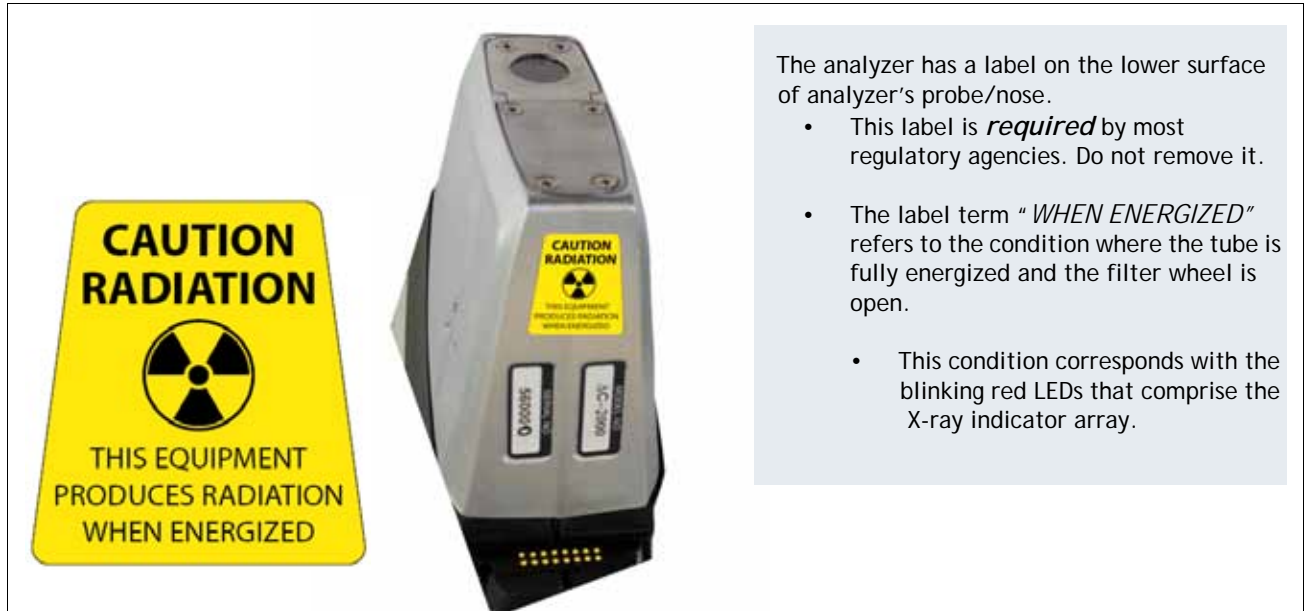
- Touch screen which displays and controls the Delta User Interface.
- Three Navigation Buttons below the screen. They permit the user to conveniently step through the Test Results Spectrum screens.



Navigation Buttons

X-Ray Label

The Delta has a warning label affixed to the lower surface of the probe.



Other Safety Features

Proximity Sensor

The Delta automatically detects when it is engaged with a test sample. It immediately shuts off the X-ray tube if:

(a) Initially there is no sample in front of the window,


— or —

(b) Instrument is pulled away from the sample before the test time has expired.

Safety Interlock Structure

For controlling the Delta's X-ray emissions and therefore minimizing the possibility of accidental exposure, there is a standard safety interlock structure consisting of the three features listed below.

Software Trigger Lock

- If five minutes elapse between tests (default time), the trigger locks automatically and you must tap on the lock icon  to unlock it. See Safety Software instructions in "*Delta User Interface Guide*".

Software Proximity Sensor

- Within two seconds of a test start, the analyzer detects a sample in front of the measurement window. If not, the test aborts, the filter wheel closes, and the x-rays shut off. The tube is placed in standby and the red light stops blinking.

Safeguards

As an owner of an Delta handheld XRF instrument, your safeguards are:

- Limited Access
- Trained Operators
- Shielding Issues

A. Limited Access

Keep the instrument in a controlled location, where only trained and authorized users are likely to have access.


B. Trained Operators

Keep a sign with the analyzer indicating that in order to use it an operator must have completed a training class provided by your company, or must have attended an Innov-X training course and completed any other requirements as dictated by the local regulating authority. When the Innov-X system is turned on, the controller screen displays a message indicating that the system should only be used by authorized personnel.

C. Shielding Issues

Background

The Delta emits a tightly collimated beam of X-ray radiation. The beam projects many meters when only air attenuates it.

	<p>NOTE</p> <p>Refer to governing regulations on compliance in the jurisdiction installed, dose limits, etc. Requirements differ from state to state, region to region, country to country. <i>DO NOT</i> rely solely on this manual for instruction.</p>
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Action

Adequate shielding is achieved by:


- Establishing a *no-admittance zone* sufficiently distant from the instrument's measurement window that allows air to attenuate the beam.
- Enclosing the beam working area with *protective panels* (for example, 1/8" stainless steel can attenuate the beam to background levels)

Contact your Innov-X Systems representative for assistance and suggestions on interlocks and applications for limiting radiation exposure.

Instrument Usage Scenarios

The Delta is used in several testing configurations. Obey the guidelines listed below.

Practical Safety Guidelines for Handheld Analyzers

	WARNING
	<ul style="list-style-type: none"> • DO NOT POINT the unit at yourself or any other person during operation. • Never perform a test by holding the sample with your fingers or in the palm of your hand. • Always wear both a ring-style and a badge-style dosimeter.

Correct Usage

Test in Place

Test targets can include pipes, valves, large pieces of scrap metal, soil, or any sample large enough to be tested in place.

In this configuration the proper procedure is as follows:

1. Always observe the relevant parts of the *Practical Safety Guidelines* shown above.
2. Point the instrument at the sample such that *no part* of your body (including hands and/or fingers) is near the measurement window.
3. Ensure that the Delta's nose (with window) is firmly placed on the target.
4. Perform the test using one of these methods:
 - Tap Start on the UI
 - or —
 - Pull the trigger (this toggles the instrument to ON state)
 - or —
 - Pull-and-hold the trigger with the "deadman trigger" active.

Employing Steps 3 & 4 assures that no operator's body part is exposed to an excess radiation dose. The radiation detected at user interface areas is < 5 µSv/h.

Take care that during testing, personnel are not located within three feet (one meter) of the Delta's probe head, in the direction of the x-ray beam. Provided the window is completely covered, there is minimal radiation being emitted around the area of the sample.

Small Component Testing

Examples of small component targets include metal turnings, weld rod, wires, fasteners, nuts and/or bolts.

For analysis of these types of components, use this procedure:

1. Always observe the relevant parts of the *Practical Safety Guidelines* shown above.
2. Place the sample on a flat surface.
3. Carefully place the nose/window over the sample.
4. Perform the test using one of these techniques:
 - Tap Start on the UI
 - or —
 - Pull the trigger (this toggles the instrument to ON state)
 - or —
 - Pull-and-hold the trigger with the "deadman trigger" active.

1. Sample lying on a flat surface



SAFETY PRECAUTIONS



WARNING

Do not test samples while sitting at a desk or table. If the desk is made of wood or another non-metallic material, some radiation will penetrate the desk and may provide exposure to legs or feet.

ANALYTICAL PRECAUTIONS



NOTE

If the sample does not completely cover the window, ensure that your *background surface* does not contain metals or even trace levels of metals, as this may affect the accuracy of the XRF result. The XRF may report the presence of additional metals in the surface material.

2. Clamp-held sample



NOTE

A handheld plastic locking clamp can be an effective and safe tool when analyzing small, irregular shaped samples.

Incorrect (Unsafe) Usage



WARNING

Never hold a sample in your hand such that any part of your body or appendages are exposed to the x-ray beam. Testing samples in this way may generate significant radiation exposure to your fingers.

Unsafe Testing Technique



The sample is held up to the measurement window with fingers. The sample does not completely cover the window. Even though the analyst is wearing a ring dosimeter, this is an unsafe testing technique. Here, the only value that the ring provides is to validate the level of unnecessary radiation exposure that has been experienced.

Unsafe Testing Technique



The sample is held up to the measurement window with fingers. The sample does not completely cover the window. To compound the danger, the analyst is not wearing a ring dosimeter. There is no measure of the radiation exposure endured.

Summary

InnovX repeats the Warning ---

NEVER hold a sample in your hand.

Testing samples in this way generates significant radiation exposure to your fingers.

Compliance

Complying Agency Statements

United States of America: FCC


Changes or modifications not expressly approved by Innov-X Systems, Inc. could void the user's authority to operate the equipment.

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications.

Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Radiation Doses for Several Scenarios

In this section we provide data, concrete examples of use and misuse of the analyzer and common questions and answers we encounter when training personnel on the safe use of the Innov-X analyzer. The goal is to explain scenarios of safe versus improper usage.

	WARNING
	<p>For the x-ray energy emitted by portable XRF analyzers (8-60 keV region), the bone in the fingers will absorb radiation about 3-5 times more than soft tissue, so the bone would be at an elevated radiation risk compared to soft tissue.</p> <p>For this reason, no person shall hold a test specimen in front of the window with the fingers in the direct beam, or direct the beam at any part of the human body.</p>
	<p>Reference: <i>Health Physics</i> 66(4):463-471;1994.</p>

The table below presents radiation doses for normal operating conditions and also for examples of misuse of the analyzer and even extreme misuse. Innov-X provides installation training that includes detailed radiation safety training and documentation designed to prevent misuse of the analyzer.

Although the doses shown below are derived from experiments with TLD (thermo-luminescent dosimeters) and may or may not represent actual absorbed dose in human tissue and bone in each scenario, they are examples of the level of x-ray radiation being emitted from the device.

The message is simple:

USE CAUTION AND PROPER TECHNIQUE when operating the device.

Example: Instrument Usage	Radiation Exposure and Comments
<p>Normal Operation- Dose to Hand</p> <p>User analyzes samples according to standard operating procedures described in this manual.</p> <p>Assumption: Operator using system with x-ray tube ON for eight hours/day, five days/week, 50 weeks/year. (Alloy sample).</p>	<p>Maximum exposure is to operator's hand, at the trigger is $< 1\mu\text{Sv/h}$. Annual exposure to hand is then $< 2\text{mSv}$.</p> <p>Maximum exposure under ICRP regulations is 500 mSv for radiation workers and 50 mSv for the general public. Thus continuous operation provides a dosage 250 times lower for a radiation worker and 25 times lower for the general public.</p>
<p>Normal Operation- Dose to Torso</p> <p>Analyzer is used under the same operating conditions described above.</p>	<p>Exposure to Torso is so low it cannot be measured (essentially background). To be conservative we use 1/2 the value as the trigger, $< 0.5\mu\text{Sv/h}$. Annual exposure using operating conditions above is then estimated at less than 1 mSv. Maximum allowed is 20 mSv under ICRP for radiation workers (1 mSv for general public).</p>
<p>Misuse Example 1:</p> <p>Operator holds samples in front of window with fingers, such that fingers are directly in the primary beam. Presumption is sample does not block any radiation.</p> <p>Do not do this!</p>	<p>At the window, in the primary beam, the maximum dose to the fingers is 20,000 mSv/hr. Assume an operator performs a 10 sec. test (typical). The dose to the operator's fingers or hand is $20,000 \times (10/3600) = 55 \text{ mSv}$. If the operator did this just ten times per year he would exceed the allowable annual dose of 500 mSv to an extremity.</p> <p>Take the extra time to test a sample on a surface or use a testing stand. Note: If the operator takes a shortcut and places his/her fingers within the primary x-ray beam at the window, they will exceed the annual dose rate.</p>
<p>Misuse Example 2:</p> <p>Operator places analyzer against body and pulls the trigger to start a test. Analyzer tests to preset testing time (usually ten seconds) unless operator pulls trigger again to stop test. This applies to analyzer being in contact with operator or with bystander.</p> <p>Do not do this!</p>	<p>Dose at exit of sampling window is 20,000 mSv/h.</p> <p>Dose for a ten second exposure with analyzer in contact with Torso: 55 mSv. If an operator did this act just once, he would exceed the annual safe dosage to the torso of 20 mSv/year by a significant amount!</p> <p>PLEASE NOTE: The maximum dose of 20 mSv/year is a whole body limit, which does not truly apply in this case because the x-ray beam size is small (about 25 mm² area at the port). Applying correction factors for the beam size is complex and beyond the scope of this manual. The important point is that for proper operation there is no reason to ever expose any part of the human body directly to the x-ray source. This example serves to provide estimated exposure in the event this occurs.</p>

<p>Misuse Example 3:</p> <p>Operator manages to initiate a test for ten seconds running normal soil mode and exposes a bystander that is standing ten cm away from analyzer port. What is exposure to bystander?</p> <p>Note: The proximity sensor would automatically shut down the x-ray tube immediately, so this is an extremely improbable occurrence. It would require a malfunction of the instrument - this safety feature is NOT modifiable.</p> <p>Note 2: Equations to scale these to other scenarios involving longer or shorter tests, and bystander being at distances other than ten cm are provided at right.</p>	<p>Dose to bystander at ten cm is 215 mSv/hr. For a ten second exposure the dose is 0.6 mSv. This is 33 times lower than the allowable dose to a nuclear worker in a year. This would have to happen 33 times to for that worker or bystander to obtain the maximum allowable dose.</p> <p>Formula for calculating other scenarios:</p> $\text{DOSE (in mSv)} = 6T/D^2$ <p>D = distance from port in inches T = testing time</p> <p>Example: Bystander is 30 cm away from port for a 30 second test. In this case the dose is calculated as:</p> $\text{DOSE} = 6(30)/30^2 = 0.2 \text{ mSv}$
--	---

Comparative Analysis:

Radiation Doses from Typical Exposures to Ionizing Radiation

Activity	Typical Dose
Smoking	<i>2.8 mSv per year</i>
Dental x-ray	<i>100 μSv per x-ray</i>
Chest x-ray	<i>80 μSv per x-ray</i>
Drinking water	<i>50 μSv per year</i>
Cross country round-trip by air	<i>50 μSv per trip</i>
Mammogram	<i>1-2 mSv per examination</i>
Yearly exposure from background* radiation * depends on geographic location	<i>3.6 mSv</i>

Radiation Safety: Common Questions & Answers

Question: When I'm shooting a piece of pipe or valve on a rack or on a table top, is there any exposure to people standing several feet away from the analyzer?

Answer: Even a thin amount of a dense metal sample (three to four mm thickness, not Al alloy) is enough to completely attenuate the emitted x-ray beam. Shooting a piece of material that covers the sampling window on the analyzer completely shields any bystanders from radiation exposure.

However, use good practice: Keep the area clear of people for at least four to five feet in front of the analyzer.

Question: If I forget to lock the trigger, I pick up the analyzer and accidentally pull the trigger, is that dangerous to nearby personnel?

Answer: No, this example of misuse is not dangerous, but it may produce a non-negligible radiation exposure to nearby personnel. For an exposure to occur, the following things must happen.

First, you must be holding the analyzer so that a bystander is actually standing in the x-ray beam being emitted. Just being near the analyzer is totally safe otherwise.

Second, the bystander must be within one meter from the nose of the analyzer to receive any appreciable dose. If all of these conditions are true, the dose received by a bystander is still extremely low. Please see Misuse Example 3 in the table above.

Third, it would require failure of the proximity hardware and software.

Question: Do I need to create restricted areas where I am using the analyzer?

Answer: No, provided you are following normal operating procedures there is no reason to restrict access to an area where the analyzer is in use. However, the operator should take precautions to keep any personnel more than three feet away from the sampling window of the analyzer in the event of accidental misuse as detailed above. Should operators also elect to test small samples as shown on pages 34 and 35, they should also be sure that no personnel are standing within about four to five feet of the sampling window.

Question: How does the x-ray tube in the Innov-X system compare to a radiography system used for taking images of metal parts?

Answer: The x-ray tube used in the Innov-X system produces between 1,000 and 10,000 times less power than most radiography systems (0.5-1 watt versus multiple-kW). A portable XRF is designed to perform surface analysis of alloys and other samples, whereas a radiography system is designed to shoot x-rays entirely through metal components in order to obtain an image on the other side of the test object. For example, many tube-based radiography systems use a 300-400 kV tube and currents in the tens or hundreds of milliamperes (mA). The Delta uses a tube operating at a maximum of 40kV and typically 6 -10 μ A. The radiation levels produced by an Delta are thousands, or tens of thousands, times lower than a radiography unit.

Question: Should we use dosimeter badges with the Innov-X analyzer?

Answer: Dosimeter badges are required by some provincial regulatory agencies, and optional with others. Innov-X recommends that operators wear badges, at least for the first year of operation, as a general precaution to flag any misuse of the analyzer. Dosimeter badges are available for the torso (generally worn in a shirt pocket) and also as “ring” badges.

The best practice is to wear a ring badge on a finger on the opposite hand used to hold the analyzer. This records accidental exposure for the most likely case - an operator grabbing a small sample and holding it in one hand while analyzing it.

Note: These badges generally have a threshold of 100 μSv and are renewed monthly. So it takes several cases of misuse even to obtain a reading on a typical badge. When purchasing a badge, obtain the type used for x-ray and low energy gamma ray radiation.

Analyzer Shut Down

There are several techniques for shutting off the Delta. They can be categorized by whether the action is taken under normal or emergency conditions. *Shut down* or *turned off* is defined as: *The analyzer cannot provide X-ray emissions.*

Under normal conditions

Use one of following actions:

- Press the trigger.
- Tap STOP icon on the UI touchscreen.
- Navigate from **Setup** > **Exit**, then choose the Power OFF icon.



- Release the trigger if in “Deadman Trigger” mode.
- Press the I/O power switch; ensure that the On/Off LED goes off.

In an emergency

Because the Innov-X system is a battery-operated, x-ray tube-based analyzer, the Emergency Response plan is simple. If you believe that the analyzer is locked up in an *OPEN* position, the red X-ray indicator array remains illuminated or blinking:

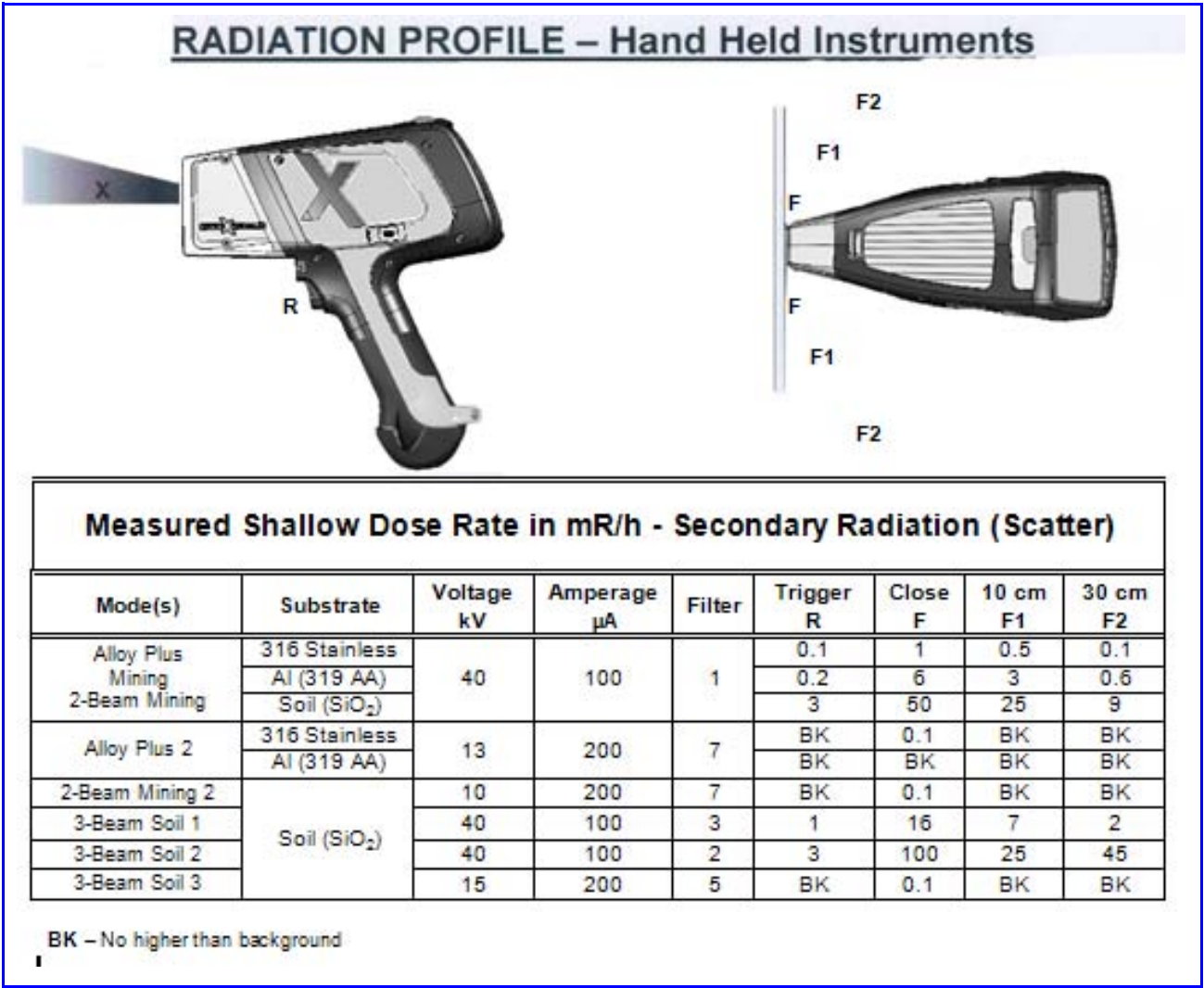
1. Press the I/O power switch as noted above. If the power does not turn off, continue to Step 2.
2. Open the battery cover and immediately remove the battery.

If you are using the AC Power Adapter:

- Remove the Battery Eliminator from the Delta’s handle
- or —
- Pull the AC cord from the AC Power Adapter or pull the plug from the receptacle.

Delta Radiation Profile

This is the current Delta Radiation Profile.



TEST CONDITION: Instrument run at normal setting for mode and represents typical production unit.

NOTES


C3. Safety Administration

C3 provides information regarding:

- Radiation safety training recommendations
- Dosimeter badges
- A typical dosimeter monitoring program
- Dosimeter service contractors
- Typical registration requirements for operating XRF equipment (in the USA)

Radiation Safety Training Recommendations

Individual companies and states have specific regulations and guidelines for using ionizing radiation generated by an X-ray tube.

	NOTES
	<p>For the convenience of clients, Innov-X has compiled a list of recommendations that:</p> <ul style="list-style-type: none"> • Provide generic guidance for an ALARA (<i>as low as reasonably achievable</i>) approach to radiation safety. • Do not replace the requirement to understand and comply with specific policies of any state or organization.

Personal Monitoring

Radiation control regulations may require implementation of a radiation monitoring program, where each instrument operator wears a film badge or TLD detector for an initial period of one year to establish a baseline exposure record. Continuing radiation monitoring after this period is recommended, but may be discontinued if accepted by radiation control regulators. See *Dosimeter Suppliers* for a list of film badges providers.

Proper Usage

Never point the instrument at a person. Never point the instrument into the air and perform a test. Never hold a sample in your hand during a test.

Establish Controlled Areas

Restrict access to the location of instrument storage and use to limit potential exposure to ionizing radiation. In use, the target should not be hand held and the area at least three paces beyond the target should be unoccupied.

Specific Controls

When not in use, store the instrument in a locked case or locked cabinet.
When in use, keep it in the direct control of a factory trained, certified operator.

Time - Distance - Shielding Policies

Operators should minimize the time around the energized instrument, maximize the distance from the instrument window, and shoot into high density materials whenever possible.

Prevent Exposure to Ionizing Radiation

All reasonable measures, including labeling, operator training and certification, and the concepts of time, distance, & shielding, should be implemented to limit radiation exposure to *as low as reasonably achievable* (ALARA).

Dosimeter Badges

A dosimeter badge consists of a radiation-sensitive material, generally an aluminum oxide crystalline layer, which is worn in a small container. It is most often attached to a person's clothing, on a belt loop, or shirt pocket. It is worn on the body in location(s) that most closely simulate the pattern of potentially absorbed dose.


The protection can also be provided in a plastic ring format. Here the detection material is lithium fluoride crystal.



These devices record a person's accumulated radiation exposure over a period of time. They monitor individuals working with, or near someone working with devices which emit ionizing radiation.

Dosimeter badges are required by some regulatory agencies, and are optional with others.

Innov-X recommends that (at a minimum) all *Delta* operators wear badges (both clip-on and ring styles) for the first year that their system is in use.

	NOTE
	<ul style="list-style-type: none"> When purchasing a badge or ring, always select the type used for <i>X-ray</i> and <i>low energy gamma</i> radiation. Innov-X suggests that the ring badge be worn on a finger of the <i>opposite hand</i> used to hold the analyzer. This records accidental exposure for the most likely case: An operator grabbing a small sample and holding it in a hand while analyzing it.

