STANDARD OPERATING PROCEDURE NO. 03
SOIL X-RAY FLUORESCENCE FIELD ANALYSIS

1.0 PURPOSE OF PROCEDURE

The purpose of this procedure is to provide a general guideline for using the Innov-X handheld x-ray fluorescence (XRF) to screen soil samples. The operator's manual should be consulted for detailed operating procedures.

2.0 EQUIPMENT AND MATERIALS

- Olympus (formerly InnovX) Delta dp-4000 Handheld XRF
- National Institute of Standards and Technology (NIST) calibration standards
- 1-gallon clear plastic zip top bag (untextured)
- Sieve, size 9.5 mm to 11.2 mm

3.0 PROCEEDURE

3.1 Calibration

There are three different calibration methods which the XRF can run. Normally the Compton Normalization method is used and generally has good accuracy for concentration ranges from the parts per million level up to two to three percent concentrations. The XRF analyzer is delivered with a factory calibration generally based upon the Compton Normalization method, so calibration is not required to use the analyzer effectively. However, the quality control procedures should be followed, which include a check of the calibration. For startup procedures and usage please refer to the manufacturer instruction manual.

3.2 Sample Preparation

A composite sample will be collected in accordance with SOP-02. The 5-part composite points should be combined in a 1-quart zip top plastic bag. The sample should be thoroughly mixed by hand, tumbler, or hand shaker after removing large clumps or clods, rocks, vegetation, or foreign objects. Employ a coarse sieve to the samples, e.g., a mesh with opening of 9.5 mm to 11.2 mm (per ASTM categories) before they are bagged and tested with the XRF.

3.3 XRF Soil Analysis

For the USS Lead site, a one minute reading should be taken for each composite sample. The XRF unit should be set up to only provide lead and arsenic readings.

1. Before using the XRF for the day, check the NIST standard and record.
2. Collect samples in a clean, resealable, clear plastic bag making sure that sufficient sample exists in the bag to completely cover the window with a sample thickness of a minimum of 0.5 inch. The bag should have a smooth surface (no texture) and be in good condition without significant wrinkles or creasing. The soil must be homogenized to result in a quality reading.
3. Place the sample on a horizontal surface and press the XRF probe window against the sample.

4. To begin testing, pull the trigger or press Start on the screen. A red warning light will flash indicating that Xrays are being emitted. "Test in progress" and time elapsed will show on the screen.
5. When the time has elapsed and the test is complete, record the measurement on the data sheet.
6. To minimize the effect of sample heterogeneity, 5 shots will be taken of each sample and the median result will be used as the sample concentration. The probe window will be placed in multiple locations on the sample bag, in a five-one-a-die pattern, with a reading for each location (repeat Steps 3 through 5).
7. When the XRF analysis is complete, retain the sample to composite with the same depth interval from the other borings in the yard (if a laboratory sample is to be collected).
8. Complete the Field Reconciliation Form with the XRF readings.

3.4 XRF Quality Assurance

1. Energy standardization —Check sample twice a day (start and end of work day).
2. Perform an instrument check to verify that no contamination exists on the analyzer window or any other component that can be "seen" by the x-rays. These should be taken at least once per day and once per 20 samples.
3. Perform 1-minute verification using NIST standard reference samples. The difference between the XRF result for an element and the value of the standard should be 20 percent or less. Performed upon startup, at the end of the day, and periodically during testing. Innov-X recommends verification check every 4 hours.
4. Collect precision samples, at a 10 percent frequency. According to USEPA Method 6200, at least one precision sample should be run per day. A NIST standard reference sample should be analyzed for 7 to 10 replicate measurements of the sample and assessed by calculating the relative standard deviation (RSD) of the replicate measurements for the analyte. The RSD values should be less than 20 percent.
    a. RSD = (SD/Mean concentration) × 100
    b. RSD = Relative standard deviation for the precision measurement for the analyte
    c. SD = Standard deviation of the concentration for the analyte Mean Concentration = Mean concentration for the analyte
5. If directed by EPA, submit selected samples to the laboratory for confirmation analysis.

4.0 DATA RECORDING

XRF sampling information to be captured in iForms or similar includes the following:

1. Lead and Arsenic readings for the same XRF NIST standard before soil sampling and after soil sampling, e.g., at the beginning of the day and at the end of the day.
2. The characteristics of the soil being XRFed needs to be recorded. This is important in order to understand any potential matrix effect issues, e.g., sand versus dark, organic soil.
3. Lead and Arsenic readings for the particular Property ID, yard, SampleID, and subsample location (each of the 5 readings). For example, Z3-SO-MAIN1234-F-0018_a where "_a" represents the first of the five XRF error readings of the same composite sample.
4. XRF manufacturer, model, and serial number.
5. Information will be downloaded daily from each XRF unit used.

5.0 RADIATION SAFETY

1. Proper usage. Never point the instrument at another person. Never point the instrument into the air and perform a test. Never hold a sample in your hand to test that the sample.
2. Establish Controlled Areas. The location of storage and use should be of restricted access 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.
3. Specific Controls. The instrument should be stored, in a locked case, or locked cabinets when not in use. When in use, it must remain in the direct control of a trained operator.
4. 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. Under no circumstances should operators point the instrument at themselves or others.
5. Prevent Exposure to Ionizing Radiation. Reasonable measures, including labeling, operator training, and the concepts of time, distance and shielding should be implemented to limit radiation exposure to As Low As Reasonably Achievable (ALARA).
6. Personal Monitoring. Radiation control regulations may require implementation of a radiation monitoring program, where each instrument operator wears a film badge or thermal luminescent detector (TLD) for an initial period of 1 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.

6.0 PREVENTATIVE MAINTENANCE

- Standardize XRF every 4 hours and on startup.
- Refer to operation manual for recommended maintenance.
- Check batteries, and recharge as needed.
- Keep 4-5 foot area clear of people in front of the analyzer when analyzing a sample.
- Keep personnel more than 3 feet away from analyzer sampling window at all times.
- Keep instrument locked in case when not in use.
- Keep instrument in controlled possession at all times when not locked in case.

7.0 REFERENCES

USEPA. 2007. *Method 6200 Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment.*

Innov-X Systems, Inc. 2006. *Instruction Manual—Innov-X Systems alpha series x-ray fluorescence spectrometers.*Version 2.2.

STANDARD OPERATING PROCEDURE NO. 04
SAMPLING EQUIPMENT DECONTAMINATION

1.0 PURPOSE OF PROCEDURE

Standard Operating Procedure (SOP)-04 describes the guidelines for decontaminating of sampling equipment.

2.0 GENERAL REQUIREMENTS

A. Procedures for decontamination of field personnel will be specifically addressed in the Health and Safety Plan (HASP).

B. Decontamination activities will be documented to verify that proper procedures are followed. Documentation will be in accordance with the requirements specified in the RI/FS Work Plan.

3.0 SAMPLING EQUIPMENT DECONTAMINATION PROCEDURES

A. All sampling equipment that may contribute to the contamination of a sample will be thoroughly decontaminated before its initial use, unless specific documentation exists that the sampling equipment has been decontaminated.

B. Generally, sampling equipment can be cleaned by hand. Delicate equipment, such as the submersible pump, will be cleaned by hand. The following procedure is given as a typical sequence that will be modified based on site conditions.

C. Other sampling equipment that can be cleaned by hand will be decontaminated as follows:

1) Wash and scrub with detergent (non-ionic)
2) Tap water rinse
3) DI water rinse
4) Isopropanol rinse
5) Air dry

D. Steel tapes, water probes, transducers, thermometers, and water quality meters will be rinsed in distilled water or cleaned in a detergent solution and rinsed in DI water after each use.

E. Pumps will be cleaned in water/detergent solution, followed by a tap water rinse, and flushed with DI water after each use.

F. Use of high-pressure steam or hot water washing may be substituted for hand scrubbing if it effectively removes contaminants and soil and can be done safely without burning or contaminating the personnel. Special racks will be used to hold equipment during high-pressure washing. High-pressure washing will not be conducted on delicate equipment.

G. More “complicated” samplers require more “complicated” decontamination procedures.

Piston and other samplers with numerous internal parts should be avoided.