Every country (including every region, state, or province within a country) can have differing regulations. Always consult your local radiation protection authority or Innov-X Systems for information and recommendations.

Dosimeter Safety Program

A typical dosimeter-based safety program uses the following steps:

1. The company develops a dosimeter program with an independent service contractor.
 - They establish the quantity of badges needed and the frequency of analysis (a monthly or quarterly interval)
2. The company receives the first lot of badges and provides them to their analyst/operators.
3. At the end of the interval:
 - The company collects the badges and returns them to the service contractor for analysis.
 - Simultaneously, the service contractor delivers another lot.
4. The company provides the new set of badges to maintain a continuous protection /monitoring program for their employees.
5. The service contractor prepares a report for the company that tabulates any X-ray dose received and identifies any personnel with readings higher than typical background radiation.
6. The safety monitoring cycle repeats with Steps 1 through 5.



NOTE

The service contractor's written records are very important to a company's overall safety documentation plan.

Dosimeter Suppliers

Some dosimeter service companies are:

Company	Location	Telephone
AEIL	Houston, TX	713-790-9719
Global Dosimetry Solutions	Irvine, CA	800-251-3331
Landauer	Glenwood, IL	708-755-7000
• Landauer, Inc.	Oxford, England	+44-1 86-537-3008
• Nagase Landauer, Ltd.	Japan	+81-3-36 66-4300
• LCIE Landauer	Paris, France	+33-(0)1-40 95 62 90
• Landauer	Beijing, China	+86-10-62 21 56 35

Registration Requirements

Contact Innov-X for assistance with locating registration requirements information.

- Most states require some form of registration. Generally they require the registration to be received within 30 days of receipt of the system.
- Some states require no registration.
- Some states require notification in advance.

Customers are advised to consult their local radiation protection authority for specific regulatory information.

Typical Device Registration Information

The following information is usually requested by a licensing agency:

Purpose of device:

Response is *Analytical or Industrial*.

Be sure to inform the government registration office that the system will NOT be used for radiography or for medical uses.

Radiation Safety Officer:

List person who monitors training, safe use, and controls access to the system.

Authorized Users:

List the analyst/operators who have been trained and authorized by the instrument owner and/or regulating agency to operate the XRF equipment.

Operating parameters of the Delta XRF analyzer:

8– 40 kV, 5 - 200 uA max.

Type of system:

Response is: *Handheld/Portable*

User Training Specified:

Indicate that only individuals receiving manufacturer training, *documented by a manufacturer's training certificate*, will operate the system. Additional training may be required. Verify with the local regulating agencies the level and type of training required.

Personal Monitoring

Many government agency registration forms require that you indicate whether or not you intend to perform dosimeter monitoring.

See "Dosimeter Safety Program" on previous page for information regarding typical personal radiation monitoring.

CAUTION



Always keep the following documentation at the job site:

- Copy of *License Registration*
- Other pertinent *government agency* documentation
- Copies of any *dosimeter analysis* reports
- Copy of this equipment's *User Manual*.

C4. Operations

This chapter provides information regarding:

- Configure the Delta Docking Station (DDS)
- Use DDS for Initial Cal Check
- Operation - General
- Start Up Procedure
- Snapshot of Delta User Interface
- Typical Test Procedure
- Ending Test Operations
- Battery Issues
- Additional Cal Check Information
- TIPS - Things You Should Know About the Delta

Safety First !

As emphasized in *"C2.Safety Information,"* it is a priority to keep the analyzer operator's safety in mind at all times.

- Operators, *before turning on the analyzer or using the Delta Docking Station*, should review the safety procedures (*"C2.Safety Information"*).

Set Up and Use the Delta Docking Station

Background

The Delta Docking Station (DDS) provides several key functions:

- Supports an automatic or on-demand Cal_Check procedure
- Charges the "Main" battery located in the instrument's handle
- Simultaneously charges a "Spare" battery in an auxiliary socket
- Provides control information so that both batteries' status can be monitored
- Allows data communication from the Delta to a PC via a powered USB cable

The first phase for preparing to operate a Delta involves:

1. Configuring the DDS with its power and communication cables
2. Using the DDS to support the Delta's initial:
 - a. Start up sequence, and
 - b. Cal Check procedure.



NOTE

A new instrument is shipped with two fully charged Li Ion batteries. Therefore, prior to **initially** using the analyzer, it is not necessary to charge a battery.



GO TO

- See [Pages 45 and 50](#) for battery information, including charging, changing, determining status, and Hot Swap techniques.
- See [Page 51](#) for Cal Check background information.

Configure Delta Docking Station



Rear View - DDS



PN 103209 & PN 103210
Powered Hub USB Cable Assembly

Configure DDS with this procedure:

1. Plug the AC power adapter's line cord (1) into suitable AC outlet.
2. Plug the AC power adapter's DC jack (2) into the 12 Vdc socket on the rear of DDS
3. {Option at this point: Powered USB cable assembly}
 - Insert connector B (3) into DATA port
 - Insert connector A (4) into USB port of PC



Power Adapter for DDS

Use the Delta Docking Station for Charging Batteries

The Delta analyzer has a multi-purpose standard accessory:
the Delta Docking Station or DDS.

In addition to providing an automatic Calibration Check, the DDS delivers two charging functions:

1. It charges the installed "Main" battery in the instrument's handle.
2. Simultaneously, it charges a spare or "Dock" battery when its inserted into the special auxiliary charging socket.

Charge status is shown in real-time on the Delta's display screen.

The second docked battery's status is also shown by the battery icon located on the rear left side of the DDS.

(either "charging = red" or "full = green")



Battery Status Screen
(Delta inserted in DDS)

Delta Docking Station Procedure for Battery Charging Functions:

1. Ensure that the DDS has DC power cable installed
See [page 44](#)
The Icon lights are Off {1}
2. With Delta Off, place instrument in cradle.
Take care that Analyzer Icon comes On (Green) {2}
The main battery in the handle is charging although there is no indicator.
3. If Delta On, the Battery Status Screen appears
4. Place a spare battery in the auxiliary socket
Battery Icon is On (Red - unless fully charged) {3}
5. Both batteries will charge; their real-time charge status is displayed.
6. When both batteries are fully charged, the Icons display Green. {4}



Cradle



Delta Docking Station Procedure for Automatic Cal_Check

1. Ensure that the DDS has DC power cable installed
See [page 44](#)
The all Icon lights are Off {1}
2. With Delta On, place instrument in cradle.
Take care that Analyzer Icon comes On (Green) {2}
Battery Status screen is displayed.
3. Ensure that the Battery Status screen stays On.

NOTE: Do not exit this screen -->> the automatic Cal_Check function will not work.



Use the Delta Docking Station for Startup - Initial Cal Check



DDS w/Empty Cradle



DDS w/Premium Delta in Cradle



1. Place analyzer into the Delta Docking Station cradle. Ensure that the Delta indicator light is ON. This signifies that the instrument is properly seated in the cradle.
2. Press analyzer's I/O switch (>1 second to turn it ON.)



DDS - Indicator Light
Delta is engaged in cradle

Radiation Safety NOTICE appears after a few seconds.

3. Read and respond to notice
Tap **START** to acknowledge that you are a certified user.
The UI begins its launch with the following messages:

Initializing System

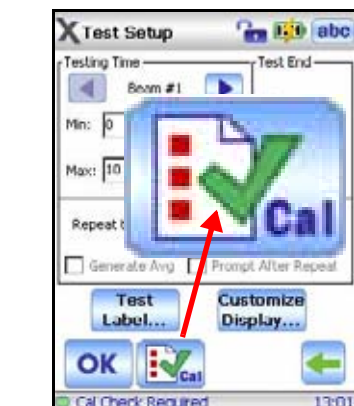
Starting System

Loading Files

4. Unit displays a Test screen using the Mode last selected.
 - 4a. If the mode should be changed, go to **Home** screen.
 - 4b. Select the Mode button
 - 4c. Choose the desired Mode

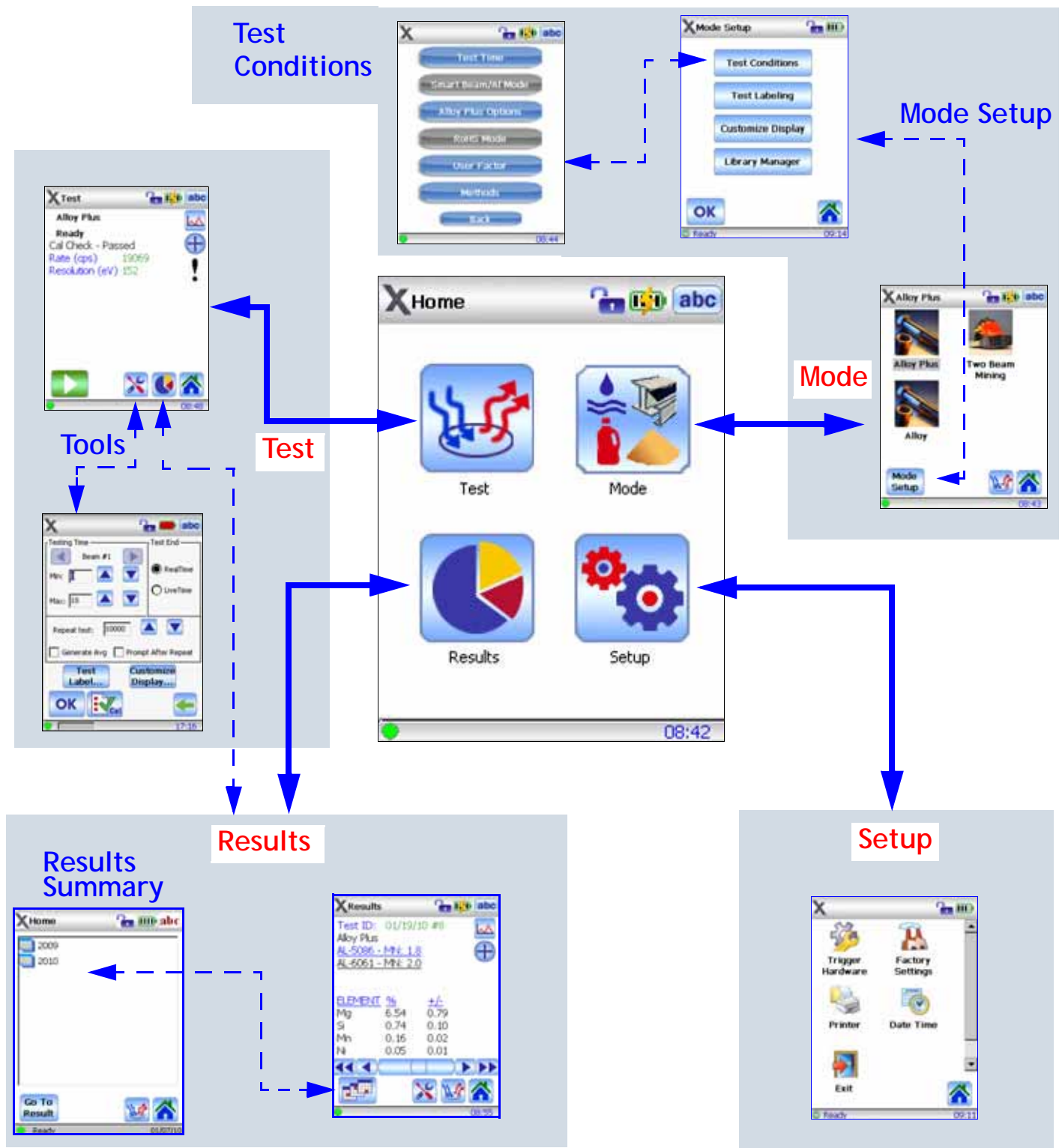


5. Return to Test (now with your selected Mode)
Note that message *Cal Check Required* is present.
6. Choose **Tools** icon:
This launches **Test Setup** screen with **Cal_Check** button
7. Tap **Cal_Check**
If necessary, unlock the **Trigger** with the icon at top of screen.
Procedure begins immediately; it concludes in about 15 seconds.
8. Message *Cal Check - Passed* means you may begin testing.
Message *Cal Check - Failed* will give error message, such as "! Wrong Count Rate"
Re-try the procedure
If unit fails repeatedly, contact InnovX service or your local distributor.



SNAPSHOT of Delta's User Interface

The Delta's user interface is introduced by the startup Radiation Safety and Initialization screens. Main operations then revolve around the Home screen.



Typical Test Procedure

Background

Details of routine testing operations vary depending on the selected analysis mode. Some relevant modes for Delta XRF users are:

<ul style="list-style-type: none"> Alloy Modes <ul style="list-style-type: none"> —Alloy Plus —Fast ID & Pass/Fail —Precious Metals 	<ul style="list-style-type: none"> Mining Modes <ul style="list-style-type: none"> —Two-Beam Mining —Mining —Car Catalyst 	<ul style="list-style-type: none"> Soil Modes <ul style="list-style-type: none"> —Environmental —Exploration 	<ul style="list-style-type: none"> Consumer Goods <ul style="list-style-type: none"> —RoHS —Consumer Products
--	--	--	---

For the test sequence (below) the instrument has:

- Mode selected (Soil 3 Beam), and
- Cal Check procedure successfully completed.

To conduct a typical test:

- Remove the instrument from the Delta Docking Station. Place the the measurement window flush against the sampling area. Ensure the sampling area is covered by the window.



WARNING

Do not point the unit at yourself or any other person during operation. Do not test small samples in your hand. Place them on a surface for testing.
See [C2, Safety Information, Pages 29-31](#) for examples of safe and unsafe testing techniques.

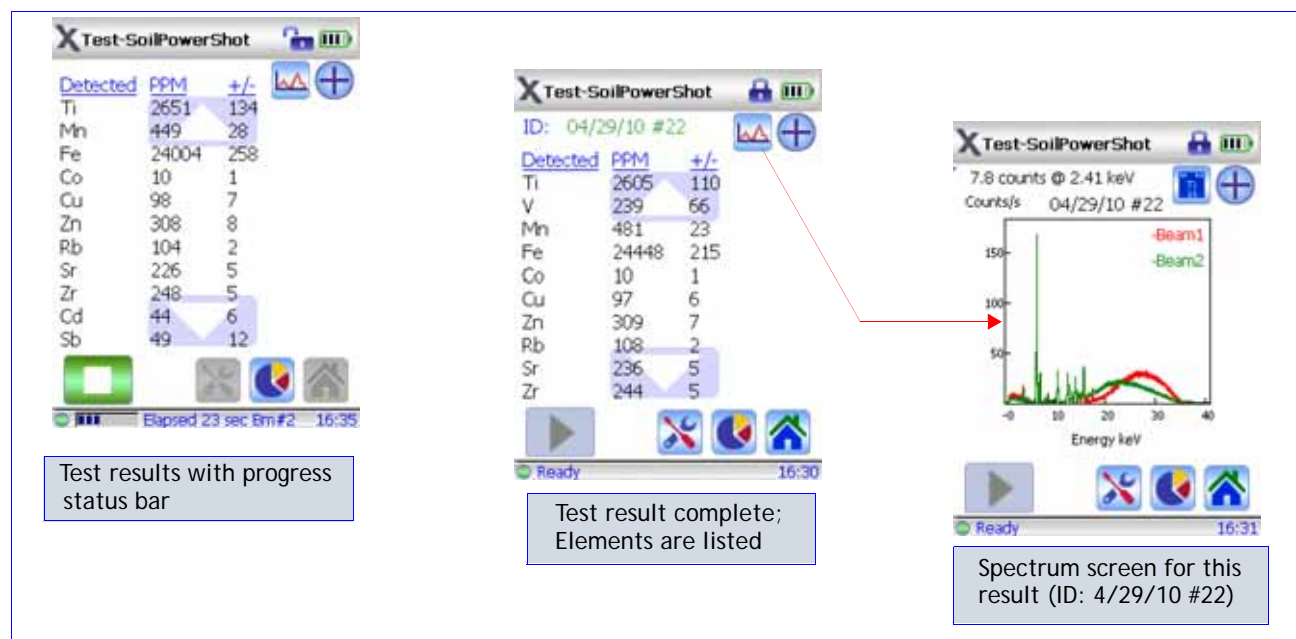
- Use one of these techniques to initiate the X-ray beam:
 - Tap Start Test (Green Arrow Icon), or
 - Pull the trigger (toggles unit ON); can release the trigger during the test, or
 - Pull-and-hold the trigger (deadman trigger function is enabled)

This is a mandatory technique in Canada.

Trigger options are configurable from:

Setup > Trigger Hardware > Trigger Settings

The Test screens are as follow:



End of Day Operations

Save Results

When finishing testing for the day (or shift, or current session) InnovX recommends that test results be saved (e.g. exported) to a PC.

A necessary prerequisite is a DATA connection between the analyzer and PC.


This connection is made in one of two ways:

- Analyzer in DDS cradle — use the powered hub USB cable assembly (PN 103209 and PN 103210) from the DDS' rear Data port to a USB port of the PC
- Analyzer NOT in DDS cradle — use the mini USB B to USB A (PN 101310) cable from the analyzer's Data port to a USB port on the PC

The UI operational sequence is:

1. Navigate from Home > Results > Calendar
2. Select Year, Month, Day listing; it lists the total number of tests for the day
3. Select Tools, then Results Setup
4. Select the Export icon (button)
5. Choose the results to be exported
6. Name the export file (or accept default name)
7. Select Destination to save to
8. Tap the Export button

The file is exported.

	<p>GO TO</p> <ul style="list-style-type: none"> • See "<i>Delta SW User Interface Guide</i>" for the details (including options) of this sequence.
---	--

Ending Test Operations

When testing and exporting are complete, the user has the following options:

- Turn off Delta with I/O switch or UI (see Exit Options procedure below); store unit in a secure location
- Place Delta in Docking Station and use the "Automatic" option:
 - Leave Delta powered ON;
 - Ensure that DDS is On (Delta ICON is lit);
 - Unit initiates Cal_Check after being idle for five minutes, then every ten hours thereafter.

Exit Option Procedure from UI



Battery Issues

1 — Changing a Battery

To CHANGE the battery:

1. Hold the instrument by the handle, upside down, so the bottom of the instrument base is pointing upward with the nose pointing away from the operator.
2. Pull the rubber latch and lift cover.
3. Remove the existing battery using the tab. A new instrument will **not** have an installed battery.
4. Insert the charged battery into the analyzer with the battery connectors facing to the left. The battery slot is keyed so that the battery can be inserted only one way.



2 — Battery Status

To TEST a Li-ion battery's charge status:

EXTERNAL battery test —

1. Push the white button on the battery.
The green lamps indicate the percent of charge, from less than 25% to 100%.
2. If a battery has a charge of less than 25%, use the Delta Docking Station to establish a full charge. See [Page 45](#).

INTERNAL MAIN battery test —

1. The battery icon from any UI screen (upper right side) shows an approximate value of charge.
2. Tap the battery icon and a more precise charge percentage is displayed as a number.



3 — HOT SWAP for Delta Battery

A battery HOT SWAP capability is a **standard feature** with the Delta analyzer. An operator can remove and replace a battery without having to shut down, restart, or Cal Check.

When the battery is removed:

- A "Shutdown" status display gives the percentage of internal charge remaining.
- If the internal charge reaches 0, you have to re-start the unit with the I/O switch, after inserting a fresh battery.
- If red X-ray indicator lights flash, the battery voltage is too low.



Cal_Check Information

Cal_Check Facts

Question: What is involved with the Cal_Check procedure?

Answer: The analyzer:

- Collects a spectrum on a known standard (Alloy 316 Stainless Steel)
- Compares a variety of parameters to values stored when the instrument was calibrated at the factory.
- When comparisons are within pre-set tolerances, the unit determines that it remains properly calibrated.

GENERAL FACTS INCLUDE:

- Cal_Check must be performed when the analyzer requests the procedure.
- The *Start* button and trigger are disabled until a successful Cal_Check is achieved.
- You can run a Cal_Check at any time during *InnovX* software operation, except during a test.
- When Cal_Check is in progress, the x-ray indicator light assembly blinks. This indicates that the X-ray tube is energized and the filter wheel is operational. In addition, a status bar appears on the UI display, showing the percentage completion for the measurement.
- The Cal_Check procedure takes about 15 seconds.

Cal_Check Procedures

There are two separate techniques:

- **In the test laboratory** - Use the DDS to initiate "on-demand" procedure. Described above in *"Use the DDS for Startup - Initial Cal Check"*:

Also have the "Automatic" option, as follows:

Leave Delta powered On and InnovX software running;
Ensure that DDS is On (Delta ICON is lit);
Unit initiates Cal_Check after being idle for five minutes,
then every 10 hours thereafter.



- **In the field** - Use the Coupon (the procedure is described below)

1. Place the 316 stainless steel Cal_Check coupon on a flat surface.
See *C2. Safety Information, Page 29* for safety measures that must be observed.
2. Position the analyzer's measurement window flush over the coupon.
3. With the Test Setup screen invoked, tap the Cal Check icon. There are now two techniques to choose from:
 - Tap the "Start Test" icon, or
 - Pull the trigger (or pull-and-hold if using "Deadman Trigger")
4. The procedure takes about fifteen seconds.



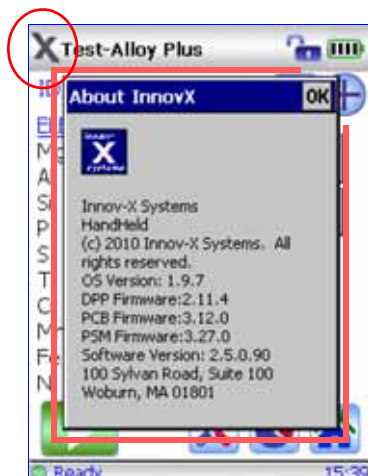
NOTE

Improperly positioning the window over the coupon can result in a failure.

5. When Cal_Check completes successfully, you may begin testing.
6. If Cal_Check fails, ensure that:
 - Coupon is positioned correctly.
 - X-ray indicator assembly is blinking during the procedure.
 - You have waited several seconds before starting the procedure.
7. If Cal_Check fails again,
 - Shut down the Delta software.
 - Shutdown and restart the analyzer.
 - Launch another attempt to Cal Check.

If Cal_Check fails repeatedly, contact Innov-X Systems Customer Service or your local distributor. Contact information is available at appendix *A6. Packing and Shipping*

TIPS - or - things you should know about the Delta



System Information

To call information "About InnovX" tap the X (InnovX icon) in upper left of UI screen. This provides various firmware and software versions that are installed on the analyzer.

UI Screen Note

All User Interface screens have a time-out (power saving) feature that causes the screen to go blank after 45 seconds if the UI is not accessed or the unit is not moved.

However, the analyzer is still running.

Restore the screen by tapping it or by moving the instrument.

Battery Status Info #1

When you turn on the instrument and you may not be aware that the battery is low, the X-ray indicator (Red LEDs) flashes dimly and briefly. The unit will not turn ON. Swap out the battery.



Battery Status Info #2

The on-screen battery icon (in upper right corner of UI) shows "real-time" battery charge status in a graphical way. Tap this icon to receive a numeric value for battery charge level.



Delta Docking Station to Delta Analyzer: Contact Status

Keep the rubber boot attached to instrument when inserting it into the DDS. This helps ensure that the DDS contact pins are engaged.

The DDS analyzer icon (rear left corner) should be ON.

If the rubber boot is not available, and the analyzer icon remains OFF, use a small piece of padding under the handle to ensure contact.

C5. Alloy Analysis Modes

Alloy analysis for the Delta family includes:

- Wide range of modes and calibrations
- Outstanding performance for a variety of materials

There are five Alloy modes/calibrations:

ALLOY

Use with: **ALL Models**



ALLOY

Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Hf,
Ta, Re, Pb, Bi, Zr, Nb, Mo, Ag, Sn, Sb

Fundamental Parameters Analysis for metal alloys.

Alloy library including 300+ grade specifications, common tramp limits, including full editing capabilities.

ALLOY Plus

Use with: **Premier and Standard Only**



ALLOY PLUS

Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Hf,
Ta, Re, Pb, Bi, Zr, Nb, Mo, Ag, Sn, Sb
PLUS Mg, Al, Si, P

Fundamental Parameters Analysis for metal alloys.

Optimized beam condition for extended light element performance.

Alloy library including 300+ grade specifications, common tramp limits, including full editing capabilities.

FastID

Use with: **All Models**



Spectral signature matching for alloy grade & chemistry calculation.
Full library editing & alloy matching capabilities included.

Pass/Fail

Use with: **All Models**



Spectral signature or chemistry matching for alloy grades.
Customer created library with min/max grade specifications.
Full library editing & alloy matching capabilities included.

Precious Metals Additions

Use with: **All Models**

ADDS Ir, Pt, Au, Rh, and Pd to [Analytical Analysis](#) calibration suite.

Introduction to Alloy Analysis Modes



The Delta family of instruments currently presents six unique modes for alloy analysis. The core analytical analyzer modes/types are:

- Alloy mode - Classic (PiN detector based) type
- Alloy Plus mode - Standard (SDD detector based) type
- Premium (SDD detector based) type

All three analyzer types can support additional alloy-oriented modes, such as

- FastID
- Pass/Fail
- Precious Metals (No added details)

Alloy analysis utilizes a Fundamental Parameters (FP) algorithm to determine elemental chemistry. This method calculates chemistry from the spectral data, without the requirement of stored fingerprints. The Analytical FP calibration is done at the factory, and requires no user set-up or recalibration. The software also searches an alloy grade library to produce a grade match based on the calculated chemistry. Analytical mode can provide a grade ID and chemistry in as little as one second, with increased precision for longer test times.

Alloy mode/Classic type supports Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Hf, Ta, Re, Pb, Bi, Zr, Nb, Mo, Ag, Sn, Sb.

Alloy Plus mode /Standard type and Premium type supports light elements Mg, Al, Si, and P in addition to the core list of elements from the Alloy mode.

Standard and Premium units expand the Limits of Detection range permitting operators to analyze these light elements without a vacuum or helium purge requirement.

Both modes have a feature, Altitude Compensation, which automatically corrects calibrations based on barometric pressure.

Determination of Grade Identification:

Analytical modes utilize a Factory Grade Library consisting of a set of minimum and maximum values for each element in an alloy.

There is a SPECIFIC Alloy Factory Grade Library for EACH Delta model.

See appendix [A8. Alloy Grade Libraries](#) for a listing of the alloys that are contained in each Factory Grade Library.

Additionally, every analyzer is shipped with a “Tramp” library comprised of seven base alloys. These seven items with their min/max element values are increasingly valuable to fast and accurate sorting in Pass/Fail and FastID modes.

The libraries can be searched individually or together. All libraries, including each Factory Grade Library, can be edited by the user. However, InnovX strongly suggests that users NOT edit the Factory Grade Library. Instead, copy the Factory Grade Library to a USER library, then make any edits on it.

Match Number Concept

After calculating chemistry with the Fundamental Parameters algorithm, *Innov-X* compares the chemical composition values to grade tables stored in a grade library. The application calculates the value for a parameter called *Match Number*. This provides an indication of how close the measured alloy's chemistry is to library values.

- The *lower* the Match Number, the *better* the match.
- A Match Number of 0 is an *exact match*, meaning that the calculated chemistry for all elements falls within the grade table specifications.

	GO TO
	<ul style="list-style-type: none"> • See "Delta User Interface Guide" (PN 103202) for a complete description of the <i>Innov-X</i> application's User Interface. • See A8. Alloy Grade Libraries for listing of each model's "Factory Grade Library" <ul style="list-style-type: none"> • A "Spectral Fingerprint" library that would be used for "FastID" and "Pass/Fail" analysis is created by the customer.

Match Issues

There are three Match determination possibilities provided within the Analytical modes:

EXACT MATCH

An unknown alloy is matched to one of the grades contained in the Grade Libraries, and a *Grade ID* appears on the *Results* screen. Often other grades are listed with their accompanying Match Numbers. The analyst has the opportunity to view their elemental chemistries and see how they differ from an exact match.

MULTIPLE MATCHES

In some cases, several grades are shown as possible matches. This can signify one of two conditions:

- There was not enough statistical information to definitively separate two or more alloys. The actual identification of the unknown alloy is one of the grades listed. Often increasing the testing time makes it possible to separate the alloys.
- There was sufficient statistical information, but the test sample did not meet any of the existing specifications with enough precision to cause an exact match identification.

NO MATCH

If no matches are found within the libraries, the words *NO MATCH* appear.

There are several causes for a NO MATCH result:

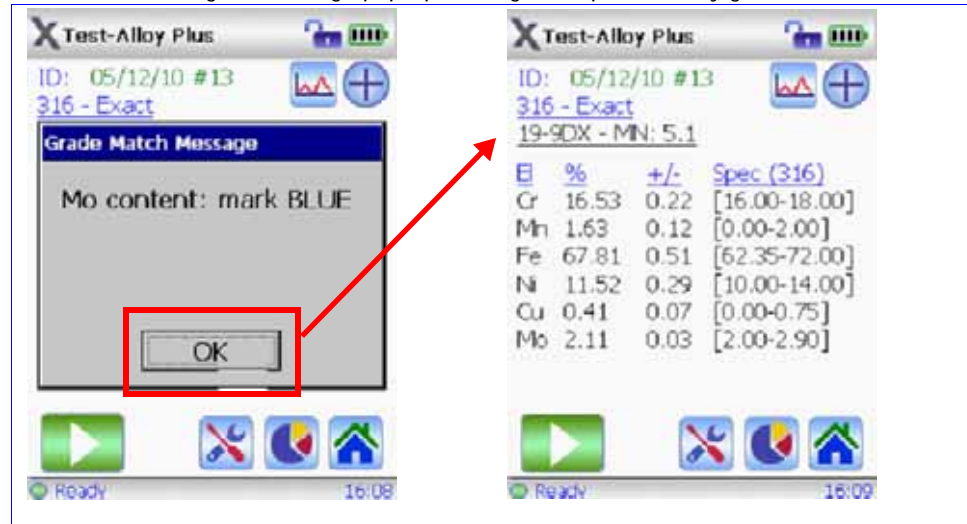
- The test sample does not meet any of the specifications in the Grade Library.
- The test sample is coated; Remove the coating by grinding, filing, or sanding and repeat the test.
- The testing time was too short.
 - Increase the testing time and measure the sample again.
- The Match Number is too low.
 - If possible, increase the Match Number

Scrap and Recycling Features

Delta analyzers in Alloy or Alloy Plus modes support many new features to specifically enhance scrap processing by maximizing speed and accuracy.

Grade Match Messaging (GMM)

User or Yard Manager can assign pop-up messages to specific alloy grades



- Quick start for next test, or view the chemistry details with one click

Grade Match Messaging offers:

- Immediate sorting instructions
- Less operator training
- More efficiency and higher throughput

SmartSort

Automated sorting decisions that allow users to maximize speed and sorting accuracy.

Some features include:

- Specific grades set up to automatically extend testing time.
- Maximize efficiency for speed testing by automatically extending test time for light elements (Mg, Al, Si, P, S)
 - Eliminate unnecessary long tests

Nominal Chemistry

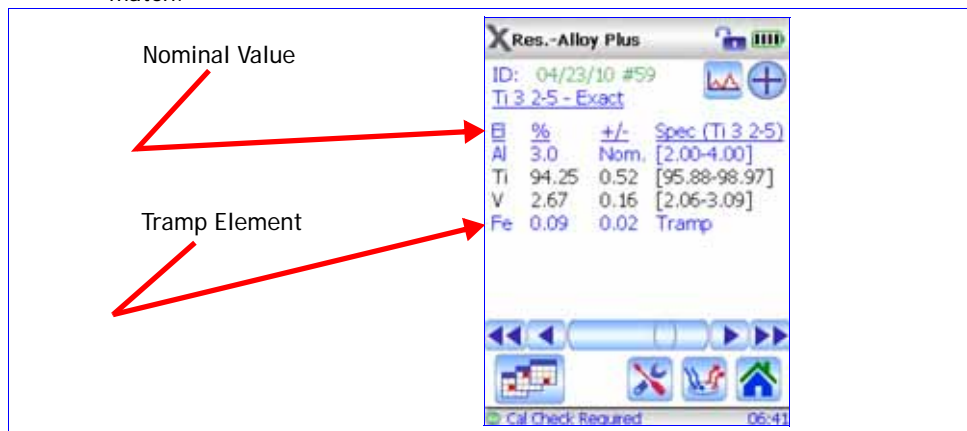
Nominal Chemistry looks for 'invisible' elements, based on grade ID, including:

- Elements not tested under active beam, (like Al in Beam 1)
- OR —
- XRF invisible elements (like B or C)

Tramp Library

Analyzers with Alloy or Alloy Plus mode come pre-loaded with a tramp library based on industry standards.

- Operators may assign other "Tramp" elements with max tolerated concentration for individual elements in seven unique graded families.
- Analyzers can report tramp material (optionally) and simplify grade match by not counting small, expected amounts of tramp elements against the grade match.



X Res.-Alloy Plus			
ID: 04/23/10 #59			
Ti 3 2-5 - Exact			
	%	+/-	Spec (Ti 3 2-5)
Al	3.0	Norm.	[2.00-4.00]
Ti	94.25	0.52	[95.88-98.97]
V	2.67	0.16	[2.06-3.09]
Fe	0.09	0.02	Tramp

Cal Check Required 06:41

See appendix [A8. Alloy Grade Libraries](#) for a discussion of the Tramp Library concept, including:

- How the InnovX Tramp Library works
- Practical advantages of the Tramp element approach
- List of seven Tramp Base Alloys

Test Sample Considerations

Coated or Painted Samples

XRF is a surface analysis technique, where X-rays penetrate a very short distance into most alloy samples. Therefore, the analyzer detects what is on the surface of an alloy, rather than what comprises the bulk of the material. If a material has been coated, plated, painted, or has had some sort of surface treatment, such as heat treating, it may be misidentified.

For example, a steel piece painted grey may show high concentrations of titanium from the paint, and may be misidentified as a titanium alloy. In another example, large amounts of metal dust or turnings on a surface may be detected by the analyzer.

To ensure proper identification of coated materials, grind an area slightly larger than the analyzing window to remove the coating. It is important to select the correct grinding material so as to not interfere with the analysis.

Do not use Silica for a Silicon analysis.

It may not be necessary to completely clean and polish all materials, however, remove obvious metal dust.

Mixed Samples, Heterogeneous Materials

Often finished metal pieces may consist of more than one type of metal. In addition, you may wish to measure mixed turnings, or an assortment of small pieces. In these cases, remember that the analyzer measures the entire area covered by the analyzing window and reports an average chemistry. For turnings, this is useful, as the analyzer provides an average composition. However, if two or more pieces of metal cover the window, the results is just an average reading, and may tell very little about the composition of one piece or the other.

When shooting metal pieces, or welds, ensure that **only** the metal of interest is covering the analyzing window.

Small and Irregularly Shaped Samples

To measure samples smaller than the analyzing window:

- Increase the testing time.
- and —
- Maximize the material in contact with the window.

The precision of analysis for small parts measurements is reduced, as the signal from smaller samples is less than for samples that completely cover the window. If possible, analyze the largest, flattest side of an irregularly shaped object.



GO TO

- See [A4. Typical Test Procedure](#) for description of a Test sequence.
- See “[Delta User Interface Guide](#)” (PN 103202) for a complete description of the *Innov-X* application’s User Interface.

Introduction to FastID Mode (All Models)



FastID mode is designed to quickly identify an alloy. It uses an empirical calibration method known as a “type” calibration. It is most useful where the number of alloys to test is small and well known.

FastID is best suited for Positive Material Identification (PMI) and QA/QC applications.

For example, where alloy producers or fabricators handle materials that may be very similar or where maximum user simplicity is a primary concern.

This mode offers:

- Simplified results (grade name only or less information on tramp elements).
- A narrow, customized grade library base upon stored spectral fingerprint reference standards.
- **Full chemical analysis based on reference standard assays.**
- Results which are the best combination of SPEED and ACCURACY.
 - A Grade and Chemistry result in as little as one to two seconds.
- User selectable match criteria settings.
- Expansion of up to 500 additional alloy grades and assays (alloy chemistries) — password protected.
- Multiple independent grade libraries. You choose to search one or more libraries.
- All libraries are editable

How FastID works:

Prerequisite: The operator creates a “custom FastID fingerprint” library by testing an array of reference standards. This list spans the number of alloys for which he is interested.

1. Delta’s XRF process allows a test sample to create a spectral fingerprint.
2. Analyzer compares this spectral fingerprint to entries from library of many certified spectra, the “custom FastID fingerprint” library.
3. Analyzer finds the best spectral match to the sample spectra: thus identifying and reporting the matching alloy grade.
4. If concentration data has been entered for the standards, the instrument then calculates the sample’s **chemistry**.

The reported chemistry data are an extrapolation from standard intensity data stored in the customer -generated fingerprint library.

The user gets a real time grade match and a precise report of the chemical composition of the sample.



NOTE

— Because FastID mode performs a spectral match to a library of reference standards, it is important that before testing, a “likely” stored reference standard be in the customer-created FastID fingerprint library.

Introduction to Pass/Fail Mode (All Models)



Pass/Fail mode is designed for high-throughput alloy sorting and quality control.

Mode Features

- All test samples are sorted by comparison to an operator-selected reference standard.
- Results are displayed as a *PASS* or a *FAIL*, depending on whether they match the reference standard.
- Pass/Fail criteria may be based on:
 - “quality of fit” to the selected spectral fingerprint
 - or —
 - elemental chemistry.
- Pass/Fail ranges may be implemented for one or more elements.
- This mode offers a full range of options from the simple sorting of mixed loads in a recycling facility to QC on specific element(s) of the most complex superalloys.

Pass/Fail mode has two options: *Fingerprint and Chemistry*.

1. Fingerprint Option

Select this method when the goal is to determine whether or not test samples are a **specific grade**.

Fingerprint Pass/Fail and FastID use the same method to determine a match.

Data from analyzed samples are compared to the reference standard fingerprint.

If the differences between the fingerprints are small enough, the sample is judged to be of the same grade as the reference sample.

This method requires:

Only that the library contains a valid fingerprint for the reference standard.

2. Chemistry Option

Select this method to determine whether the chemistries for specific elements fall **within specified min/max grade specifications**.

Chemistry pass/fail process is:

1. Analyzer uses the fingerprint method to determine whether the sample matches the reference sample.
If it does not, it automatically fails.
2. If Step 1 has a match, the alloy chemistry is calculated from assays stored for the standard fingerprint.
3. The calculated chemistry for each element is compared to the values stored in a *Grade Table*.

In order for a sample to pass, all the chemistries must be within “n” standard deviations of the min and max values specified in the grade table.

Number “n” is specified by the user.

This method requires three items:

(1) a valid fingerprint, (2) assays for that fingerprint, and (3) Min/Max values saved in the library.

C6. Mining Modes

There are three Mining modes

MINING

Use with: **ALL models**



Mining

Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, As, Pb,
Bi, Zr, Mo, Ag, Cd, Sn, Sb

(elements may be customized on request)

Fundamental Parameter based calibration for **ore grading and percent-level** analysis of **process bulk** samples.

Suitable for measurement of percentage level analyte concentrations, 0.5% and greater.

2 BEAM MINING

Use with: **Premier and Standard Only**



Mining

Mg, Al, Si, P, S, Cl, K, Ca,
Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, As, Pb,
Bi, Zr, Mo, Ag, Cd, Sn, Sb

(elements may be customized on request)

Mining mode optimized for **SDD based systems** to enhance **SPEED** and **LOD** for light elements

CAR Catalyst

Use with: **All Models**

Mode ICON
T.B.A.

Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Hf,
Ta, Re, Pb, Bi, Zr, Nb, Mo, Ag, Sn, Sb

PLUS Rh, Pt, Pd

Accurate analysis of **bulk recycled catalyst** materials

The Mining modes/types are:

Mining mode - Classic (PiN detector based) type

2 Beam Mining mode - Standard (SDD detector based) type

Beam Mining mode - Premium (SDD detector based) type

Car Catalyst mode - All types

These modes utilize a Fundamental Parameters algorithm which automatically corrects for inter-element results.

The units can analyze:

- *in situ* (directly on the ground),
- prepared soil samples (in sample cups)
- bagged samples

Best Practices

Check Standards

Measure a check standard after each Cal Check, and periodically throughout the day. This confirms that data continues to be as accurate as possible.

The standards provided with Delta instruments are contained in special XRF sample cups. These cups have film windows (through which the soil can be viewed and analyzed) on one side, and solid caps on the other side.

Sample Presentation

in situ testing

In situ testing is performed by pointing the analyzer at the ground. Clear any grass or large rocks away and hold the analyzer with the probe head front flush to the ground. Since dirt can accumulate on the analyzer window, wipe the window clean after each analysis. Ensure the window is not ripped or punctured.

Bagged or prepared sample testing

Analyze prepared samples in a sample cup, through its Mylar window. Place the instrument's measurement window directly over the sample cup with the Mylar side up.

Preparation considerations include:

- Avoid measuring very thin samples, as this can affect results. Prepare samples cups to contain at least 15 mm of packed samples.
- When analyzing bagged samples, ensure that sufficient sample material exists in the bag to completely cover the window with a sample thickness of a minimum of 15 mm.
- When using bags, cheaper bags (having thinner plastic walls) are better than more expensive ones (which have thicker plastic walls).

Optional Accessories

Accessories that can assist in Mining mode testing are:

- A-035: Soil Foot
- 990055: Soil Extension Pole
- A-020-D: Workstation - portable, fully shielded, closed beam test stand for bench-top or remote controlled testing.
- Trimble Xplorer Package

Typical Test Procedure



GO TO

See ["A4. Typical Test Procedure"](#) for description of a Delta Test sequence..

Mining Mode Options

Test length in Mining Mode is user defined.

	GO TO
	Refer to <i>"Delta User Interface Guide, User Factors"</i> for procedure to modify User Factors..

Factors

Mining modes allow you to create your own set of factors, focusing on particular elements of interest or correcting for matrix effects. You can make several different *Factor* tables, allowing analysis of a variety of samples.


Setting Mining User Factors

Example:

A group of samples covering the full concentration range for each element of interest are identified. Each sample is homogenized and split. A portion of each sample is sent to an outside lab for analysis. The other portion is analyzed with the analyzer. For best results ensure that the samples are very well homogenized, and characterized, so the correlation is quite good.

Procedure:

Plot the data. Innov-X data must be on the X-axis, lab values on the y-axis.

	CAUTION
	ENSURE that you use this order: <u>Innov-X data on X-axis and lab data on the Y-axis.</u>

Determine the linear best fit with both the slope and intercept for each element. The slope and intercepts for these graphs are entered directly into the analyzer. In many cases it is sufficient to enter just a correction for the slope as the intercept is almost zero. In others, enter the slope and intercept. You can enter multiple sets of user factors for different applications, or different ore bodies. A group of factors is given a name, and then the factors are entered. The factor set can then be selected by name.

NOTES

C7. Soil Modes

The Delta family currently supports two soil modes, **Soil** and **3 Beam Soil**. Each mode has a possibility of two default element suites, **Environmental** and **Exploration**. Note that both calibration packages cannot reside on one instrument.:

SOIL Environmental

Use with: **All Models**



Soil 3 Beam

K, Ca, S, P, Cl, Ti, Cr, Mn, Fe, Ni, Cu, Zn, Hg, As, Pb, Se, Rb, Sr, Zr, Mo, Ag, Cd, Sn, Sb, Ba

(elements may be customize on request)

Compton Normalization algorithm designed for achieving lowest Limit of Detection (LOD) possible for **SOIL** and **BULK** samples. PowerShot and SmartShot 3-beam modes included.

SOIL Exploration

Use with: **All Models**



Soil 3 Beam

K, Ca, S, P, Cl, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Hg, As, Pb, Bi, Se, Th, Rb, U, Sr, Zr, Mo, Ag, Cd, Sn, Sb

(elements may be customized on request)

Compton Normalization algorithm designed for achieving lowest Limit of Detection (LOD) possible for exploration samples. PowerShot and SmartShot 3-beam modes included.

Soil Mode Beam Selection

SmartShot Beam Mode:


Uses a single incident beam setting (Transition Metals) optimized to deliver ultra fast results with solid LOD performance across the periodic table. SmartShot offers excellent sensitivity in the fastest testing time possible.

PowerShot Beam Mode:


Offers a fully optimized, multi-beam analysis method that provides exceptional LODs for all elements analyzed - heavy metals, transition metals, and light elements. PowerShot can be used to analyze the full element range, or to focus in on a particular element of interest, such as Cr, Cd, Ni, or Cu. Any or all of the following beams conditions may be selected:

- Heavy Elements
- Transition Metals
- LEAP (Light Elements)


Use this procedure to configure beam selections:



From the Mode Setup screen select Test Conditions.



Select the LEAP option



Configure one or more **Powershot(s)**. Note that multiple beams may be chosen. Press Save to return to soil testing.

Best Practices

Check Standards

Measure a check standard after each standardization, and periodically throughout the day, for a minimum of one minute. Elemental concentrations for elements of interest, in the range expected at the site, plus or minus the error on the reading, should be within 20 percent of the standard value. [A2.Soil Testing](#) describes recommended quality assurance considerations in detail.

The standards provided with the analyzer are contained in XRF sample cups. These containers have a film window (through which the soil can be viewed) on one side, and a solid cap on the other side. Always measure samples through the film window.

Sample Preparation

Preparation considerations include:

- Avoid measuring very thin samples, as this can affect results. Prepare samples cups to contain at least 0.5" (usually 4-8 grams) of packed samples.
- When analyzing bagged samples, ensure that sufficient sample material exists in the bag to create a sample thickness of a minimum of 15 mm for a spot size that is larger than the analyzer's measurement window.
- When using bags, cheaper bags (having thinner plastic walls) are better than more expensive ones (which have thicker plastic walls).

C8. Consumer Goods Analysis Modes

There are two consumer goods modes:

RoHS



RoHS

RoHS regulated elements- **Cr, Hg, As, Pb, Br, Cd,**
PLUS Cl, Ti, Fe, Co, Ni, Cu, Zn, Sn, Sb, Ba

Analysis software for measurement of restricted elements in electronics and consumer goods. Auto-compensations built in for metal, polymer, and mixed matrices.

Consumer Products



Consumer

Analysis software designed for CPSIA & Prop 65 testing. **Pb** content displayed as **Pass/Fail** based on regulated limits.

Additional elements Cl, Ti, Cr, Fe, Co, Ni, Cu, Zn, Hg, As, Br, Cd, Sn, Sb, Ba also reported.

Introduction to RoHS Mode

Toxic metals in consumer electronics are the focus of EU regulations that have worldwide ramifications. These new directives currently include:

- *Restriction of Hazardous Substances (RoHS)*
 - Designates maximum allowable levels of Pb, Cd, Cr⁶⁺, Hg and certain Br-containing flame retardants (PBB and PBDE) in new electrical and electronic equipment sold into the EU.

The limits for RoHS elements are:

- <0. 1% Pb, Cr⁶⁺, Hg, Br (as flame retardants, PBB and PBDE)
- <0.01% Cd

The Innov-X analyzer is a screening tool for RoHS Compliance. It is used to:

- Directly analyze the amount of toxic metals in electronics,
- Identify quickly whether a plastic is made of or contains:
 - PVC
 - A brominated flame retardant.

XRF measures *total elemental composition*, regardless of speciation of the element.

Therefore, it reports

- Total chromium including the concentration of hexavalent chromium plus any other forms of Cr.
- Total bromine, however cannot distinguish the type of brominated flame retardant present in analyzed materials.

In order for XRF to be quantitative, samples must be:

- Homogeneous
- Have a certain minimum sample thickness
 - Five (5) mm for polymers and light alloys
 - Fifteen (15) mm for liquid samples
 - One (1) mm for other alloys

If samples are heterogeneous, too thin, or too small, only qualitative screening is possible.

The IEC-ACEA (International Electro-technical Commission - Advisory Committee on Environmental Aspects) recommends XRF screening.

Test Overview

The Delta analyzer controlled by *InnovXPC* application software (in RoHS mode) automatically executes a test sequence to determine:

- Whether a sample is an **alloy**, **polymer**, or **mixed**.
 - “Mixed” indicates heterogeneous samples consisting of both polymer and alloy, such as wires or circuit boards.
- Whether each RoHS element **passes**, **fails**, or is **inconclusive** when compared to a set of stored criteria.
 - These criteria are either those recommended by the IEC, or ones added by the user.

The sequence begins with the instrument utilizing tube settings appropriate for analyzing a polymer sample. The following logic applies:

- If the sample is determined to be a polymer or mixed, the test continues, and a calibration based on a polymer matrix is used.
- If the sample is found to be a metal alloy, the analyzer switches to a secondary test, using an alloy matrix calibration, in order to determine correct alloy concentrations.

Check Standards

Innov-X Systems recommends that a check standard be measured after each Cal Check procedure, and periodically throughout the day.

Two certified standards are provided for verification.

- At least one standard should be measured for a minimum of two minutes.
- Concentrations for target elements (plus or minus the error on the reading) should be within 20% of the standard value.
- Standards provided are contained in XRF sample cups with a Mylar window (through which the plastic pellets can be viewed) on one side, and a solid cap on the other side.
- Samples should be measured in the sample cup, through the Mylar window.