H. To the extent possible, sample tubing shall be either dedicated to a particular well/location or disposed after each use. Tubing that cannot be dedicated to a particular well/location or disposed shall be cleaned in water/detergent solution, followed by a tap water rinse, followed by a DI water rinse. Sufficient rinse water shall be used to flush out all the wash water and tap water rinse.

I. Sampling and downhole equipment that has come into contact with non-aqueous phase liquids (NAPLs) or high concentration dissolved-phase contaminants (10 ppm range VOCs) will also undergo a rinse with isopropanol to ensure removal of organics. The decontamination steps for high concentration organics are:

1) Wash and scrub with detergent (non-ionic)
2) Tap water rinse
3) Isopropanol rinse
4) DI water rinse.
5) Air dry

4.0 WELL MATERIALS DECONTAMINATION PROCEDURES

Well-casing materials not received in their original packaging, whether constructed of PVC, carbon or low carbon steel, or other materials, will be cleaned with a steam cleaner or high-pressure hot-water washer before they are installed. Well construction materials will be handled while wearing clean gloves.

STANDARD OPERATING PROCEDURE NO. 05
SAMPLE CONTAINERS AND PRESERVATION

1.0 PURPOSE OF PROCEDURE

Standard Operating Procedure (SOP)-05 describes the procedures required for preparation of sample containers, preservation of samples, and sample holding times as required in the RI/FS Work Plan or as otherwise specified. This sampling protocol will be implemented to maintain the quality and integrity of the samples to be analyzed.

2.0 GENERAL REQUIREMENTS

A. Sample containers and preservation methods will be selected and used to ensure the accuracy and validity of chemical and physical analyses. Materials or procedures that might cause overt disturbance, contamination, oxidation or other chemical decomposition or reaction of the sample will be avoided. This SOP addresses only containers and preservation of chemical analytical samples.

B. Sample preservation methods will adequately meet the requirements established in the QAPP.

C. Use of containers and preservation procedures will be consistent with the analytical procedures used by the analytical laboratory.

D. Preservation of soil samples will be limited to refrigeration.

These techniques are intended to retard biological action, retard hydrolysis of chemical compounds, keep metals in solution, and reduce volatility of constituents.

E. Environmental samples will be kept on ice or refrigerated to maintain the sample temperature at 4 +/- 2°C. Samples will not be frozen, and dry ice will not be used as the cooling agent (due to shipping restrictions).

F. Sample bottle lids will be hand-tightened.

3.0 CONTAINER TYPES

A. Containers in which sample water is collected will be constructed of materials that remain inert with respect to the suspected contaminants. The containers will have screw- type lids to assure adequate sealing of the bottles. Teflon inserts will be placed inside the lids of glass containers to prevent sample reaction to the lid and improve the quality of the seal.

B. The volumes of the containers used in sample collection are dependent upon the analytical requirements. Sampling personnel will verify volume requirements with the lab.

C. Sample containers will generally be supplied by the laboratory contracted to perform the analyses. In these cases, sample bottles will be cleaned before shipment to the field. Each bottle will be clearly labeled as to the type of sample it will contain (e.g., metals, VOCs, etc.).

STANDARD OPERATING PROCEDURE NO. 06
SAMPLE STORAGE, PACKAGING, AND SHIPMENT

1.0 PURPOSE OF PROCEDURE

Standard Operating Procedure (SOP)-06 describes the guidelines for sample classification, storage, packaging, and shipment to ensure proper handling of samples.

2.0 SAMPLE STORAGE

A. Samples to be sent offsite will be stored in a manner consistent with the requirement for sample preservation to maintain the quality of the sample. All samples collected will be labeled and immediately placed on ice in a cooler. Samples preserved by cooling will be stored in such a way as to maintain the acceptable range of temperature for the duration of the holding time.

B. Samples will not be stored on site for extended periods and will be protected from environmental extremes. Shipment to the laboratory will be completed as soon as possible and within the specified holding times for each particular analysis to allow analysis within the holding time limits.

C. If temporary storage is necessary, samples will remain in an area that has been designated as the "sample storage area," which will be locked and secured to maintain sample integrity and chain-of-custody requirements. Separate containers will be used to store or transport low, medium, and high concentration samples.