Sample Presentation

Since many pieces of plastic analyzed for ROHS compliance are very small, take care to measure them in a safe and accurate manner. See the IEC-ACEA recommendations for minimum thickness of test samples.

IEC Quantitative Screening Requirements

RoHS requirements are derived from the *“Directive 2002/95/EC of the European Parliament and of the Council of the European Union on the restriction of the use of certain hazardous substances in electrical and electronic equipment.”*

Dated 27 January 2003.

Important Current Issues



- At this User Manual's release date (May, 2010), the IEC requirements (including limits and exemptions) have **not been formally accepted**. A timetable for acceptance has not been established.
- Users must be aware that the information in Figure 1 concerning RoHS screening limits has been extracted from **proposed/draft** IEC-ACEA documentation.
- Innov-X strongly advises users to have their own compliance departments determine the **current status** of the requirements that they must meet.

Elemental Range/Limits for RoHS Compliance

—RoHS— Elements	Polymer Materials				
	PASS	Lower Limit	Inconclusive	Upper Limit	FAIL
Cd	P	$\leq(70-3s)$	$< X <$	$(130 +3s)\leq$	F
Pb	P	$\leq(700-3s)$	$< X <$	$(1300+3s)\leq$	F
Hg	P	$\leq(700-3s)$	$< X <$	$(1300+3s)\leq$	F
Br	P	$\leq(300-3s)<$	X		
Cr	P	$\leq(700-3s)<$	X		
	Metallic Materials				
	P	Lower Limit	Inconclusive	Upper Limit	FAIL
Cd	P	$\leq(70-3s)$	$< X <$	$(130 +3s)\leq$	F
Pb	P	$\leq(700-3s)$	$< X <$	$(1300+3s)\leq$	F
Hg	P	$\leq(700-3s)$	$< X <$	$(1300+3s)\leq$	F
Br			N/A		
Cr	P	$\leq(700-3s)<$	X		
	Electronics				
	P	Lower Limit	Inconclusive	Upper Limit	FAIL
Cd	P	LOD	$< X <$	$(150 +3s)\leq$	F
Pb	P	$\leq(500-3s)$	$< X <$	$(1500+3s)\leq$	F
Hg	P	$\leq(500-3s)$	$< X <$	$(1500+3s)\leq$	F
Br	P	$\leq(250-3s)<$	X		
Cr	P	$\leq(500-3s)<$	X		

Figure 1: Proposed Screening Limits for RoHS Elements

Grade Definitions for Screening

Grade	Proposed Screening Criteria
PASS	Results for ALL elements are lower than the lower limits shown in Figure 1.
FAIL	Result for ANY element higher than the higher limits shown in Figure 1.
INCONCLUSIVE	Result of the quantitative analysis, for any of the elements Hg, Pb, or Cd, is in the region defined as intermediate, OR if the result of the elements BR and Cr is higher than the higher limits shown in Figure 1, the analysis is inconclusive. Additional investigation must be performed.



GO TO

- See [A4. Typical Test Procedure](#) for description of a Test sequence.
- See "[Delta User Interface Guide](#)" (PN 103202) for a complete description of the *Innov-X* application's User Interface.

Introduction to Consumer Products Mode

Background

This mode is dedicated to testing items for Lead (**Pb**) content.

The result output is **Pass/Fail** based on the regulated limits that are specified in:

- [CPSIA](#) (the Consumer Products Safety Improvement Act of 2008)
- [Prop 65](#) - refers to California Proposition 65 enacted in 1986

Additional elements that may be reported are:

Cl, Ti, Cr, Fe, Co, Ni, Cu, Zn, Hg, As, Br, Cd, Sn, Sb, Ba

Regulatory limits vary with the governing body, state-to-state, and country-to-country. European Union nations generally use the RoHS limits and testing practices.

The regulatory limits for Pb are:

- CSPIA: 300ppm currently, but going to 100ppm
- RoHS: see Table 1.0 on Page 70



GO TO

- See [A4. Typical Test Procedure](#) for description of a Test sequence.
- See "[Delta User Interface Guide](#)" (PN 103202) for a complete description of the *Innov-X* application's User Interface.

A1. Overview: X-Ray Fluorescence (XRF) Spectrometry

Basic Theory

Although most commonly known for diagnostic applications in the medical field, x-rays are the basis of many powerful analytical measurement techniques, including X-ray Fluorescence (XRF) Spectrometry.

XRF Spectrometry determines the elemental composition of a material. This method identifies elements in a substance and quantifies the amount present of those elements. An element is defined by its characteristic X-ray emission wavelength (λ) or energy (E). The amount of an element present is determined by measuring the intensity of its characteristic line.

All atoms have a fixed number of electrons (negatively charged particles) arranged in orbitals around their nucleus. The number of electrons in a given atom is equal to the number of protons (positively charged particles) in the nucleus. In the classical Periodic Table of Elements, the Atomic Number is specified by the number of protons. Each Atomic Number is assigned an elemental name, such as Iron (Fe), with Atomic Number 26.

XRF Spectrometry typically utilizes activity in the first three electron orbitals, the K, L, and M lines, where K is closest to the nucleus. Each electron orbital corresponds to a specific and different energy level for a given element.

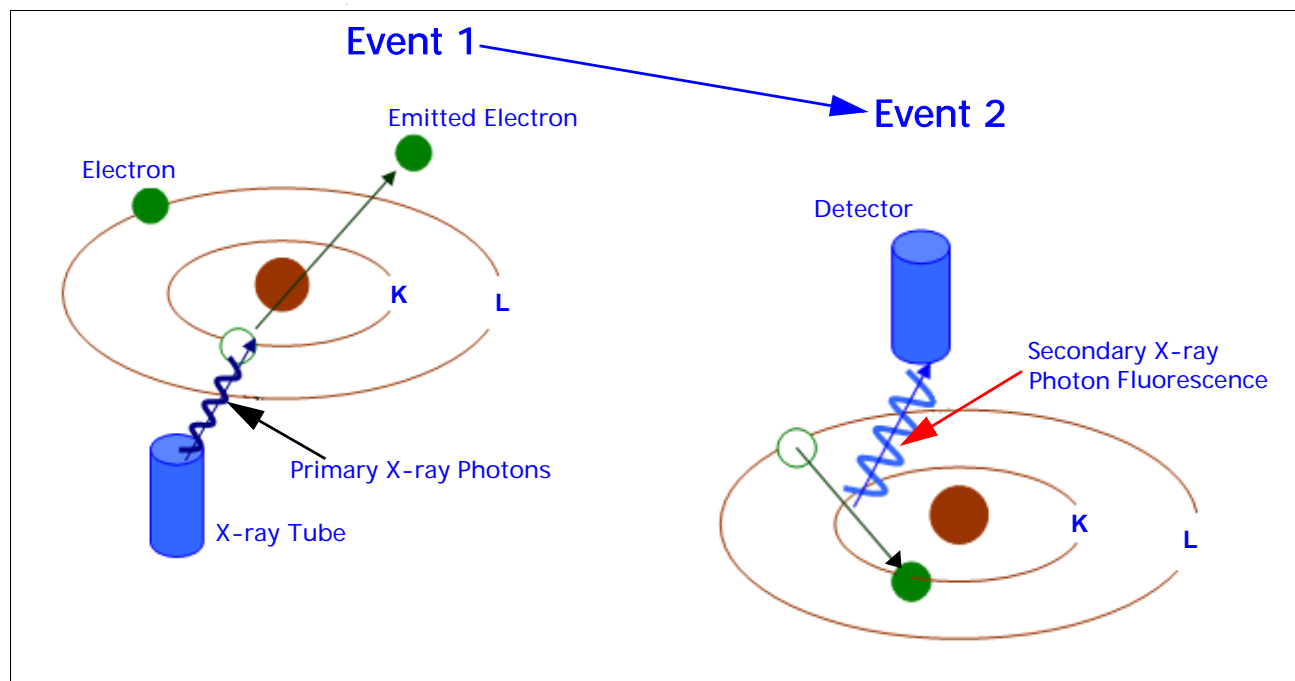
In XRF Spectrometry, high-energy primary X-ray photons are emitted from a source (X-ray tube or *radioisotope*) and strike the sample. The primary photons from the X-ray source have enough energy to knock electrons out of the innermost, K or L, orbitals. When this occurs, the atoms become unstable ions. Electrons seek stability; therefore, an electron from an outer orbital, L or M, moves into the newly vacant space at the inner orbital. As the electron from the outer orbital moves into the inner orbital space, it emits an energy known as a secondary X-ray photon.

This phenomenon is called **fluorescence**.

The secondary X-ray produced is characteristic of a specific element.

The energy (E) of the emitted fluorescent X-ray photon is determined by the difference in energies between the initial and final orbitals of the individual transitions.

This is described by the formula $E=hc/\lambda$ where **h** is Planck's constant; **c** is the velocity of light; and λ is the characteristic wavelength of the photon.

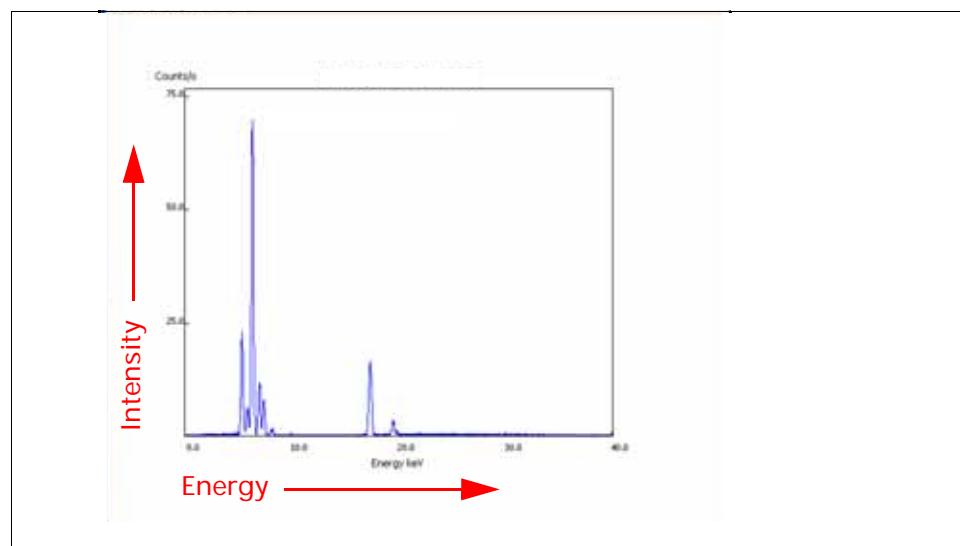


Creating a Secondary X-ray - Photon Fluorescence

Wavelengths are inversely proportional to the energies; they are characteristic for each element.

For example, the Ka energy for Iron (Fe) is about 6.4keV. The number of element-specific characteristic X-rays produced in a sample over a given period of time, or the intensity, is measured. This determines the quantity of a given element in that sample.

Typical spectra for EDXRF Spectrometry appear as a plot of Energy (E) versus the Intensity (I).



Typical Spectrum Plot: Energy vs. Intensity

History

Timeline for XRF Spectrometry

- Wilhelm Roentgen discovered X-rays in 1895.
- Henry Moseley first published methods for identifying and quantifying elements using XRF in 1913.
- XRF research and development continued, especially during WWII.
 - Critical developments in the aircraft, automotive, steel, and other metals industries increased the need to identify alloys *quickly* and *reliably*.
- The first commercial XRF Spectrometers became available in the early 1950's. These systems were based on **Wavelength Dispersive (WD) XRF** technology.
 - The characteristic **wavelength** of an element was measured **one element at a time**.
 - WDXRF systems were useful for elemental analyses, however, the equipment had the following properties:
 - Large size
 - High initial cost
 - Required highly skilled operators to use and maintain them.
- In the late 1960's, **Energy Dispersive (ED) XRF** technology emerged as a viable commercial choice:
 - EDXRF measured the characteristic **energy** of an element.
 - Improvements in solid state detectors offered better energy resolution of the signal.
 - Had potential to collect and display information on **all** of the elements in a sample at the **same time**.
- Many of the early EDXRF systems used radioisotopes for excitation. They had the following properties:
 - Required changing sources to determine all the elements of interest.
 - Did not easily resolve multiple elements in a single analytical run.
- The current state-of-the-art in EDXRF is the result of
 - Advancements in technology (particularly X-ray tubes, solid-state components, electronics, computers, software)
 - Application of the technology by instrument manufacturers, research scientists, engineers, and industrial users.
- Now a mature technology, XRF Spectrometry is routinely used for R&D, QC, production support, and regulatory compliance.

Elemental Analysis

Investigators involved with elemental analysis generally have two working instrument techniques — *Wet Chemistry* and *XRF Spectrometry*. They are compared operationally as follows.

Wet Chemistry

Important considerations are:

- Instrument techniques are *time-consuming*.
 - Often takes twenty minutes to several hours for specimen preparation and analysis.
- Specimen is *destroyed*.
- Often necessary to employ *concentrated acids* or other *hazardous materials*.
- Requires *disposal* of waste streams generated during the analytical process.
- Relatively *high cost* per sample.

However, wet chemistry instrument techniques are necessary if the primary measurement requirement involves elemental concentrations in the PPB (or lower) range

XRF Spectrometry

Important considerations are:

- Easily and quickly identifies and quantifies elements over a wide dynamic concentration range, from PPM levels up to virtually 100% by weight.
- Does *not destroy* the sample.
- Overall sample turnaround time is *fast*.
 - Requires little, if any, specimen preparation.
 - Often results are available within seconds, minutes for some details.
- Relatively *low cost* per sample

Interferences

All elemental analysis techniques experience chemical and physical interferences. They must be corrected or compensated for in order to achieve adequate analytical results.

WET CHEMISTRY ISSUES

Most suffer from interferences that are corrected only by extensive and complex specimen preparation techniques.

XRF SPECTROMETRY ISSUES

The primary interference is from other specific elements in a substance that can influence (matrix effects) the analysis of the target element(s) of interest.

However, this interference style is well known and documented.

Both types of analyzer techniques benefit from (a) instrumentation advancements, and (b) mathematical corrections in the system's software.

In certain cases, the *geometry* of the sample can effect XRF analysis.

- This is compensated for by:
 - Grinding or polishing the sample
 - Pressing a pellet
 - Making glass beads

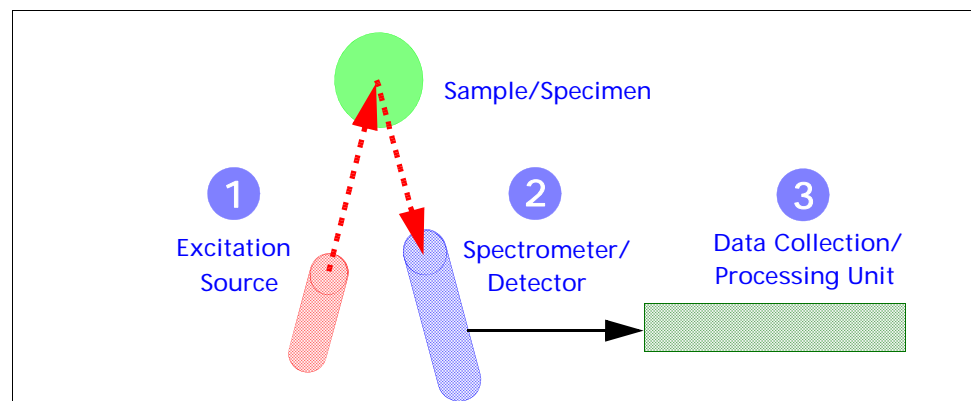
Quantitative analysis

XRF Spectrometry supporting quantitative analysis typically employs one of two software applications:

- **Empirical Methods**
Uses calibration curves derived from standards similar in property to the target unknown sample.
- **Fundamental Parameters (FP)**
FP is frequently preferred because it allows elemental analysis to be performed *without* standards or calibration curves. The analyst can use the system immediately. Modern computers support this *no-standard* mathematical analysis, FP, accompanied by stored libraries of known materials. These systems quickly determine not only the elemental composition of an unknown material, but even identify the unknown material itself.

EDXRF Spectrometers

An EDXRF instrument typically has three major subsystems:



Three Subsystems of EDXRF Analyzer

EDXRF analyzers are mechanically very simple; there are *no moving parts* in the excitation and detection subsystems. However, a bench-top analyzer can have moving parts.

When compared to WDXRF systems, EDXRF systems exhibit the following attributes:

- Ease of use
- Rapid analysis time
- Lower initial purchase price
- Substantially lower long-term maintenance costs

EDXRF analysis equipment is useful for many applications, including:

- Environmental analysis
- RoHS/WEEE compliance
- Scrap alloy sorting
- Forensic science
- Archaeometry

NOTES

A2. Soil Testing

This appendix explains usage of all of the company's hand-held portable analyzers with the *Soil* or *3 Beam Soil* mode option installed.

This document offers instructions/procedures and regulations, as well as useful reference material, regarding:

- Portable XRF equipment usage in accordance with accepted methods.
- Basic overview of the technique of x-ray fluorescence (XRF).
- Appropriate data quality assurance protocols.
- Sample preparation steps for operators analyzing prepared soil samples.
- Tables of certified values for selected standards.

If LEAP mode is enabled, refer to configuration help in [PN 103202](#).

Section 1: Commonly Accepted Methods for Field Portable XRF

A commonly accepted method is shown: *Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment*.

Features of this method are:

- It is a field screening method, for analysis of *in-situ* or bagged samples.
- The method provides basic quality assurance methods, including calibration verification, determination of instrument precision, accuracy and limit of detection.
- The method recognizes that some XRF instruments do not require site-specific calibrations by the operator, that is, the factory calibration provides appropriate data quality.
- The method recommends that a minimum of 5-10% of samples tested by XRF be confirmed by an outside laboratory, using a total-digestion EPA analytical reference method.

The purpose of this method is **NOT** to replace laboratory analysis.

There are two primary sources of error in assessing a site for metal concentration: *Analytical error* and *Sampling error*.

ANALYTICAL ERROR

The error in the analysis of any one sample by whatever technique is used, for example XRF, ICP, or AA.

SAMPLING ERROR

This arises when **too few** samples are collected and tested.

In this case an incomplete picture of the extent of metals contamination may be obtained. Although any one sample may be analyzed with very high analytical accuracy, measuring too few samples may result in contamination plumes being mis-judged in size, or depth into the soil. In extreme cases contamination can be missed entirely.

Methods have been developed to reduce Sampling Errors by **increasing** the number of samples measured. In general, a large number of screening-level measurements provide a better characterization of contamination than a small number of measurements produced by sample removal and analytical analysis. A large number of in-situ samples provide detailed data on contamination profiles, depth (provided surface soil is moved aside), and approximate contamination levels. Portable XRF can provide results with a high degree of analytical accuracy on any given sample.

Section 2: Overview of Field Usage:

Field portable XRF is generally used in three ways to test for metals in soil:

IN-SITU SOIL TESTING:

The XRF is placed directly onto the ground for soil testing. Operators remove any plant growth and foreign objects so that the analyzer probe is flush to the soil.

BAGGED SOIL SAMPLE TESTING:

A soil sample is collected in a thin plastic bag (i.e. a *Baggie*) and testing occurs directly through the Baggie. Except for a few elements - namely Cr, V and Ba - testing through the thin plastic bag has little effect on the test result. However, results for Cr, V and Ba will be lower by 20-30%.

PREPARED SOIL SAMPLE TESTING:

Prepared sample testing assures the operator of the maximum possible accuracy. Prepared sample tests require a sample to be collected, dried if necessary, sieved and ground into a powder. The prepared sample is then placed into a baggie or XRF cup for analysis.

Sample prep procedures are provided on Section 8: Sample Prep Procedures and Testing Protocols on page 130.

ALL analytical methods require a *uniform, homogenous* sample for the best results. XRF is no different!

The methods generally used, namely In-situ and bagged sample testing, are considered *field-screening methods*. Although a field-screening method, in-situ testing is a valuable technique because it generates a great deal of data very quickly. Prepared soil samples generally offer the best accuracy, albeit with several minutes of sample preparation required per sample.

Subsection 2-A: Data Quality Objectives

The objective of testing is generally to determine the mixture of in-situ versus prepared sample testing. It is important to understand your data quality objectives (DQO) in order to determine the appropriate mix of field screening and prepared sample testing.

In-situ testing usually provides only *screening-level data quality*.

This is because analytical testing always requires a uniform, homogeneous sample matrix. A laboratory achieves this by digesting the sample into a hot acid before analysis. Testing directly on the ground does not ensure that uniformity is met.

Preparing a sample provides a uniform sample and likely better analytical data quality, although several minutes of testing time is required.

Most portable XRF operators use a mixture of in-situ and prepared sample testing. The exact mixture of in-situ and prepared sample testing depends upon the goals of the soil testing. The examples below serve as guidelines.

Example 1: Initial site investigation to provide detailed contamination data with efficient use of laboratory analysis costs.

PROBLEM:

The site needs to be assessed for metals contamination. Little information is available about what metals are present, likely contamination levels or geographic profile of contamination.

The goal of testing is to determine what metals are present at what levels, both in area and in depth into soil. Additionally, testing will locate possible contamination plumes and/or possible sources of contamination.

RECOMMENDED TESTING PLAN:

This example uses predominately in-situ testing. The analyst will perform in-situ testing, and gather samples into plastic bags for XRF analysis. A testing grid should be established in two or three dimensions, every several feet. XRF tests can be taken at each location or bagged samples can be collected from each location for later analysis. The in-situ data for each element analyzed may be plotted in a 2-dimensional grid (X, Y coordinates versus elemental concentration) to profile a site. These concentration profiles are ideal for showing contamination patterns, boundaries and plumes. Combining this data with historical use data from the site often allows the operator to deduce sources of contamination. Obtaining this level of geographic data with purely laboratory analysis would produce excessive analytical costs.

Prepared sample analysis should also be done to confirm the regions where in-situ data indicates low or non-detected levels of metal contaminant. There is little need to prepare areas where in-situ testing indicates high concentration levels.

Innov-X recommends this procedure:

For locations where in-situ tests indicate low or non-detected concentrations, calculate the total number of in-situ tests, collect 5% of this number of tests from the various locations. Prepare these samples according to instructions on "Section 8: Sample Prep Procedures and Testing Protocols on page 130." Use these prepared samples to confirm the findings of the in-situ testing. Send a subset of these prepared samples to a laboratory for confirmatory results.

COST JUSTIFICATION:

To adequately characterize a site may require 100-200 samples/acre to be sure the contaminated areas are firmly established. This work may be done with in-situ testing to generate laboratory savings of \$5,000 - \$10,000/acre depending upon the number of elements being analyzed. The cost reduction in off-site analysis often justifies the price of the XRF.

Example 2: Monitor remediation efforts and assure site meets clearance levels before contractors leave the site.

GOAL:

Minimize remediation costs by only treating contaminated soil, and obtain immediate verification that various site locations meet clearance objectives.

RECOMMENDED TESTING PLAN:

This type of project uses a lot of both in-situ and prepared sample testing. Use in-situ testing to thoroughly delineate contamination regions in both area and depth. To determine depth profiles, test surface soil, remove at least 1-2', and retest. Repeat this step as necessary to profile contamination depth to guide remediation activities (XRF is a surface technique and only analyzes the first few mm of soil sample). As part of clearance, collect several samples from *cleared* area. Prepare samples according to "[Section 8: Sample Prep Procedures and Testing Protocols](#)" on page 92. Test with portable XRF.

If XRF indicates that concentration levels are:

- In excess of clearance requirements, then continue remediation efforts.
- Below clearance requirements, then discontinue remediation efforts, and send a subset of the samples to an analytical laboratory to confirm results. Most operators safely assume that the cleanup requirements have been met for the elements in question, but await final analysis from the laboratory.

If XRF lists concentration levels as non-detected, but the detection level reported exceeds clearance requirements, send samples to a laboratory for final results.

Cost Justification: In-situ results are used to guide remediation efforts, in order to obtain maximum efficiency. Efficiency is produced because contamination boundaries are firmly established, thus avoiding remediation efforts with *clean* soil. Prepared sample testing is used to assure that clearance requirements are met on-site in near real-time (pending laboratory confirmation). Costs savings are generated by avoiding clearance failures. The contractors can leave the site earlier and will not be called back to the site for additional cleanup.

IMPORTANT NOTE:

Never clear a site based solely on in-situ testing. Always use well-prepared samples to make a clearance decision.

Example 3: Minimize volume of hazardous waste for treatment or disposal.

GOAL:

For some cleanup projects, the cost of soil disposal in a hazardous waste landfill is much greater than disposal in a standard landfill. Testing soil samples with XRF may minimize the amount of *clean* soil that is inadvertently shipped to a hazardous-waste landfill.

RECOMMENDED TESTING PLAN:

This example is almost entirely based on prepared sample testing. Representative samples are removed from the soil being hauled to landfill. Obtaining an accurate analysis of the samples is crucial for making a hazardous versus non-hazardous determination. For this reason, prepared sample testing is strongly recommended.

IMPORTANT NOTE:

These types of samples are subject to Toxicity Characteristic Leaching Procedures (TCLP) for the landfill determination. In general, 20 times the XRF result should be less than the allowable limit for the metal in question. Please contact Innov-X Systems for more details on testing samples versus TCLP regulatory requirements.

Section 3: Quality Assurance

Quality assurance is detailed for both the proper use of the analyzer and for verifying the data quality of in-situ testing. All operators should perform the QC procedure, regardless of their data quality objectives. There must be strict requirements about quality assurance. Additionally, Innov-X recommends that operators verify the data quality of in-situ test results, if they are using in-situ data to guide their reporting or remediation decisions. Procedures are listed below:

Proper verification of instrument operation

Quality assurance here consists of testing known standards to verify calibration, as well as testing blank standards to determine limits of detection and to check for sample cross-contamination or instrument contamination. We recommend a detailed procedure, which is provided here in abbreviated form.

Components of instrument QC:

- An energy calibration check sample at least twice daily
- An instrument blank for every 20 environmental samples
- A method blank for every 20 *prepared* samples
- A calibration verification check sample for every 20 samples
- A precision sample at least one per day
- A confirmatory sample for every 10 environmental samples

Energy Calibration Check: The Innov-X analyzer performs this automatically; this is the purpose of the standardization check when the analyzer is started. The software does not allow the analyzer to be used if the standardization is not completed.

Instrument Blank: The operator should use the SiO₂ (silicon dioxide) blank provided with the analyzer. The purpose of this test is to verify there is no contamination on the analyzer window or other component that is *seen* by the x-rays. We recommend an instrument blank at least once per day, preferably every 20 samples. For either in-situ or prepared-sample testing, the operator should just test the SiO₂ blank to be sure there are no reported contaminant metals.

Method Blank: The purpose of the method blank is to verify that cross-contamination is not introduced into samples during sample preparation. We recommend following the sample preparation procedures with clean SiO₂ once every 20 prepared samples. This QC step is not required if the operator is not preparing samples.

Calibration Verification: Innov-X provides standard reference samples for calibration check by operator. The operator should perform a two minute test on a standard. The difference between the XRF result for an element and the value of the standard should be 20% or less. Calibration Verification should be performed upon instrument startup and periodically during testing.



NOTE

Innov-X recommends a calibration check every 4 hours. Some users make a calibration check every 20 samples. Reference standards are generally applicable for Pb, As, Cr, Cu, Zn. Innov-X provides additional reference standards for Priority Pollutant metals including Cd, Se, Ag, Hg, Ag, Ba, Sn, Sb, and Ni.

Precision Verification: It is good practice to make a minimum of one precision sample run per day by conducting from 7 to 10 replicate measurements of the sample. The precision is assessed by calculating a relative standard deviation (RSD) of the replicate measurements for the analyte. The RSD values should be less than 20 percent for most analytes, except chromium, for which the value should be less than 30 percent.

Confirmatory Sample: It is recommended that one confirmatory sample is run for every 10 samples collected. It is good practice to have confirmatory samples collected from the same sample material that is analyzed on site, but are sent to an off-site laboratory for formal analysis. The purpose of a confirmatory sample is to judge the accuracy of the data obtained by analysis on site and to allow corrections, if necessary."

Important Notes about confirmatory samples:

Innov-X always recommends that customers compare prepared-sample results to laboratory results. To do this, collect and prepare a sample following the protocols shown on "[Section 8: Sample Prep Procedures and Testing Protocols](#)" on page 92. Take a subsample and submit to the laboratory for analysis. The single largest error in XRF analysis is lack of sample preparation. For the best comparison, always use prepared samples.

Determining data quality of in-situ testing:

For operators relying extensively on in-situ testing, it is important to determine the data quality of this testing at a given site. *This protocol is not intended for every sample, but rather for a small percentage of samples considered representative of the site.* If the operator can demonstrate that quantitative data is achieved with little or no sample preparation, then the site characterization will be completed much more quickly but correctly.

For example, an operator may be able to demonstrate that the XRF result changes considerably when samples are passed through a 2 mm sieve, but that XRF results do NOT change appreciably upon finer sieving. In this case, the operator can conclude that good XRF data is achievable with only 2 mm sieving. Sieving only to this level requires far less time than a more robust sample preparation.

A protocol to determine the *appropriate level of sample preparation* is the following:

1. Delineate a region of soil approximately 4" x 4".
2. Perform several in-situ tests in this area, or collect the top (approximately) quarter inch of soil from this region, bag the soil, test through the bag. In either case, average the results.
3. If you did not bag the in-situ test sample, collect the top (approximately) quarter inch of soil from this region and sieve through the 2 mm sieve provided. Otherwise sieve the bagged sample used for the in-situ test. Thoroughly mix the sieved sample, and place some of the sieved material into an XRF cup, and perform a test of this sample.
4. If the results of this prepared sample differ by:
 - Less than 20% with the average in-situ result, this indicates the soil in this region is reasonably homogeneous. The data quality in this case is probably at the semi-quantitative level, rather than just screening data.
 - More than 20%, this indicates the soil is not very homogeneous, and there are serious particle size effects affecting your in-situ measurements.
In this case, sieve the sample through the ~250 μ m sieve. Mix this sample and place a sub-sample into an XRF cup for testing. If this result differs from the previous by less than 20% then this indicates that at a minimum the 2 mm sieving is necessary to achieve higher data quality.
If this result differs by more than 20% from the sample sieved through 2 mm, then particle size effects are still affecting the XRF result. In this case samples should be sieved through 125 μ m to assure data quality at the quantitative level.

Section 4: Calibration for Innov-X Portable XRF

The Innov-X analyzer can run *three* different calibration methods, described below.

COMPTON NORMALIZATION:

In nearly all cases, customers use the Compton Normalization method. This method (recognized in EPA 6200) offers speed, ease of use, and generally good accuracy for concentration ranges from the ppm level up to 2-3% concentrations. As most field-testing is seeking to remediate or locate environmental contaminants, the upper limit of the calibration (2-3%) is generally not a limitation.

FUNDAMENTAL PARAMETERS (FP):

If customers do require a calibration up to 100% concentration (i.e. a pure element), then Innov-X recommends they also include the Fundamental Parameters (FP) software module with the analyzer. The FP module may be added at time of purchase or as an upgrade at any later date.



NOTE

In general, customers do not need to calibrate Innov-X analyzers for soil testing. The analyzer is delivered with a factory calibration, generally based upon the Compton Normalization (CN) method. The CN method has been proven over the past several years to provide a robust calibration generally independent of site-specific soil matrix chemistry. All customers should follow the QC procedure described in Section 3, which includes a check of the calibration.

EMPIRICAL CALIBRATION:

In this case, customers run standards to generate calibration curves for various elements in specific soil matrices. With a well-prepared sample, the empirical method generally yields the most accurate result. In our experience, the accuracy gains going *from* Compton Normalization *to* Empirical Mode are small and not worth the extra effort in setting up calibration curves.

The empirical calibration module is an optional software package, available for an upgrade fee at the time of purchase, or as an upgrade at any later date.



CAUTION

The *greatest source of error* for in-field XRF analysis of soil is *lack of adequate sample preparation*, thus there is little gained in developing a sophisticated empirical calibration if the operator does not grind and homogenize the all measured samples.

Calibration Requirements:

The concentration of an element in a soil sample is well-described by the formula:

$$w_i = \frac{k_i}{M(Z, i)} I_i$$

where:

k_i = calibration constant for element i

ω_i = concentration of element i - the quantity being measured

I_i = measured x-ray intensity from element i

$M(Z, I)$ = Soil matrix value

The factory calibration determines the value of the calibration constants k_i for each element, and a typical value $M(Z, I)$. The calibration method - either CN, fundamental parameters, or empirical - performs the necessary corrections to the value $M(Z, I)$ that are important for the site-specific soil chemistry. The XRF analyzer uses the measured intensity of each element's fluorescence from the sample, and the calibration data, to produce elemental concentrations.

COMPTON NORMALIZATION CALIBRATION:

The Compton Normalization method calibration consists of the analysis of a single, well-characterized standard, such as an SRM or SSCS. The standard data are normalized to the Compton peak. The Compton peak is produced from incoherent backscattering of X-ray radiation from the excitation source and is present in the spectrum of every sample. The matrix affects the way in which source radiation is scattered off the samples. This scatter is directly related to the intensity of the Compton peak. For that reason, normalizing to the Compton peak can reduce problems with matrix effects that vary among samples. Compton normalization is similar to the use of internal standards in analysis for organic analytes.

FUNDAMENTAL PARAMETERS CALIBRATION:

The fundamental parameters (FP) calibration is a *standardless* calibration. Rather than establishing a unit's calibration curve by measuring its response to standards that contain analytes of known concentrations, FP calibration relies on the known physics of the spectrometer's response to pure elements to set the calibration. Built-in mathematical algorithms are used to adjust the calibration for analysis of soil samples and to compensate for the effects of the soil matrix. The FP calibration is performed by the manufacturer, but the analyst can adjust the calibration curves (slope and y-intercept) on the bases of results of analyses of check samples, such as SRMs which are analyzed in the field.

EMPIRICAL CALIBRATION:

The empirical calibration method requires that a number of site-specific calibration standards (SSCS) are used to establish calibration parameters. The instrument response to known analytes is measured and used to create calibration curves. Empirical calibration is effective because the samples used closely match the sample matrix. SSCSs are well-prepared samples collected from the site of interest in which the concentrations of analytes have been determined by inductively coupled plasma (ICP), atomic absorption (AA), or other methods.

The standards should contain all the analytes of interest and interfering analytes. Manufacturers recommend that 10 to 20 calibration samples be used to generate a calibration curve. The empirical method is the least desirable calibration method as it requires that new standards and curves are generated for each site that is analyzed.

Section 5: Effects of Moisture on XRF Results

Sample moisture has two effects on XRF results:

- It alters the soil chemistry, since water is another chemical compound that comprises the soil matrix.
- Moisture impedes the ability to properly prepare samples.

There is a further testing consideration:

- Laboratory results are provided on a *dry weight* basis.

EFFECT ON SOIL CHEMISTRY:

While the presence of significant moisture does impact the soil chemistry, modern XRF analyzers all perform automatic corrections for variations in soil chemistry from site to site. Indeed, such variations are expected, and that is the reason analyzers use Compton Normalization or fundamental parameters, in order to correct for moisture content changes as well as other differences in soil geochemistry.

It is known that moisture content above 20 percent may cause problems, since moisture alters the soil matrix for which the FPXRF has been calibrated. However, the Compton Normalization or fundamental parameters methods are implemented in order to automatically correct results for changes to the soil matrix. Thus, we believe that soil moisture is not a significant effect on accuracy due to effects of soil matrix, except for the *dilution* effect that can cause discrepancies with laboratory results which is described below.

SAMPLE PREPARATION ISSUES:

The inability to adequately prepare a wet sample is, we believe, the single biggest contributor to errors when testing wet samples. It is very difficult to grind or sieve a wet sample. The highest quality XRF results are generally obtained from prepared samples.



CAUTION

If the operator is unwilling to dry the sample to prepare it, comparisons to the laboratory may yield poorer correlation since the samples are not homogeneous.

Laboratory Tests on Dry-Weight Basis:

Laboratories always dry samples prior to analysis. They report percent weight content based upon a dry sample basis. Portable XRF may often be used to analyze wet samples in the field, and results are thus reported that include the moisture content. Thus, with all other factors the same, the laboratory will report results higher than portable XRF. The results are higher by the amount of moisture content in the sample. For example, laboratory results will be 10% higher compared to XRF results, if the sample contained 10% by weight water when it was tested with XRF. Recall, this applies to samples where other possible sources of error are the same or negligible.

Section 6: Comparing XRF Results to Laboratory Results

Innov-X strongly recommends that operators compare prepared sample results to laboratory results. This is because prepared-sample results yield the best possible accuracy with portable XRF. Moreover, the most common source of error is due to non-uniform samples. No analytical technique, including the XRF technique, can properly account for non-uniform sample types.

To perform a comparison between XRF results and laboratory:

1. Collect a sample and prepare it according to the sample preparation guide given in *"Section 8: Sample Prep Procedures and Testing Protocols"* on [page 90](#).
2. Take a sub-sample (5-10 grams) of the fully-prepared sample, place it into an XRF cup and perform at least a one-minute test on that sample.
3. Send the same sample to the laboratory for wet chemistry analysis.
4. Require the laboratory to use a *total-digestion method*. If the laboratory does not, they may not extract all of the elemental metal from the sample. In this case, the lab result will be lower than the XRF result. Incomplete sample digestion is one of the *most common* sources of laboratory error, thus it is very important to request a total digestion method.

Example of Error: The operator collects a bag of sample, performs XRF analysis on one part of the bag, and sends the bag, or part of the bag of sample to a laboratory for analysis. The laboratory reports a very different value than the operator obtained with the XRF.

Problem:

Since the sample is very non-homogeneous, the operator did not obtain a result that was representative of the entire bag of sample. The lab analyzed a different part of the sample and obtained a very different result due to the non-uniformity of the sample. The solution to this problem is, at a minimum, to test several locations in the bag of sample and report the average value. Also note the differences between the tests, as this is indicative of the non-uniformity of the sample. The operator should send entire bag of sample to the lab, and instruct the lab to prepare the sample before removing the sub-sample for lab analysis.

Best Practice:

The operator should homogenize and prepare the entire bag of sample, and then collect a sub-sample for XRF testing. After testing, the *same sample* should be sent to the lab.

Section 7: Common Interferences

Interference occurs when the spectral peak from one element overlaps either partially or completely with the spectral peak of another.

Case 1 Conditions

If the instrument is calibrated for both elements, one causing the interference and one being interfered with, it is generally capable of correctly handling the interference. In this instance, the element being interfered with may be measured with a poorer detection limit or poorer precision, but the analytical results should still be acceptable for field-portable XRF.

Case 2 Conditions

If the XRF is not calibrated for the element causing the interference, then the instrument may report the presence of elements not in the sample, or greatly elevated concentrations of elements in or not in the sample.

Example CASE 1: Lead and arsenic

Lead and arsenic. Most XRFs are calibrated for lead and arsenic. Lead interferes with arsenic (not vice-versa though). The net effect is a worsened detection limit for arsenic, and poorer precision. The XRF handles the correction automatically, but the precision is affected. The loss of precision is also reported by the XRF.

(Refer to Innov-X Applications Sheet: [In-field Analysis of Lead and Arsenic in Soil Using Portable XRF](#) which is now available as [Arsenic and Lead in Soil.PDF](#) for more detail).

Example CASE 2: Bromine

Bromine in the sample, but the XRF is not calibrated for bromine. Bromine, as a fire retardant, is being seen more and more in soil and other sample types. For this reason, Innov-X analyzers include Br in the calibration data. If Br is not calibrated, but is present in the sample, the analyzer will report highly elevated levels of Pb, Hg and As. The levels will depend upon the concentration of Br in the sample.

Interferences between elements can be broadly categorized into two types:

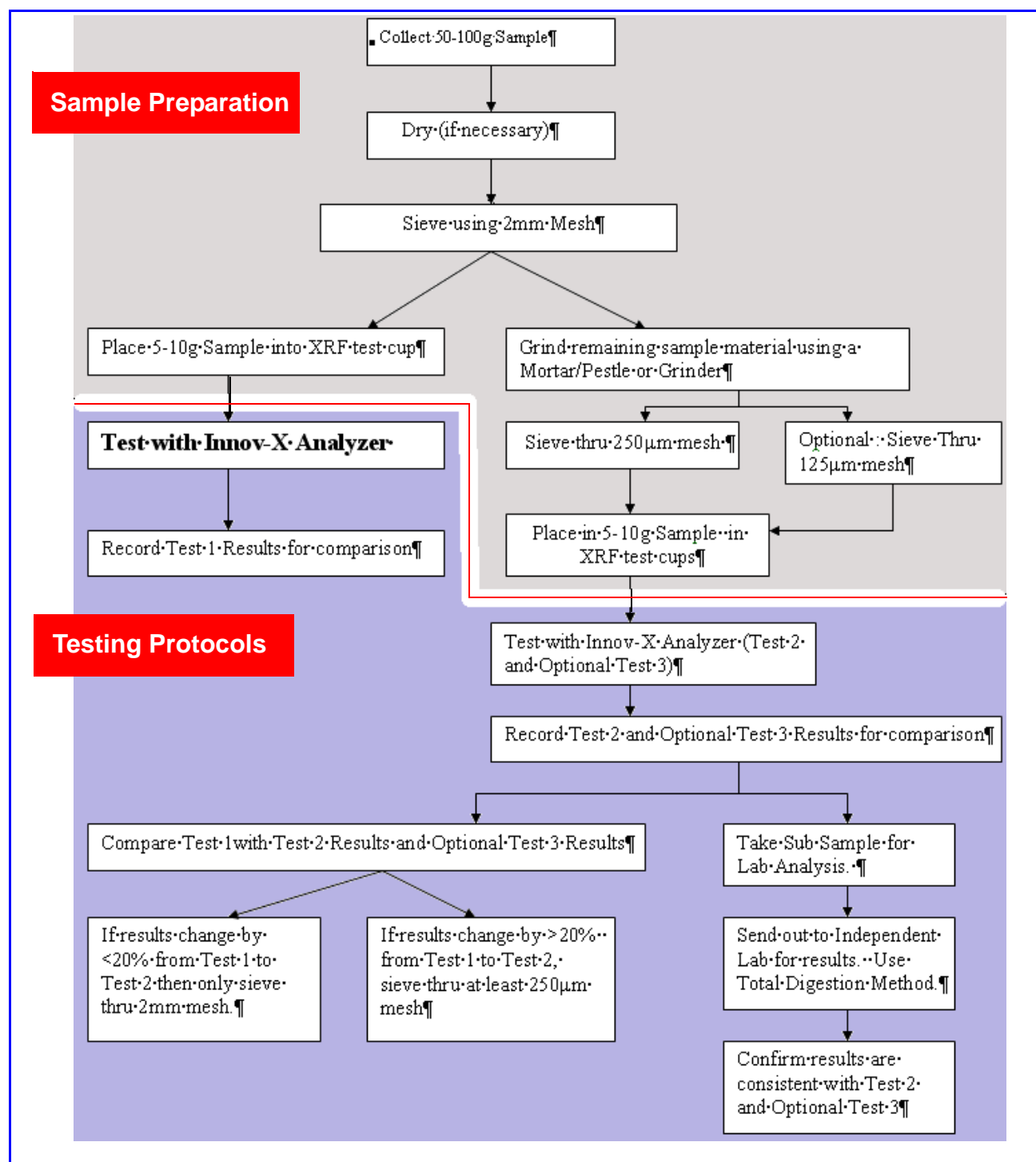
Z, Z-1, Z+1 interferences

Occurs when high levels of an element of atomic number Z are present. This can cause elevated levels of elements with atomic number Z-1 or Z+1. Generally, portable XRFs have good correction methods, so this interference only causes problems with very high levels of the element in question. Example: High concentrations of Fe (Z=26) in excess of 10% may cause elevated levels of Mn or Co (Z=25 or Z=27 respectively).

K/L interferences

Occurs when the L-shell line of one element overlaps with the K-shell spectral line of another element. The most common example is the lead/arsenic interference where the L-alpha line of lead is in nearly the exact same location as the K-alpha line of arsenic.

Section 8: Sample Prep Procedures and Testing Protocols



Section 9: NIST Certificates of Analysis

Innov-X systems provides three NIST standards (subject to change with availability). Each standard's certified values are given in Table 1 and Non-certified values are in Table 2 in the graphics below.

☒ SRM2709

SRM2710

SRM2711




Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Carbon	1.2	Cerium	42
		Cesium	5.3
		Dysprosium	3.5
		Europium	0.9
		Gallium	14
		Gold	0.3
		Hafnium	3.7
		Holmium	0.54
		Iodine	5
		Lanthanum	23
		Molybdenum	2.0
		Neodymium	19
		Rubidium	96
		Samarium	3.8
		Scandium	12
		Thorium	11
		Tungsten	2
		Uranium	3
		Ytterbium	1.6
		Yttrium	18
		Zirconium	160

Table 1. Certified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Aluminum	7.50 ± 0.06	Antimony	7.9 ± 0.6
Calcium	1.89 ± 0.05	Arsenic	17.7 ± 0.8
Iron	3.50 ± 0.11	Barium	968 ± 40
Magnesium	1.51 ± 0.05	Cadmium	0.38 ± 0.01
Phosphorus	0.062 ± 0.005	Chromium	130 ± 4
Potassium	2.03 ± 0.06	Cobalt	13.4 ± 0.7
Silicon	29.66 ± 0.23	Copper	34.6 ± 0.7
Sodium	1.16 ± 0.03	Lead	18.9 ± 0.5
Sulfur	0.089 ± 0.002	Manganese	538 ± 17
Titanium	0.342 ± 0.024	Mercury	1.40 ± 0.08
		Nickel	88 ± 5
		Selenium	1.57 ± 0.08
		Silver	0.41 ± 0.03
		Strontium	231 ± 2
		Thallium	0.74 ± 0.05
		Vanadium	112 ± 5
		Zinc	106 ± 3

SRM2709



SRM2710

SRM2711



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2710

Montana Soil

Highly Elevated Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2710 is a highly contaminated soil that was oven-dried, sieved, radiation sterilized, and blended to achieve a high degree of homogeneity. A unit of SRM 2710 consists of 50 g of the dried material.

The certified elements for SRM 2710 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should NOT be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

Noncertified Values: Noncertified values shown below are provided for information only. An element concentration value is not certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Carbon	3	Bromine	6
		Cerium	57
		Cesium	107
		Chromium	39
		Cobalt	10
		Dysprosium	5.4
		Europium	1
		Gallium	34
		Gold	0.6
		Hafnium	3.2
		Holmium	0.6
		Indium	5.1
		Lanthanum	34
		Molybdenum	19
		Neodymium	23
		Rubidium	120
		Samarium	7.8
		Scandium	8.7
		Strontium	330
		Thallium	1.3
		Thorium	13
		Tungsten	93
		Uranium	25
		Ytterbium	1.3
		Yttrium	23

Table 1. Certified Values

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Aluminum	6.44 ± 0.08	Antimony	38.4 ± 3
Calcium	1.25 ± 0.03	Arsenic	626 ± 38
Iron	3.38 ± 0.10	Barium	707 ± 51
Magnesium	0.853 ± 0.042	Cadmium	21.8 ± 0.2
Manganese	1.01 ± 0.04	Copper	2950 ± 130
Phosphorus	0.106 ± 0.015	Lead	5532 ± 80
Potassium	2.11 ± 0.11	Mercury	32.6 ± 1.8
Silicon	28.97 ± 0.18	Nickel	14.3 ± 1.0
Sodium	1.14 ± 0.06	Silver	35.3 ± 1.5
Sulfur	0.240 ± 0.006	Vanadium	76.6 ± 2.3
Titanium	0.283 ± 0.010	Zinc	6952 ± 91

SRM2709

SRM2710



SRM2711



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2711

Montana Soil

Moderately Elevated Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2711 is a moderately contaminated soil that was oven-dried, sieved, radiation sterilized, and blended to achieve a high degree of homogeneity. A unit of SRM 2711 consists of 50 g of the dried material.

The certified elements for SRM 2711 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should NOT be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

Noncertified Values: Noncertified values, shown below, are provided for information only. An element concentration value may not be certified, if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Carbon	2	Bromine	5
		Cerium	69
		Cesium	6.1
		Chromium	47
		Cobalt	10
		Dysprosium	5.6
		Europium	1.1
		Gallium	15
		Gold	.03
		Hafnium	7.3
		Holmium	1
		Indium	1.1
		Iodine	3
		Lanthanum	40
		Molybdenum	1.6
		Neodymium	31
		Rubidium	110
		Samarium	5.9
		Scandium	9
		Thorium	14
		Tungsten	3
		Uranium	2.6
		Ytterbium	2.7
		Yttrium	25
		Zirconium	230

Table 1. Certified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Aluminum	6.53 ± 0.09	Antimony	19.4 ± 1.8
Calcium	2.88 ± 0.08	Arsenic	105 ± 8
Iron	2.89 ± 0.06	Barium	726 ± 38
Magnesium	1.05 ± 0.03	Cadmium	41.70 ± 0.25
Phosphorus	0.086 ± 0.007	Copper	114 ± 2
Potassium	2.45 ± 0.08	Lead	1162 ± 31
Silicon	30.44 ± 0.19	Manganese	638 ± 28
Sodium	1.14 ± 0.03	Mercury	6.25 ± 0.19
Sulfur	0.042 ± 0.001	Nickel	20.6 ± 1.1
Titanium	0.306 ± 0.023	Selenium	1.52 ± 0.14
		Silver	4.63 ± 0.39
		Strontium	245.3 ± 0.7
		Thallium	2.47 ± 0.15
		Vanadium	81.6 ± 2.9
		Zinc	350.4 ± 4.8

NOTES

A3. Specifications

Handheld Analyzer	Description	
Weight	3.35 lbs. (Base wt.), 3.70 lbs. (1.6 kg) with battery	
Excitation Source	4 Watt X-ray tube - Au, Ag, Rh, or Ta anode (application optimized), 8-40 keV, 5 - 200 μ A, eight filter positions	
Detector	Various: Si PIN diode or Si Drift detector, thermo-electrically cooled, high resolution	
Power	Removable Li-ion batteries, or AC power unit	
Battery Life	Battery life varies depending on usage patterns	
Display	Color "transflective" touchscreen (800 x 600) with 16-bit LCD interface)	
Cal Check Coupon	316 Stainless Steel Alloy -- see also Docking Station	
Power Requirements for AC Adapter	110-220 VAC, 50-60 Hz, 600 W max (P/N 100043)	
Pressure Correction	Built-in barometer for automatic altitude correction	
Operating Environment	Temperature: -10°C to 45°C Humidity: 10 to 90% Relative Humidity, non-condensing Altitude Rating: 2000 meters	Operating specifications are posted as nominal.
Operating System	Windows Embedded CE®	
Application Software	Innov-X Systems' proprietary Data Acquisition and Processing package.	
USB Interface	USB 2.0	
Docking Station	Description	
Dimensions	L=14.0 in x W=8.75 in x H=4.75 in (H w/ spare battery = 6.5 in); Wt = 3.25 lbs.	
Power Requirement	AC adapter standard: 110-220 VAC, 50-60 Hz, 600 W max	
Cal Check Cup	Automatically performs Cal Check on inserted instrument	
Battery Charging	Charges battery in unit; can simultaneously charge spare battery in separate socket	
Accessories	Description	
AC Battery Charger	P/N 120253	
TestStand/Workstation	A-020-D - portable equipment stand making a fully shielded closed beam system. Uses Delta PC Software.	
Soil Foot	A-035	
Soil Extension Pole	P/N 990055	
Trimble Xploration Package (P6000FDC)	XPLOER FIELD DATA COLLECTION BUNDLE Trimble Nomad 800G-LC - Ruggedized 800Mhz Field Computer with integrated GPS, Wi-Fi, Bluetooth, Camera, 16Gb Memory, SD Card Slot Options: Barcode Scanner, Cell Phone Interface, Fully DGPS compatible running Windows Mobile 6.0 OS.	

Trimble Xploration
Package
(P6000TFG)

XPLOER TOTAL FIELD GEOCHEM BUNDLE

Includes all features supplied with **Field Data Collection Package** and:
Soil Extension Pole - Ergonomic extension pole facilitating soil analysis by a standing (and walking) operator.

Soil Foot - Attaches to nose of DELTA analyzer balancing analyzer on ground for hands-free, extended in-situ testing.

ioGAS - ioAnalytics GeoChemical Analysis Software Suite, an advanced spatial data analysis package for visualization, interrogation & validation of geochemical data for mineral exploration, mining, & environmental industries.

A4. Typical Delta Test Sequence

Innov-X Delta User Interface

Operators manage their measurement, analysis, and results activities from the *Innov-X* UI when in ANY of the Alloy modes/calibrations. This Appendix presents:

- Typical Sample Test Procedure, and
- Prerequisites for testing necessary for various modes



GO TO

The operational features of the *Home*, *Mode*, *Setup*, *Test*, and *Results* screens are explained in "*Delta User Interface Guide*" (PN 103202). In this document, "*Delta User Manual*," see *C4, Operations, Page 47* for a SNAPSHOT of the UI.

Typical Sample Test Procedure

This sample procedure features the **Alloy Plus** mode.

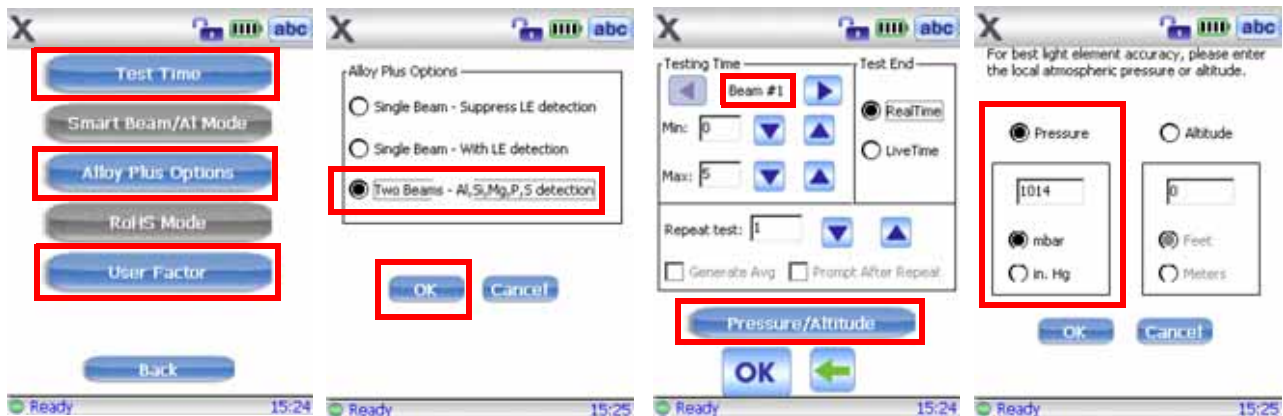
Other modes follow a similar sequence, however, the **Test Condition** screens are generally unique to each testing situation.

Use these steps:

1. From the **Home** screen, tap **Mode** icon
2. From **Mode** screen, tap the **Alloy Plus** icon
3. Select **Mode Setup** button



4. Select **Test Conditions**, then **Test Time**
5. Ensure that the **Testing Time** and other parameters are properly selected



6. Press OK button, the Back, then OK again
7. Return to Home screen, select Test to call the *Test-Alloy Plus* screen

Testing Tips for Certain Modes

Alloy:

- Place the analyzer's window on/over the test specimen, cover the window completely.
- Remember the Safety information ([C2, Pages 29-31](#)) to ensure your own personal safety.
- Take care not to damage the window film, such as when testing "metal turnings" or hot surfaces.

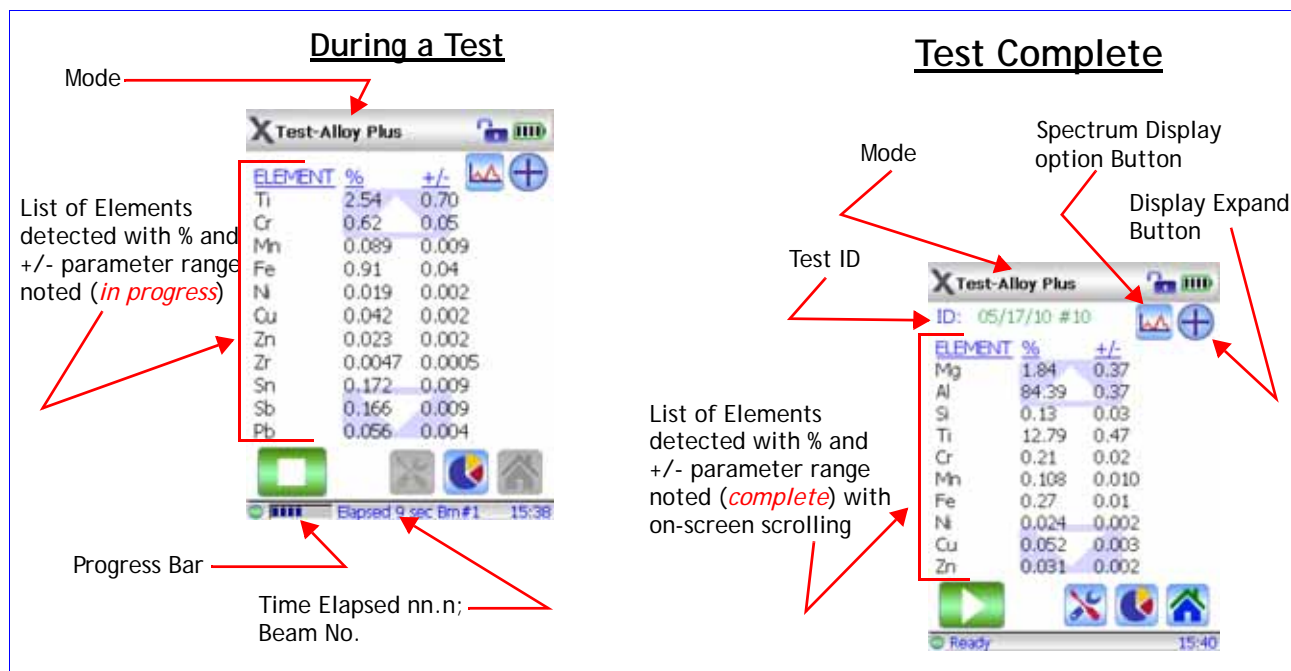
Soil or Mining:

- Place the analyzer's measurement window directly over the sample cup with the film side up.
- Specify the altitude or atmospheric pressure to gain increased accuracy for light elements.
- Confirm that the Testing Time and other parameters are properly selected
- Take care not to damage the window film, such as when testing uneven surfaces

RoHS:

- In order for XRF testing to be *quantitative*, samples must be:
 - Homogeneous
 - Have a certain minimum sample thickness
 - Five (5) mm for polymers and light alloys
 - Fifteen (15) mm for liquid samples
 - One (1) mm for other alloys
- If samples are heterogeneous, too thin, or too small, only *qualitative* screening is possible.

8. Start a new test by using one of these methods:
 - a. Tap Start button on the UI (toggles Delta to X-ray ON state)
 - or —
 - b. Pull the trigger (toggles instrument to X-ray ON state)
 - or —
 - c. Pull-and-hold the trigger when the "Deadman Trigger" is active.



Best Practices for Testing

Alloy Mode

Prior to a test session, analysts should have an understanding of *Innov-X's* implementation of:

- **Grade Libraries** -> populated by alloy Grades -> their chemistry characterized by Grade Tables.
See [Grade Libraries](#) for library management procedures.
- **Match Issues** including the concept and use of Match Numbers.
See [Match Issues](#)

Prior to initiating testing with the analyzer software, ensure that the following tasks are complete:

1. Analyzer does not require a Cal Check procedure.
2. The analyzer's measurement window is properly positioned in front of or over the test sample.
3. The *Match Cutoff* and *nSigma* parameters are configured.

Mining and Soil Modes

Check Standards

- Measure a **check standard** after each Cal Check, and periodically throughout the day. Test for a recommended minimum of one minute.
- Concentrations for elements of interest, in the range expected, plus or minus the error on the reading, should be within 20 percent of the standard value.
- [A2.Soil Testing](#) describes recommended quality assurance details.

- The standards provided with the analyzer are contained in XRF sample cups. These containers have a window (through which the soil can be viewed) on one side, and a solid cap on the other side.

Always measure samples through the window.

Sample Presentation

IN SITU TESTING

In situ testing is performed by pointing the analyzer at the ground. Clear any grass or large rocks away and hold the analyzer with the probe head front flush to the ground. Since dirt can accumulate on the analyzer window, gently wipe the window clean after each analysis. Ensure the window is not ripped or punctured.

BAGGED OR PREPARED SAMPLE TESTING

Analyze prepared samples in a sample cup, through its window. Place the instrument's measurement window directly over the sample cup with the film side up.

Preparation considerations include:

- Avoid measuring very thin samples, as this can affect results. Prepare samples cups to contain at least 15 (usually 4-8 grams) mm of packed samples.
- When analyzing bagged samples, ensure that sufficient sample material exists in the bag to create a sample thickness of a minimum of 15 mm for a spot size that is larger than the analyzer's measurement window.
- When using bags, cheaper bags (having thinner plastic walls) are better than more expensive ones (which have thicker plastic walls).

Consumer Mode

RoHS Best Practices

Check Standards

Innov-X Systems recommends that a check standard be measured after each Cal Check procedure, and periodically throughout the day.

Two certified standards are provided for verification.

- At least one standard should be measured for a minimum of two minutes.
- Concentrations for target elements (plus or minus the error on the reading) should be within 20% of the standard value.
- Standards provided are contained in XRF sample cups with a window (through which the plastic pellets can be viewed) on one side, and a solid cap on the other side.
- Samples should be measured in the sample cup, through the window.

Sample Presentation

Since many pieces of plastic analyzed for ROHS compliance are very small, take care to measure them in a safe and accurate manner.

See the IEC-ACEA recommendation for minimum thickness of test samples as shown in Chapter [C.8](#).

A5. User Maintenance

5.1 Alternative Techniques for Powering or Charging the Delta

5.1.1. AC Power Adapter Kit

The kit is an **optional** accessory.

It is supplied with a 10 foot AC power cord, a switching AC adapter, and a Battery Module. This kit eliminates the Li-ion battery requirement, however the user is constrained by the length of the AC power cord.

The procedure for using the AC adapter is shown below.



WARNING

Ensure that each AC supply circuit has adequate power load capability and is provided from a grounded AC receptacle.

TO SETUP THE ADAPTER:

1. Plug the three-prong male plug into the receptacle.

Female Plug →



← Male Plug

2. Insert the three-prong female plug into the male receptacle on the *Switching AC Adaptor* brick.



3. Slide the Battery Eliminator unit into the analyzer with the contacts to the left. It is keyed to only go in oriented this way.

Contacts →



4. The user now has no limit to instrument power; not restricted by battery charge level. The range of action is constrained by the length of the power cord.

5.1.2. Li ion Battery Charger Assembly

This section describes the procedure for charging the Li-ion batteries when the Delta Docking Station is **NOT** available. It features a single socket standalone battery charger (P/N 120253). The unit takes about two hours to completely charge a battery. Status of the battery's charge is shown by two lights on the power adaptor.

TO CHARGE THE BATTERY:

1. Plug the three-prong male plug into the Modes receptacle.



2. Plug the three-prong female plug into the male receptacle on the *AC Adaptor* brick.



3. Plug the connector labeled *CH4500 24 VDC* into the connector labeled *SWC* on the back of the charger.

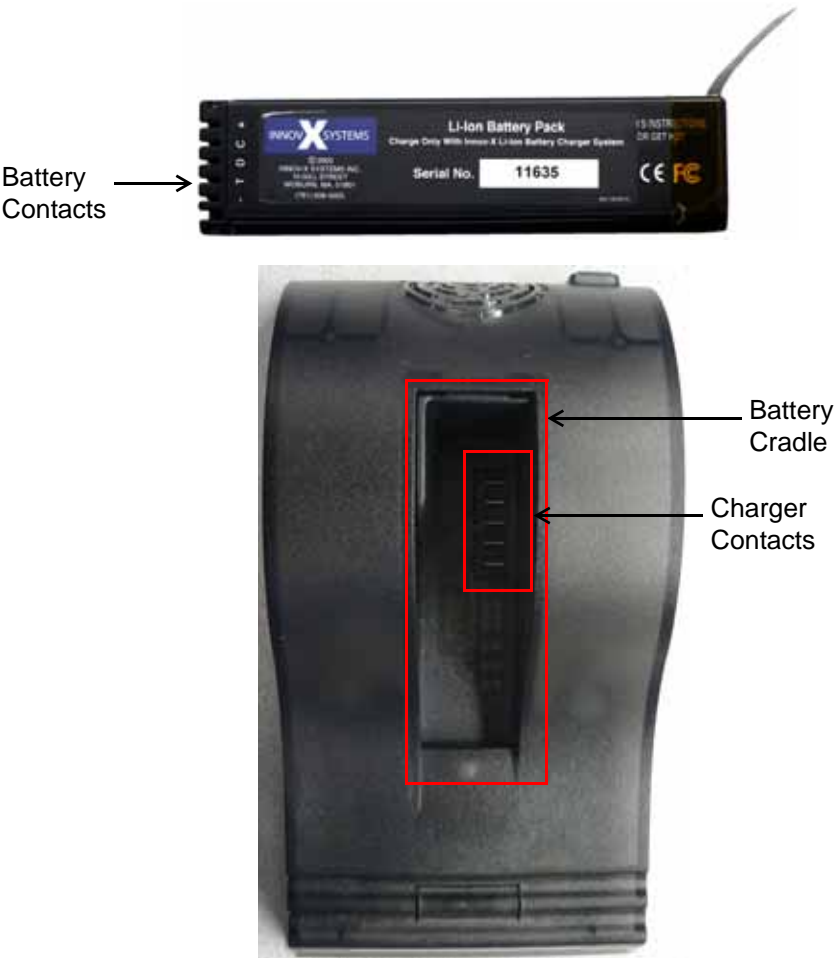


4. Insert the Li-ion battery into the charger with the contacts facing right.



CAUTION

Forcing the battery in with the contacts oriented improperly will injure the contacts and destroy the battery.



Battery Charger Status Lights

Left Light (Green only)	Right Light (Red only)	Status
On - Flashing	Off	Battery is charging
On - Solid	Off	Battery is charged
Off	On - Solid	Error. Remove battery and replace on charger. If the error persists, call Innov-X Systems Technical support.
Off	Off	No battery is on charger

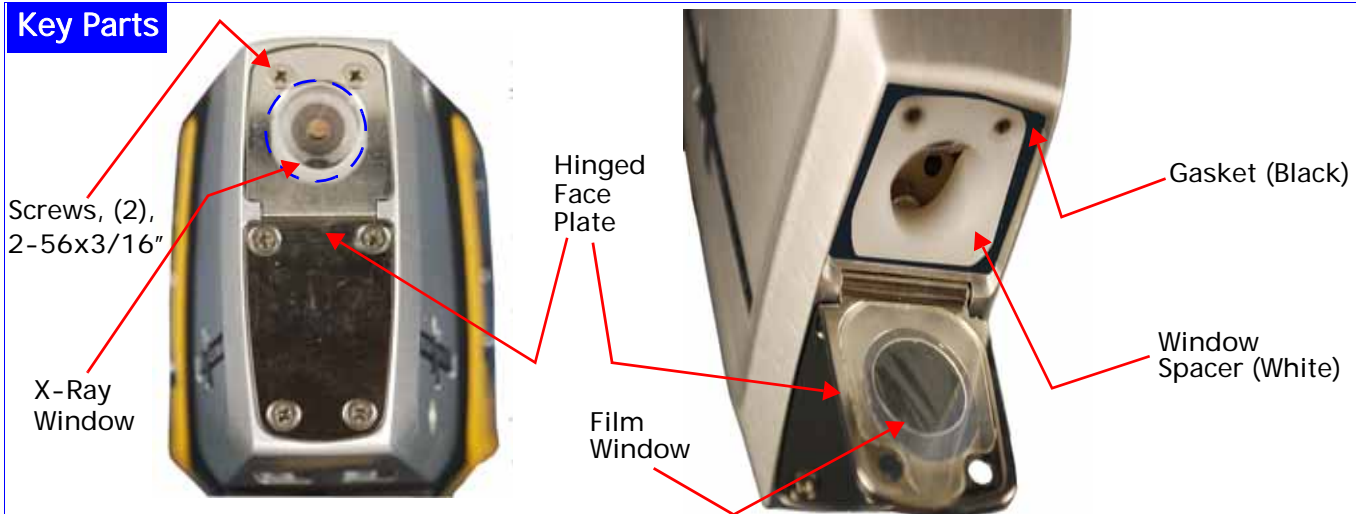


5.2 — Window Replacement for “Hinged Plate” Analyzers

How to Replace the Window on a Delta Analyzer (All Models)


Tools: *Required* - Small Phillips Head Screwdriver
Recommended - Tweezers or Needle nose pliers; Can of compressed air
As needed: - Lint-free wipes or swabs

Key Parts



Remove Old Window

Use these guidelines:

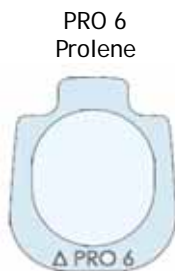
- During this entire removal process, keep dust or foreign material out of the instrument.
- Ensure that the instrument is powered off.
- Take care to have clean hands.
- Orient the instrument with the nose pointing up.
- Review the  TIPS section on next page **before** opening the hinged plate.
- Use Phillips head screwdriver to remove the two screws.
 - Carefully set aside the screws.
- Swing the hinged face plate out to expose the film window.
- Remove the old window.
- Observe the white plastic window spacer and the black rubber gasket
- Remove any dirt or foreign material from the gasket with compressed air
- Take EXTREME CARE to not harm any internal components



CAUTION Avoid expensive internal damage, DO NOT INSERT anything (fingers or tools) into the instrument.

Replacement Window Details

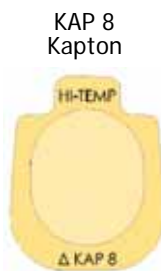
- Windows assemblies are made from either **Prolene**, **Mylar**, or **Kapton** film.
 - Users must specify the proper material to match their original application and calibration.
- The selected film is adhered to **one** side of a polyester backing plate; this creates a “front” and “back” surface
 - “Back” surface is completely covered with film.
 - “Front” surface shows a circular ridge where the film only is present, showing through the center hole of backing plate. This front surface also has a part type designator.
- Ensure that the “back” surface faces up when installing a new window (See below).



P/N: -->> 102999



103000



103173 (Hi-Temp)

103115 (Classic Standard)



Install Replacement

Follow these steps:

- Remove new window assembly from shipping envelope.
- Handle it by edges or use tweezers.
- Apply canned compressed air to clear any contaminants from the surfaces.
- Place window assembly on the hinged plate with **film back surface facing up**.
 - Center it to avoid the screw holes.
- Carefully rotate the hinged plate up to secure the window assembly.
- Insert the two screws; tighten only to “snug”.
 - Don’t over-tighten them or strip the threads.

TIPS



- Do not touch the film in the center opening (on either side).
- When using the screwdriver, keep a finger or thumb between the tool and the X-ray window. This prevents accidentally sticking the tool through the film and causing internal damage.
- The cost for replacement windows is quite low. Users are encouraged to change them frequently in order to obtain optimum test results.
- When not operating an instrument, experienced users routinely keep it the Delta Docking Station cradle.
 - This keeps the window clean, and
 - Prevents accidental damage to the window film, and
 - Takes advantage of the battery charging and Cal Checking procedure that the DDS offers.

NOTES

A6. Packing and Shipping

If the instrument is not returned in the protective case, it can be damaged during shipping. Innov-X Systems reserves the right to void the warranty on instruments that are damaged during return shipping that are sent without the protective case.

Prior to returning a unit, contact Customer Service at the appropriate depot:

<p>— United States —</p> <ul style="list-style-type: none"> • Phone: 1-781-938-5005 • Fax: 1-781-938-0128 • Email: Service@Innov-Xsys.com 		<p>— Europe —</p> <ul style="list-style-type: none"> • Phone: +31 (0)73 62 72 590 • Fax: +31 (0)73 62 72 599 • Email: info@innovx-europe.com 	
<p>— Canada —</p> <ul style="list-style-type: none"> • Phone: 1-778-960-6279 • Fax: 1-604-568-2474 • Toll Free Fax: 1-888-873-6598 • Email: service@innovx.ca 		<p>— Australia —</p> <ul style="list-style-type: none"> • Phone: 02 9577 9500 • Fax: 02 9519 1850 • Email: service@innovx.com 	
<p>• Mail & Shipping Address: Innov-X Systems, Inc. 100 Sylvan Road Woburn, MA 01801</p>		<p>• Mail & Shipping Address: Innov-X Systems, Inc. Kasteleinenkampweg 9R 5222 AX 's-Hertogenbosch The Netherlands</p>	
<p>• Mail & Shipping Address: Innov-X Canada 1201 West Georgia, Ste. 2 Vancouver BC Canada V6E 3J5</p>		<p>• Mail & Shipping Address: Innov-X Systems Australia PTY LTD Suite 6, Level 3 215 Euston Road Alexandria NSW 2015 Australia</p>	

Or call your local distributor.

Ensure that you receive the required RMA number.

Follow these instructions to return your XRF Analyzer:

1. Pack the analyzer in the black protective case in which it arrived, using the original packing materials.
2. Include the RMA in the case and reference the RMA number in your shipping documents.
3. Close the protective case and either:
 - Secure it with plastic zip ties,
 - or —
 - Pack the protective case within another box.

Regulations for Shipping Products with Lithium Ion Batteries

The United States and many other countries have instituted regulations that require shippers to use a special Caution label referring to a Lithium Ion Battery.

- Label must be prominently displayed on the outer shipping container of any product that contains a Lithium Ion battery.
- Shipper may copy the label shown below to facilitate making the warning label. Use a color copier if possible.



A7. Legal Information

This section provides copies of the following:

- Delta Analyzer Limited Warranty including:
 - Limitation of Liability
 - Warranty Period, Returns, and Repairs
 - Instructions for Contacting Innov-X
- End User Software License Agreement including:
 - Use, Restrictions, and Termination of Software
 - Governmental End User Conditions
 - Limited Warranty and Limitation of Liability

Innov-X Delta Analyzer Limited Warranty

General Terms

EXCEPT AS EXPRESSLY SET FORTH IN THIS LIMITED WARRANTY, INNOV-X SYSTEMS, INC. (INNOV-X) MAKES NO OTHER WARRANTIES OR CONDITIONS, EXPRESSED OR IMPLIED, INCLUDING ANY IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE. INNOV-X EXPRESSLY DISCLAIMS ALL WARRANTIES AND CONDITIONS NOT STATED IN THIS LIMITED WARRANTY. ANY IMPLIED WARRANTIES THAT MAY BE IMPOSED BY LAW ARE LIMITED IN DURATION TO THE LIMITED WARRANTY PERIOD.

This Limited Warranty applies to Innov-X analyzers sold or leased from Innov-X, its affiliates, authorized resellers, or country distributors (collectively referred to in this Limited Warranty as “Innov-X.”

Innov-X warrants that the analyzer and all its internal and external components that you have purchased are free from defects in materials or workmanship under normal use during the Limited Warranty Period. The Limited Warranty Period starts on the date of shipment by Innov-X. You may be required to provide proof of purchase or lease as a condition of receiving warranty service. You are entitled to warranty service according to the terms and conditions of this document if a repair to your Innov-X analyzer is required within the Limited Warranty Period.

During the Limited Warranty Period, Innov-X will repair or replace the defective component parts. All component parts removed under this Limited Warranty become the property of Innov-X. In the unlikely event that your Innov-X analyzer has a recurring failure, Innov-X, at its discretion, may elect to provide you with a replacement unit of Innov-X’s choosing that is at least equivalent to your Innov-X analyzer. This is your exclusive remedy for defective products. The repaired or replacement analyzer is warranted for the remainder of the limited Warranty Period.

YOU SHOULD MAKE PERIODIC BACKUP COPIES OF THE DATA STORED ON THE ANALYZER’S SYSTEM COMPUTER AS A PRECAUTION AGAINST POSSIBLE FAILURES, ALTERATION, OR LOSS OF THE DATA. BEFORE RETURNING ANY UNIT FOR SERVICE, BE SURE TO BACK UP DATA AND REMOVE ANY CONFIDENTIAL, PROPRIETARY, OR PERSONAL INFORMATION. INNOV-X IS NOT RESPONSIBLE FOR DAMAGE TO OR LOSS OF ANY PROGRAMS, OR DATA. INNOV-X IS NOT RESPONSIBLE FOR THE RESTORATION OR REINSTALLATION OF ANY PROGRAMS OR DATA OTHER THAN SOFTWARE INSTALLED BY INNOV-X WHEN THE ANALYZER IS MANUFACTURED.

Innov-X does not warrant that the operation of this analyzer will be uninterrupted or error-free. Innov-X is not responsible for damage that occurs as a result of your failure to follow the instructions that came with the Innov-X analyzer.

This Limited Warranty does not apply to expendable parts. This Limited Warranty does not extend to any analyzer from which the serial number has been removed or that has

been damaged or rendered defective (a) as a result of accident, misuse, abuse, or other external causes; (b) by operation outside the usage parameters stated in user documentation that shipped with the product; (c) by modification or service by anyone other than (i) Innov-X, or (ii) an Innov-X authorized service provider; (d) installation of software not approved by Innov-X.

These terms and conditions constitute the complete and exclusive warranty agreement between you and Innov-X regarding the Innov-X analyzer you have purchased or leased. These terms and conditions supersede any prior agreements or representations --- including representations made in Innov-X sales literature or advice given to you by Innov-X or any agent or employee of Innov-X --- that may have been made in connection with your purchase or lease of the Innov-X analyzer. No change to the conditions of this Limited Warranty is valid unless it is made in writing and signed by an authorized representative of Innov-X.

Limitation of Liability

IF YOUR INNOV-X ANALYZER FAILS TO WORK AS WARRANTED ABOVE, YOUR SOLE AND EXCLUSIVE REMEDY SHALL BE REPAIR OR REPLACEMENT. INNOV-X'S MAXIMUM LIABILITY UNDER THIS LIMITED WARRANTY IS EXPRESSLY LIMITED TO THE LESSER OF THE PRICE YOU HAVE PAID FOR THE ANALYZER OR THE COST OF REPAIR OR REPLACEMENT OF ANY COMPONENTS THAT MALFUNCTION IN CONDITION OF NORMAL USE.

INNOV-X IS NOT LIABLE FOR ANY DAMAGE CAUSED BY THE PRODUCT OR THE FAILURE OF THE PRODUCT TO PERFORM INCLUDING ANY LOST PROFITS OR SAVINGS OR SPECIAL, INCIDENTAL, OR CONSEQUENTIAL DAMAGES. INNOV-X IS NOT LIABLE FOR ANY CLAIM MADE BY A THIRD PARTY OR MADE BY YOU FOR A THIRD PARTY.

THIS LIMITATION OF LIABILITY APPLIES WHETHER DAMAGES ARE SOUGHT, OR A CLAIM MADE, UNDER THIS LIMITED WARRANTY OR AS A TORT CLAIM (INCLUDING NEGLIGENCE AND STRICT PRODUCT LIABILITY), A CONTRACT CLAIM, OR ANY OTHER CLAIM. THIS LIMITATION OF LIABILITY CANNOT BE WAIVED OR AMENDED BY ANY PERSON. THIS LIMITATION OF LIABILITY WILL BE EFFECTIVE EVEN IF YOU HAVE ADVISED INNOV-X OR AN AUTHORIZED REPRESENTATIVE OF INNOV-X OF THE POSSIBILITY OF ANY SUCH DAMAGES.

Software

This Limited Warranty does not warrant software products. The Innov-X software installed on the analyzer's system computer is covered by the Innov-X End User Software License Agreement.

Warranty Period

The warranty period for an Innov-X Delta Analyzer is one year. This warranty does not extend to expendable parts. Extended warranties are available from Innov-X.

Warranty Returns

A Return Material Authorization (RMA) Number must be obtained from the INNOV-X Service Department before any items can be shipped to the factory. Returned goods will not be accepted without an RMA Number. Customer will bear all shipping charges for warranty repairs. All goods returned to the factory for warranty repair should be properly packed to avoid damage and clearly marked with the RMA Number.

Warranty Repairs

Warranty repairs will be done either at the customer's site or at the INNOV-X plant, at our option. All service rendered by INNOV-X will be performed in a professional manner by qualified personnel.

Contacting Innov-X

Be sure to have the following information available before you call Innov-X:

- Analyzer serial number, model name, and model number
- Applicable error messages
- Description of problem
- Detailed questions

Methods of Contact

— United States —

- Phone: 1-781-938-5005
- Fax: 1-781-938-0128
- Email: Service@Innov-Xsys.com
- Mail & Shipping Address: Innov-X Systems, Inc.
100 Sylvan Road
Woburn, MA 01801

— Canada —

- Phone: 1-778-960-6279
- Fax: 1-604-568-2474
- Toll Free FAX: 1-888-873-6598
- Email: service@innovx.ca
- Mail & Shipping Address: Innov-X Canada
1201 West Georgia, Ste. 2
Vancouver BC
Canada V6E 3J5

— Europe —

- Phone: +31 (0)73-62 72 590
- Fax: +31 (0)73-62 72 599
- Email: info@innovx-europe.com
- Mail & Shipping Address: Innov-X Systems, Inc.
Kasteleinenkampweg 9R
5222 AX 's-Hertogenbosch
The Netherlands

— Australia —

- Phone: 02 9577 9500
- Fax: 02 9519 1850
- Email: service@innovx.com
- Mail & Shipping Address: Innov-X Systems Australia
PTY LTD
215 Euston Road, # 6 / L3
Alexandria, NSW, 2015
Australia

Or call your local distributor.

End User Software License Agreement

THIS END USER SOFTWARE LICENSE AGREEMENT IS FOR THE SOFTWARE USED TO OPERATE THE INNOV-X SYSTEMS' Delta™ XRF ANALYZER AND LIMITED PRODUCT WARRANTY

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A8. Alloy Grade Libraries

Every Delta unit is supplied with four libraries:

1. "Factory" Library unique to every Model
 - See following pages for tables showing Model/Factory grade names
2. Tramp Library
3. User Library #1 (user may store more than 500 grade names)
4. User Library # 2 (user may store more than 500 grade names)

Libraries are editable. However, InnovX does not recommend that users edit the "Factory" grade library.

Tramp Library

Every analyzer is shipped with a "Tramp" library comprised of seven base alloys. The "Tramp" library supports other grade libraries.

Users can set Tramp Limits, element by element, alloy base by alloy base, to meet their specific requirements.

A single click can select/deselect (globally) the Tramp feature.

How the Tramp Library works:

1. Tramp Grades are matched to alloy bases rather than specific grades.
 - Each sample is determined to be one of seven possible base alloys - see list below
 - Analyzer applies the Tramp Grade/base specific tramp limits from the matching Tramp Grade;
2. These "Tramp Limits" or "alloy base specific" limits are applied when an element is detected in a specific grade.
 - But, the nearest grade match has no specification for that element,
 - And, the concentration of the sample is less than the max limit specified by the matching Tramp Grade.
3. When conditions of #2 are met, the element is reported on the User Interface screen.
 - In **blue**;
 - Is labeled as a **"Tramp"** material in the grade comparison table;
 - But the grade match is not penalized.

PRACTICAL ADVANTAGES
of this "tramp element"
approach:

- Faster sorting,
- Fewer ambiguous or incorrect matches,
- Improved grade library integrity,
- Prominent labeling of tramp elements.

TRAMP Library Base Alloys

_AlAlloyBase

Common Tramp Elements: Pb, Bi, Sn, Fe, Cu, Zn

_CoAlloyBase

Common Tramp Elements: Al, Ti, V, Cu, Nb, Ta, Zr

_CuAlloyBase

Common Tramp Elements: S, As, Ag, Sb, Sn; not as common Pb, Co, Ni

_FeAlloyBase

Common Tramp Elements: V, Co, Cu, Ni, As - sometimes Si, W, Nb

_GenericAlloyBase

Common Tramp Elements: V, Co, Cu, Ni, As - sometimes Si, W, Nb

_NiAlloyBase

Common Tramp Elements: V, Co, W, Zr, Nb - sometimes Ta, Mo, Cr, Cu

_TiAlloyBase

Common Tramp Elements: Fe is common, Cu & Si show up at low levels.

Factory Grade Library {Table A9.1-Classic}

Aluminums- C	Cobalt Alloys - C	Specialty Grades- C	Copper Alloys -C		
2007 2011 2018 2117 2618 4032 5454 6040 6061 6070 6253 6262 7005 7016 7019 7039 7050 7072 7075 7104 1100-plus 2024-plus 2098-2195 2219-2519 3003 or 4 or 5 355-2 5052-plus 5086-plus 6063-plus 7049-149-249	Alloy 686 AlnicoVIII Cobalt Elgiloy F75 FSX-414 Haynes188 Haynes36 HS-1 HS-12 HS-19 HS-21 HS25-L605 HS-31 HS-4 HS-6B Jetalloy MarM302 MarM509 MarM905 MP35N MPN159 Star J Ultimet	60Sn-40Pb 63Sn-37Pb 96-4 AZ31B AZ91A or C SAC 300 SAC 305 SAC 400 SAC 405 97-3 Ag Au Bi Cb 103 CP Ta Cr Densalloy Hf Mn Mo Nb Ni Pb Pd Re Sb Se Sn TungCarb C TungCarb S V W Zn Zr Zr 2 or 4 Zr 702 Zr 704 Zr 705	C 110 C 172 C 194 C 210 C 220 C 260 C 270 C 310 C 314 C 330 C 332 C 340 C 342 C 360 C 377 C 425 C 443 C 464 C 482 C 485	C 510 C 524 C 534 C 544 C 623 C 630 C 655 C 667 C 673 C 675 C 706 C 710 C 715 C 745 C 752 C 814 C 836 C 857	C 864 C 867 C 868 C 875 C 8932 C 903 C 922 C 932 C 937 C 955 C194HiCu C197HiCu Elec Cu Muntz NarloyZ SeBiLOYI SeBiLOYII SeBiLOYIII
Low Alloy Steels-C	Chrome- Moly Steels -C	Nickel Alloys - C			
3310 4130 4140 4340 8620 9310 12L14 A10 Carb 1-2 Moly Carbon Steel 20Mo4	1 1-4 Cr 2 1-4 Cr 5 Cr 9 Cr P91	B 1900 B-1900 Hf C-1023 GMR235 GTD222 Hast BC1 HastB HastB2 HastB3 HastC2000 HastC22 HastC276 HastC4 HastF HastG HastG2 HastG3 HastG30 HastN HastR HastS HastW HastX Haynes230 HR160 HyMu80 I-102I-49 I-600 I-601	I-617 I-625 I-690 I-700 I-702 I-706 I-713 I-718 I-720 I-722 I-725 I-738 I-750 I-792 I-800 I-801 I-825 I-901 I-903 I-907-909 I-939 IN100Mar M002 MarM200 MarM246 MarM247	MarM421 Monel400 Monel411 MonelK500 MuMetal Ni 200 NichromeV Nim101 Nim263 Nimonic75 Nimonic80A Nimonic90 PWA1480 PWA1484 RA333 Rene125 Rene142 Rene220 Rene41 Rene77 Rene80 Rene95 Supertherm Udimet500 Udimet520 Udimet700 Waspaloy	

Factory Grade Library {Table A9.2-Classic}

Stainless Grades - C		
201	21-6-9	Haynes556
203	25-4-4	Incoloy840
304	254SMO	Invar 36
309	26-1	Kovar
310	29-4	M152
316	29-4-2	Maraging35
317	29-4C	0
321	302HQ	MaragingC2
329	410 Cb	00
330	410-16-20	MaragingC2
347	904L	50
422	A-286	MaragingC3
430	AL6XN	00
431	Alloy42	N-155
434	Alnicoll	Ni-hard#1
440	Alnicoll	Ni-hard#4
441	AlnicoV	Ni-Span902
446	AMS350	Nitronic40
2003	AMS355	Nitronic50
2101	CD4MCU	Nitronic60
2507	Custom450	RA330
13-8 Mo	Custom455	RA85H
15-5 PH	Duplex2205	Zeron100
15Mn7Cr	E-bite	
17-4 PH	Ferallium25	
17-7 PH	5	
19-9DL	GreekAs-	
19-9DX	coloy	
20Cb3	H12	
20Mo6	H13	

Ti Grades - C
Cp Ti
Cp Ti Pd
Ti 12
Ti 17
Ti 3 2-5
Ti 6-22-22
Ti 6-2-4-2
Ti 6-2-4-6
Ti 6-4
Ti 6-6-2
Ti 8
Ti 8-1-1
Ti10-2-3
Ti15-3-3-3
Ti3-11-13
Ti5 - 2-5
Ti6-2-1-1
TiBetaC

Tool Steels- C
A2
A6
A7
D2 or D4
D7
H-11
M1
M2
M4
M42
O1
O2
O6
O7
S1
S5
S6
S7
T1

Delta CLASSIC

Factory Grade Library {Table A9.3-Standard}

Delta STANDARD

Aluminums-S	Cobalt Alloys - S	Specialty Grades- S	Copper Alloys -S		
319	Alloy 686	60Sn-40Pb	C 110	C 510	C 864
333	AlnicoVIII	63Sn-37Pb	C 172	C 524	C 867
380	Cobalt	96-4	C 194	C 534	C 868
383	Elgiloy	AZ31B	C 210	C 544	C 875
384	F75	AZ91A or C	C 220	C 623	C 8932
2007	FSX-414	SAC 300	C 260	C 630	C 903
2011	Haynes188	SAC 305	C 270	C 655	C 922
2018	Haynes36	SAC 400	C 310	C 667	C 932
2024	HS-1	SAC 405	C 314	C 673	C 937
2117	HS-12	97-3	C 330	C 675	C 955
2618	HS-19	Ag	C 332	C 706	C194HiCu
3004	HS-21	Au	C 340	C 710	C197HiCu
4032	HS25-L605	Bi	C 342	C 715	Elec Cu
5042	HS-31	Cb 103	C 360	C 745	Muntz
5052	HS-4	CP Ta	C 377	C 752	NarloyZ
5083	HS-6B	Cr	C 425	C 814	SeBiLOYI
5086	Jetalloy	Densalloy	C 443	C 836	SeBiLOYII
5154	MarM302	Hf	C 464	C 857	SeBiLOYIII
5454	MarM509	Mn	C 482		
6040	MarM905	Mo	C 485		
6061	MP35N	Nb			
6070	MPN159	Ni			
6253	Star J	Pb			
6262	Ultimet	Pd			
7005		Re			
7016		Sb			
7019		Se			
7039		Sn			
7050		TungCarb C			
7072		TungCarb S			
7075		V			
7104		W			
1100-plus		Zn			
2014-17 std		Zr			
2024-plus		Zr 2 or 4			
2098-2195		Zr 702			
2219-2519		Zr 704			
3003 or 4 or 5		Zr 705			
3003 or 5					
355-2					
356-57-std					
5052-plus					
5056-82					
5086-plus					
6063-plus					
7049-149-249					
	Low Alloy Steels-S	Chrome-Moly Steels -S	Nickel Alloys - S		
	3310	1 1-4 Cr	20Mo4	I-102	MarM247
	4130	2 1-4 Cr	B 1900	I-49	MarM421
	4140	5 Cr	B-1900 Hf	I-600	Monel400
	4340	9 Cr	C-1023	I-601	Monel411
	8620	P91	Colmonoy	I-602	MonelK500
	9310		6	I-617	MuMetal
	12L14		GMR235	I-625	Ni 200
	A10		GTD222	I-690	NichromeV
	Carb 1-2		Hast BC1	I-700	Nim101
	Moly		HastB	I-702	Nim263
	Carbon Steel		HastB2	I-706	Nimonic75
	P20		HastB3	I-713	Nimonic80A
	135 N		HastC2000	I-718	Nimonic90
			HastC22	I-720	PWA1480
			HastC276	I-722	PWA1484
			HastC4	I-725	RA333
			HastF	I-738	Rene125
			HastG	I-750	Rene142
			HastG2	I-792	Rene220
			HastG3	I-800	Rene41
			HastG30	I-801	Rene77
			HastN	I-825	Rene80
			HastR	I-901	Rene95
			HastS	I-903	Supertherm
			HastW	I-907-909	Udimet500
			HastX	I-939	Udimet520
			Haynes214	IN100	Udimet700
			Haynes230	MarM002	Waspaloy
			HR160	MarM200	
			HyMu80	MarM246	

Factory Grade Library {Table A9.4-Standard}

Stainless Grades - S			Ti Grades - S		Tool Steels- S	
201	17-4 PH	H12	Cp Ti		A2	
203	17-7 PH	H13	Cp Ti Pd		A6	
303	19-9DL	Haynes556	Ti 12		A7	
304	19-9DX	Incoloy840	Ti 17		D2 or D4	
309	20Cb3	Invar 36	Ti 3 2-5		D7	
310	20Mo6	Kovar	Ti 6-22-22		H-11	
316		M152	Ti 6-2-4-2		M1	
317	21-6-9	Maraging350	Ti 6-2-4-6		M2	
321	25-4-4	MaragingC200	Ti 6-4		M4	
329	254SMO	MaragingC250	Ti 6-6-2		M42	
330	26-1	MaragingC300	Ti 8		O1	
347	29-4		Ti 8-1-1		O2	
410	29-4-2	N-155	Ti10-2-3		O6	
416	29-4C	Ni-hard#1	Ti15-3-3-3		O7	
420	302HQ	Ni-hard#4	Ti3-11-13		S1	
422	410 Cb	Ni-Span902	Ti5 - 2-5		S5	
430	410-16-20	Nitronic40	Ti6-2-1-1		S6	
431	904L	Nitronic50	TiBetaC		S7	
434	A-286	Nitronic60			T1	
440	AL6XN	RA330				
441	Alloy42	RA85H				
446	AlnicoII	Zeron100				
2003	AlnicoIII					
2101	AlnicoV					
2205	AMS350					
2205	AMS355					
2507	CD4MCU					
13-8 Mo	Custom450					
15-5 PH	Custom455					
15Mn7Cr	E-bite					
	Ferallium255					
	GreekAscoloy					


Factory Grade Library {Table A9.6-Premium}

Stainless Grades - P		
201	17-4 PH	H12
203	17-7 PH	H13
303	19-9DL	Haynes556
304	19-9DX	Incoloy840
309	20Cb3	Invar 36
310	20Mo6	Kovar
316	21-6-9	M152
317	25-4-4	Maraging350
321	254SMO	MaragingC200
329	26-1	MaragingC250
330	29-4	MaragingC300
347	29-4-2	N-155
410	29-4C	Ni-hard#1
416	302HQ	Ni-hard#4
420	410 Cb	Ni-Span902
422	410-16-20	Nitronic40
430	904L	Nitronic50
431	A-286	Nitronic60
434	AL6XN	RA330
440	Alloy42	RA85H
441	Alnicoll	Zeron100
446	Alnicoll	
2003	AlnicoV	
2101	AMS350	
2205	AMS355	
2205	CD4MCU	
2507	Custom450	
13-8 Mo	Custom455	
15-5 PH	E-bite	
15Mn7Cr	Ferallium255	
	GreekAscology	

Ti Grades - P
Cp Ti
Cp Ti Pd
Ti 12
Ti 17
Ti 3 2-5
Ti 6-22-22
Ti 6-2-4-2
Ti 6-2-4-6
Ti 6-4
Ti 6-6-2
Ti 8
Ti 8-1-1
Ti10-2-3
Ti15-3-3-3
Ti3-11-13
Ti5 - 2-5
Ti6-2-1-1
TiBetaC

Tool Steels- P
A2
A6
A7
D2 or D4
D7
H-11
M1
M2
M4
M42
O1
O2
O6
O7
S1
S5
S6
S7
T1

Delta PREMIUM

	GO TO
	<ul style="list-style-type: none">• See “Delta User Interface Guide” (PN 103202) for a complete description for using Alloy modes’ new capabilities such as:<ul style="list-style-type: none">— Grade Match Messaging,— SmartSorting,— Nominal Chemistry, and— Tramp Library management.

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DRAFT

Field Screening for Metal Constituents Using a Field Portable XRF (OP#3020)

APPENDIX C

EPA Method 6200 – X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soils and Sediments

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

SW-846 is not intended to be an analytical training manual. Therefore, method procedures are written based on the assumption that they will be performed by analysts who are formally trained in at least the basic principles of chemical analysis and in the use of the subject technology.

In addition, SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed below for soil and sediment samples. Some common elements are not listed in this method because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). These light elements are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed below are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF. The following RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Antimony (Sb)	7440-36-0
Arsenic (As)	7440-38-0
Barium (Ba)	7440-39-3
Cadmium (Cd)	7440-43-9
Chromium (Cr)	7440-47-3
Cobalt (Co)	7440-48-4
Copper (Cu)	7440-50-8
Lead (Pb)	7439-92-1
Mercury (Hg)	7439-97-6
Nickel (Ni)	7440-02-0
Selenium (Se)	7782-49-2
Silver (Ag)	7440-22-4
Thallium (Tl)	7440-28-0
Tin (Sn)	7440-31-5

Analytes	CAS Registry No.
Vanadium (V)	7440-62-2
Zinc (Zn)	7440-66-6

In addition, the following non-RCRA analytes have been determined by this method:

Analytes	CAS Registry No.
Calcium (Ca)	7440-70-2
Iron (Fe)	7439-89-6
Manganese (Mn)	7439-96-5
Molybdenum (Mo)	7439-93-7
Potassium (K)	7440-09-7
Rubidium (Rb)	7440-17-7
Strontium (Sr)	7440-24-6
Thorium (Th)	7440-29-1
Titanium (Ti)	7440-32-6
Zirconium (Zr)	7440-67-7

1.2 This method is a screening method to be used with confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnace atomic absorption spectrometry (GFAA), inductively coupled plasma-atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)). This method's main strength is that it is a rapid field screening procedure. The method's lower limits of detection are typically above the toxicity characteristic regulatory level for most RCRA analytes. However, when the obtainable values for precision, accuracy, and laboratory-established sensitivity of this method meet project-specific data quality objectives (DQOs), FPXRF is a fast, powerful, cost effective technology for site characterization.

1.3 The method sensitivity or lower limit of detection depends on several factors, including the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. Example lower limits of detection for analytes of interest in environmental applications are shown in Table 1. These limits apply to a clean spiked matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (100 -600 second) count times. These sensitivity values are given for guidance only and may not always be achievable, since they will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of performance-based sensitivity is presented in Sec. 9.6.

1.4 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.5 Use of this method is restricted to use by, or under supervision of, personnel appropriately experienced and trained in the use and operation of an XRF instrument. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use either sealed radioisotope sources or x-ray tubes to irradiate samples with x-rays. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This latter process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples. The three electron shells include the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α), beta (β), or gamma (γ) etc., which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

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, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than

the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.77 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments, specifically, in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF -- Field portable x-ray fluorescence.

3.2 MCA -- Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS -- Site-specific calibration standards.

3.4 FP -- Fundamental parameter.

3.5 ROI -- Region of interest.

3.6 SRM -- Standard reference material; a standard containing certified amounts of metals in soil or sediment.

3.7 eV -- Electron volt; a unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One, Chapter Three, and the manufacturer's instructions for other definitions that may be relevant to this procedure.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup (i.e., against the cup window), the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 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. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_{β} line of element Z-1 with the K_{α} line of element Z. This is called the K_{α}/K_{β} interference. Because the $K_{\alpha}:K_{\beta}$ intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_{α} and K_{β} energies are 4.95 and 5.43 keV, respectively, and the Cr K_{α} energy is 5.41 keV. The Fe K_{α} and K_{β} energies are 6.40 and 7.06 keV, respectively, and the Co K_{α} energy is 6.92 keV. The difference between the V K_{β} and Cr K_{α} energies is 20 eV, and the difference between the Fe K_{β} and the Co K_{α} energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_{α} /lead (Pb) L_{α} and sulfur (S) K_{α} /Pb M_{α} . In the As/Pb case, Pb can be measured from the Pb L_{β} line, and As can be measured from either the As K_{α} or the As K_{β} line; in this way the interference can be corrected. If the As K_{β} line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_{α} line. If the As K_{α} line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in reporting of a "nondetect" or a "less than" value (e.g., <300 ppm) for As, regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis using other techniques (e.g., flame atomic absorption spectrometry (FLAA), graphite furnace atomic absorption spectrometry (GFAA), inductively coupled plasma-

atomic emission spectrometry, (ICP-AES), or inductively coupled plasma-mass spectrometry, (ICP-MS)).

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as Method 3050, or a total digestion procedure, such as Method 3052, is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project-specific data quality objectives (DQOs).

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method (see Table 8), the confirmatory method used was Method 3050, and the FPXRF data compared very well with regression correlation coefficients (r often exceeding 0.95, except for barium and chromium). The critical factor is that the digestion procedure and analytical reference method used should meet the DQOs of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Sec. 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10° F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The user is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals listed in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

NOTE: No MSDS applies directly to the radiation-producing instrument because that is covered under the Nuclear Regulatory Commission (NRC) or applicable state regulations.

5.2 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals.

Licenses for radioactive materials are of two types, specifically: (1) a general license which is usually initiated by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) a specific license which is issued to named persons for the operation of radioactive instruments as required by local, state, or federal agencies. A copy of the radioactive material license (for specific licenses only) and leak tests should be present with the instrument at all times and available to local and national authorities upon request.

X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply, however, if the tube is properly positioned within the instrument, this is only a negligible risk. Any instrument (x-ray tube or radioisotope based) is capable of delivering an electric shock from the basic circuitry when the system is inappropriately opened.

5.3 Radiation monitoring equipment should be used with the handling and operation of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs or badges should be worn in the area of maximum exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

6.0 EQUIPMENT AND SUPPLIES

The mention of trade names or commercial products in this manual is for illustrative purposes only, and does not constitute an EPA endorsement or exclusive recommendation for

use. The products and instrument settings cited in SW-846 methods represent those products and settings used during method development or subsequently evaluated by the Agency. Glassware, reagents, supplies, equipment, and settings other than those listed in this manual may be employed provided that method performance appropriate for the intended application has been demonstrated and documented.

6.1 FPXRF spectrometer -- An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation sources -- FPXRF instruments use either a sealed radioisotope source or an x-ray tube to provide the excitation source. Many FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron Fe-55 (^{55}Fe), cadmium Cd-109 (^{109}Cd), americium Am-241 (^{241}Am), and curium Cm-244 (^{244}Cm). These sources may be contained in a probe along with a window and the detector; the probe may be connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. This is due to the ever increasing time required for the analysis rather than a decrease in instrument performance. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments. An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum necessary for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of

accelerating voltage is governed both by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material and by the instrument's ability to cool the x-ray tube. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample presentation device -- FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For FPXRF instruments operated in the intrusive mode, the probe may be rotated so that the window faces either upward or downward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors -- The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon $\text{Si}(\text{Li})$. The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The $\text{Si}(\text{Li})$ detector must be cooled to at least -90°C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a $\text{Si}(\text{Li})$ detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 L. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2 –270 eV; silicon pin diode–250 eV; $\text{Si}(\text{Li})$ –170 eV; and gas-filled, proportional counter–750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data processing units -- The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amplitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in ppm on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 3,000 to 5,000 sets of numerical analytical results. Most FPXRF instruments are menu-driven from software built into the

units or from PCs. Once the data-storage memory of an FPXRF unit is full or at any other time, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery and battery charger.

6.3 Polyethylene sample cups -- 31 to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film -- Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 µm thick.

6.5 Mortar and pestle -- Glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers -- Glass or plastic to store samples.

6.7 Sieves -- 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels -- For smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags -- Used for collection and homogenization of soil samples.

6.10 Drying oven -- Standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Pure element standards -- Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if designated for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.3 Site-specific calibration standards -- Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.3.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of 10 samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.3.2 Each sample should be oven-dried for 2 to 4 hr at a temperature of less than 150 °C. If mercury is to be analyzed, a separate sample portion should be dried at ambient temperature as heating may volatilize the mercury. When the sample is dry, all large, organic debris and nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be homogenized (see Sec. 7.3.3) and then a representative portion ground with a mortar and pestle or other mechanical means, prior to passing through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.3.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 g of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 g of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.4 Blank samples -- The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the established lower limit of detection. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.5 Standard reference materials -- Standard reference materials (SRMs) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. When these SRMs are unavailable, alternate standards may be used (e.g., NIST 2702).

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, "Inorganic Analytes."

9.0 QUALITY CONTROL

9.1 Follow the manufacturer's instructions for the quality control procedures specific to use of the testing product. Refer to Chapter One for additional guidance on quality assurance (QA) and quality control (QC) protocols. Any effort involving the collection of analytical data should include development of a structured and systematic planning document, such as a Quality Assurance Project Plan (QAPP) or a Sampling and Analysis Plan (SAP), which translates project objectives and specifications into directions for those that will implement the project and assess the results.

9.2 Energy calibration check -- To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting,

which would indicate drift within the instrument. As discussed in Sec. 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (more than 10 °F).

9.2.1 The energy calibration check should be run at a frequency consistent with manufacturer's recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.2 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak. The intensity and channel number of the pure element as measured using the source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank samples -- Two types of blank samples should be analyzed for FPXRF analysis, specifically, instrument blanks and method blanks.

9.3.1 An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window. The instrument blank can be silicon dioxide, a polytetrafluoroethylene (PTFE) block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the established lower limit of detection should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. If the method blank does not contain the target analyte at a level that interferes with the project-specific data quality objectives then the method blank would be considered acceptable. In the absence of project-specific data quality objectives, if the blank is less than the lowest level of detection or less than 10% of the lowest sample concentration for the analyte, whichever is greater, then the method blank would be considered acceptable. If the method blank cannot be considered acceptable, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration verification checks -- A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ± 20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision measurements -- The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent. If both in situ and intrusive analytical techniques are used during the course of one day, it is recommended that separate precision calculations be performed for each analysis type.

The equation for calculating RSD is as follows:

$$\text{RSD} = (\text{SD}/\text{Mean Concentration}) \times 100$$

where:

RSD	=	Relative standard deviation for the precision measurement for the analyte
SD	=	Standard deviation of the concentration for the analyte
Mean concentration	=	Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the sensitivity, but decreases sample throughput.

9.6 The lower limits of detection should be established from actual measured performance based on spike recoveries in the matrix of concern or from acceptable method performance on a certified reference material of the appropriate matrix and within the appropriate calibration range for the application. This is considered the best estimate of the true method sensitivity as opposed to a statistical determination based on the standard deviation of

replicate analyses of a low-concentration sample. While the statistical approach demonstrates the potential data variability for a given sample matrix at one point in time, it does not represent what can be detected or most importantly the lowest concentration that can be calibrated. For this reason the sensitivity should be established as the lowest point of detection based on acceptable target analyte recovery in the desired sample matrix.

9.7 Confirmatory samples -- The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on project-specific data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument calibration -- Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments, namely: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental parameters calibration -- FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are necessary, although site-specific samples with confirmed and validated analytical results for all elements present could be used.
- Cost is reduced because fewer confirmatory laboratory results or calibration standards are necessary.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Sec. 7.3. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective energy FP calibration -- The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ± 20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ± 20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference

C_k = Certified concentration of standard sample

C_s = Measured concentration of standard sample

10.2.2 BFP calibration -- BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended

count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ± 20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ± 20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical calibration -- An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Sec. 7.3; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is necessary. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are necessary to perform an adequate empirical calibration. The exact number of standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton normalization method -- The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline reading. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_{α} peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later during analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, remove any large or nonrepresentative debris from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Also, the soil surface must be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide example performance data for this method, this modest amount of sample preparation was found to take less than 5 min per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for in situ analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on the desired method sensitivity. Due to the heterogeneous nature of the soil sample, in situ analysis can provide only "screening" type data.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 g or 250 cm³, which is enough soil to fill an 8-ounce jar. However, the exact dimensions and sample depth should take into consideration the heterogeneous deposition of contaminants and will ultimately depend on the desired project-specific data quality objectives. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Sec. 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the time necessary for homogenization procedure using the fluorescein dye ranged from 3 to 5 min per sample. As demonstrated in Secs. 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, the direct analysis through the plastic bag is possible without the more labor intensive steps of drying, grinding, and sieving given in Secs. 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps should be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 g) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hr in the convection or toaster oven at a temperature not greater than 150 °C. Samples may also be air dried under ambient temperature conditions using a 10- to 20-g portion. Regardless of what drying mechanism is used, the drying process is considered complete when a constant sample weight can be obtained. Care should be taken to avoid sample cross-contamination and these measures can be evaluated by including an appropriate method blank sample along with any sample preparation process.

CAUTION: Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 min per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 μm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle, and sieves must be thoroughly cleaned so that any cross-contamination is below the established lower limit of detection of the procedure or DQOs of the analysis. If all recommended sample preparation steps are followed, there is a high probability the desired laboratory data quality may be obtained.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in ppm and can be downloaded to a personal computer, which can be used to provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation. See the manufacturer's instructions regarding data analysis and calculations.

13.0 METHOD PERFORMANCE

13.1 Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2 The sections to follow discuss three performance evaluation factors; namely, precision, accuracy, and comparability. The example data presented in Tables 4 through 8 were generated from results obtained from six FPXRF instruments (see Sec. 13.3). The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from "nondetect" to tens of thousands of mg/kg. These data are provided for guidance purposes only.

13.3 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode

detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.4 All example data presented in Tables 4 through 8 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.5 Precision measurements -- The example precision data are presented in Table 4. These data are provided for guidance purposes only. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from "nondetects" to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 4 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the lower limit of detection for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 4. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the lower limit of detection so that an RSD value calculated at 5 to 10 times this limit was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 5 shows these results. These data are provided for guidance purposes only. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the lower limit of detection for the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the in situ homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 5 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square,

measurements of different soil samples were actually taking place within the square. Table 5 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five instead of ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy measurements -- Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 6 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 6 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 6. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 6.

Table 7 provides a more detailed summary of accuracy data for one particular FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. These data are provided for guidance purposes only. Table 7 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability -- Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 8. Similar trends in the data were seen for all instruments. These data are provided for guidance purposes only.

Table 8 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. These data are provided for guidance purposes only. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--intrusive, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not

ground; and preparation 4—intrusive, with sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 8 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 8 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Sec. 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time necessary to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 min. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 min per sample. Lastly, when grinding and sieving is conducted, time has to be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 A. D. Hewitt, "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis," American Environmental Laboratory, pp 24-32, 1994.

13.8.2 S. Piorek and J. R. Pasmore, "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer," Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals, Las Vegas, Nevada, February 24-26, 1993, Vol 2, pp 1135-1151, 1993.

13.8.3 S. Shefsky, "Sample Handling Strategies for Accurate Lead-in-soil Measurements in the Field and Laboratory," *International Symposium of Field Screening Methods for Hazardous Waste and Toxic Chemicals*, Las Vegas, NV, January 29-31, 1997.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St., N.W. Washington, D.C. 20036, <http://www.acs.org>.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex, X-MET 920 User's Manual.
2. Spectrace Instruments, "Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction," 1994.
3. TN Spectrace, Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.
4. Unpublished SITE data, received from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The following pages contain the tables referenced by this method. A flow diagram of the procedure follows the tables.

TABLE 1

EXAMPLE INTERFERENCE FREE LOWER LIMITS OF DETECTION

Analyte	Chemical Abstract Series Number	Lower Limit of Detection in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: Refs. 1, 2, and 3

These data are provided for guidance purposes only.

TABLE 2
SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	432	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Refs. 1, 2, and 3

TABLE 3
SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Ref. 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4
EXAMPLE PRECISION VALUES

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the Lower Limit of Detection					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84 ^a	NR	24.80 ^a	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85 ^a	NR	24.92 ^a	20.92 ^a	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69 ^a	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32 ^a	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

These data are provided for guidance purposes only.

Source: Ref. 4

^a These values are biased high because the concentration of these analytes in the soil samples was near the lower limit of detection for that particular FPXRF instrument.

NR Not reported.

NA Not applicable; analyte was reported but was below the established lower limit detection.

TABLE 5

EXAMPLES OF PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive- Undried and Unground	Intrusive- Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium ^a	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel ^a	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver ^a	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

These data are provided for guidance purposes only.

Source: Ref. 4

^a These values may be biased high because the concentration of these analytes in the soil samples was near the lower limit of detection.

ND Not detected.

NR Not reported.

TABLE 6
EXAMPLE ACCURACY VALUES

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Ref. 4. These data are provided for guidance purposes only.

n: Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.

SD: Standard deviation; NA: Not applicable; only two data points, therefore, a SD was not calculated.

%Rec.: Percent recovery.

-- No data.

TABLE 7
EXAMPLE ACCURACY FOR TN 9000^a

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Ref. 4. These data are provided for guidance purposes only.

^a All concentrations in milligrams per kilogram.

%Rec.: Percent recovery; ND: Not detected; NA: Not applicable.

-- No data.

TABLE 8

EXAMPLE REGRESSION PARAMETERS FOR COMPARABILITY¹

	Arsenic				Barium				Copper			
	n	r ²	Int.	Slope	n	r ²	Int.	Slope	n	r ²	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r ²	Int.	Slope	n	r ²	Int.	Slope	n	r ²	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

Source: Ref. 4. These data are provided for guidance purposes only.

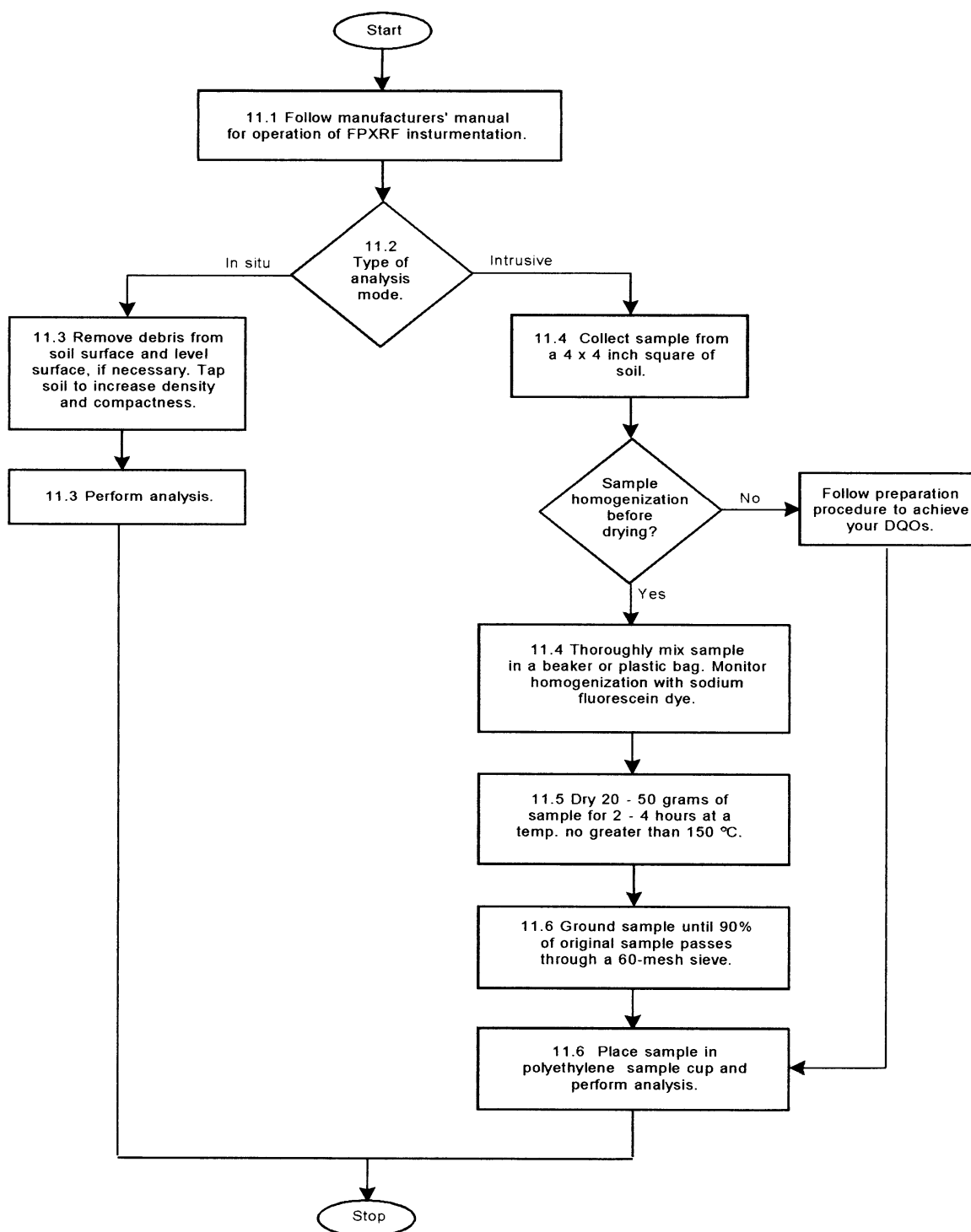
¹ Log-transformed data

n: Number of data points; r²: Coefficient of determination; Int.: Y-intercept

— No applicable data

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT



Coal Combustion By-Product (CCB) Visual Inspection Procedures

Pines Area of Investigation

1.0 Qualifications/Training

Visual inspections are conducted by a team of AECOM personnel. Each inspection team includes at least one geologist. Geologists are educated and trained in examining soils and other materials encountered at and below the ground surface. Geologists perform “visual inspections” of soils and other materials as a routine part of their professional activities. Information from visual inspections is fundamental to geologic and hydrogeologic interpretation at environmental sites across the country.

At the Pines Area of Investigation, geologic materials are formally described using the Unified Soil Classification System (USCS). The USCS provides a structure to characterize unconsolidated geologic materials based on factors such as texture, grain size, sorting, density, plasticity, etc. The geologists conducting the visual inspections have been trained and are experienced in the use of the USCS.

All geologic work, including visual inspections, is conducted under the supervision of a geologist licensed by the State of Indiana.

2.0 Evaluation of CCB Sample Standards

Prior to initiating CCB visual inspections during the Remedial Investigation, sample standards of known CCB products from a coal-fired generating station were obtained from NIPSCO. Samples of fly ash, bottom ash, and boiler slag were reviewed by personnel conducting the visual inspections. A sample of mill slag (from iron/steel processes) was also obtained and reviewed. Notations were made of physical characteristics of these materials including grain size, grading, color, and other visually identifiable features. These characteristics were compared to those of native materials in the Pines Area of Investigation, including sand, peat, and clay.

To support additional visual inspections during the expanded Supplemental Soil Characterization, additional visual references will be prepared spanning a range of coarse and fine-grained mixtures.

3.0 Visual Inspection Locations

Initial visual inspections have been completed along accessible roads and alleys within established right-of-ways and on private properties where CCBs appeared to extend beyond the right-of-way and access was granted by the property owner. Additional properties to be inspected were identified following the Supplemental Soil Characterization activities.

Inspections are conducted by visually examining the shallow soils at regular intervals within the right-of-way and/or private property. Right-of-way inspection locations were spaced approximately 50 ft apart and located on both sides of roadways and alleys, if applicable, in an effort to determine lateral extents between the roadway and the end of the established right-of-way. At both previously-inspected and future private property locations, inspection locations are spaced 50 ft apart moving perpendicular to roadways as necessary. Visual inspection locations that fall within paved or inaccessible areas are moved to the nearest feasible location. At transitional areas the distance between visual-inspection areas are reduced to determine the approximate boundary of suspected CCBs. On private property, inspections will also take place in areas where the homeowner suggests that CCBs may be present.

A Trimble GeoXT or similar unit will be used for the mapping of visual inspection locations. Individual inspection locations will be documented in this manner, as well as the limits of areas of suspected CCBs. This information on locations will be downloaded into an ESRI Shapefile format for map generation.

4.0 Visual Inspection Logging

The following steps will be completed by trained AECOM geologists at each visual inspection location:

1. An initial scan of the surface will be completed to note ground surface conditions, local topography, proximity to roadways and driveways where suspected CCBs may be present, and for the presence of suspected CCBs at the surface.
2. A slotted soil recovery probe will be advanced by hand to a depth of approximately 6 inches below ground surface.
3. Soil collected in the probe will be inspected visually. Visual inspection will make note of soil characteristics including, but not limited to: color, texture, grain size, grading, plasticity, organic matter, and moisture. Mineralogy may be considered, as appropriate. In addition, soil structure and stratigraphy will be noted as an additional characteristic. The sample will be inspected with reference to the characteristics of the standards and native soils noted in Section 2, above. Soil characteristics will be used to determine if fill material is suspected CCBs. If necessary additional measures such as use of a hand lens or mild hydrochloric acid solution will be utilized to classify the soil. If soil collected in a single probe is inconclusive, additional samples will be collected in the immediate area as necessary to make a determination.
4. The inspection will make note where the amount of CCBs on a property appears to be minor or incidental, such as where CCBs may have been tracked onto the property from roadways or right-of-ways, in contrast to where CCBs were brought into an area in larger volumes for specific reasons such as filling or road base. The incidental nature of the CCBs will be indicated by a small proportion of CCBs mixed with other non-CCB material present only in the shallowest portion of the soil probe.
5. Where CCBs or suspected CCBs make up more than about 50% of the material in the core, the geologist will make a preliminary estimate the portions of fly ash and boiler slag/bottom ash that are present. To date, it has not been possible to make this determination in the field. Fly ash is very fine-grained and cannot be identified by sight. However, because of its fine-grained characteristics, texture will be used as a surrogate. Where significant amounts of CCBs are present, fine-grained

materials can be estimated as fly ash and coarser material as bottom ash/boiler slag. To aid in this determination, the geologist will be provided with approximately three standards for different ranges for mixtures (e.g., 25-75%, 50-50%, and 75-25% mixtures) to facilitate this visual determination in the field.

6. Soil will be returned to the sampling location and, to the extent possible, surface conditions restored.
7. The visual inspection location and, if applicable, the suspected CCB area boundary will be documented for mapping.

After completion of the visual inspection, the location will be noted to either contain or not contain suspect CCBs.

A field notebook will be maintained to note survey areas inspected and initial survey conditions. A visual inspection data sheet will be filled out for each inspection location. Data sheets will note sampling location, soil type, presence or absence of suspect CCB material, portion of fly ash and bottom ash/boiler slag if applicable, and ground cover type. A field drawn map will be generated during the visual inspection process showing approximate boundaries of the suspect CCB materials.

In the event that the ground is frozen inhibiting advancement of the soil probe, inspection activities will cease. If hard ground is encountered, sampling locations will be moved to the nearest accessible location.

5.0 References

ASTM. 1993. Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System). ASTM Standard D2487-93.

AECOM. 2005. Remedial Investigation/Feasibility Study Work Plan; Volume 2 – Field Sampling Plan. Pines Area of Investigation, AOC II, Docket No. V-W-'04-C-784. September 16, 2005.