D. Samples will not be stored in refrigerators or other areas where food or drink may also be stored, and vice versa.

3.0 ENVIRONMENTAL SAMPLE PACKAGING

3.1 Regulatory Considerations

A. DOT regulations do not consider environmental samples hazardous. In addition, DOT has stated that dilute solutions of chemical preservatives are not subject to Hazardous Materials Regulations.

B. In accordance with 49 CFR 173.24(a), the following is required for all packages: "Each package used for shipping hazardous materials will be so designed and constructed, and its contents so limited, that under conditions normally incident to transportation:

1) There will be no significant release of the hazardous materials to the environment;

2) The effectiveness of the packaging will not be substantially reduced; and

3) There will be no mixture of gases or vapors in the package which could, through any credible spontaneous increase of heat or pressure, or through an explosion, significantly reduce the effectiveness of the packaging."

C. In accordance with 94 CFR 173.6(a), the following are required for all shipment by air: "Each package will be so designed and constructed, and its contents so limited, that under conditions normally incident to transportation:

1) There will be no significant release of materials to the environment.

2) Inner containers that are breakable (such as earthenware, glass, or brittle plastic) will be packaged to prevent breakage and leakage under conditions normally incident to transportation. Those completed packaging's will be capable of withstanding a 4-foot drop on solid concrete in the position most likely to cause damage. Cushioning and absorbent materials will not react dangerously with the contents.

3) For any packaging with a capacity of 110 gallons or less containing liquids, sufficient outage (ullage) will be provided to prevent liquid contents from completely filling the packaging at 130° F. The primary packaging (which may include composite packaging), for which retention of the liquid is the basic function, will be capable of withstanding, without leakage, an internal absolute pressure of not less than 26 pounds per square inch or no less than the sum of the absolute vapor pressure of the contents at 130° F (55° C) and the atmospheric pressure at sea level, whichever is greater.

4) Stoppers, corks, or other such friction-type closures will be held securely, tightly, and effectively in place with wire, tape, or other positive means. Each screw-type closure on any inside plastic packaging will be secured to prevent the closure from loosening due to vibration or substantial changes in temperature."

3.2 Shipping Containers

A. Sample containers will be placed inside a strong shipping container capable of withstanding a 4-foot drop on solid concrete in the position most likely to cause damage. A metal or plastic cooler (ice chest) with a hard plastic liner withstands this test. The drainage hole at the bottom of the cooler will be taped shut so that the contents from the broken containers or water from ice bags will not escape. The shipping container will be taped shut to form an adequate seal around the lid to prevent any leakage in the event that the cooler is turned over.

B. The shipping container will be marked "THIS END UP" with arrows indicating the proper upward position of the container affixed to all four sides. To prevent cross- contamination, the shipping container will be adequately cleaned between shipments.

3.3 Ice

A. Ice will be placed into 1-gallon size plastic bags (minimum 2-ml thick) and sealed tightly.

The amount of ice used will depend on the available space in the cooler, but 10 pounds per 20 quarts of cooler volume would be the minimum to assure sufficient cooling to less than 4°C. Alternatively, gel freezer packs (blue ice) will be used to cool the samples. Dry ice (CO_2) will not be used.

3.4 Glass Containers

A. The lid of the container will be tightened to assure an adequate seal. The container may be placed into a clear plastic bag that has a minimum thickness of 2-ml (many 1-quart and 1-gallon size zip-lock bags are suitable) and this bag sealed. They will be wrapped and cushioned in an inert packing material such as bubble wrap or shredded paper.

3.5 Plastic Containers

A. Plastic containers do not require individual cushioning material, but will be packed to prevent movement during transport. Caps will be tightly screwed on. These bottles may also be placed into a 2-ml thick plastic bag and the bag sealed.

4.0 SHIPPING OF SAMPLES

4.1 Environmental Samples

A. Environmental samples may be shipped to the testing laboratory by commercial common carrier or lab currier.

STANDARD OPERATING PROCEDURE NO. 07
SAMPLE CONTROL AND CUSTODY PROCEDURES

1.0 PURPOSE OF PROCEDURE

Standard Operating Procedure (SOP)-07 describes the guidelines for controlling sample identification and chain-of-custody (C-O-C) in order to maintain the quality and integrity of samples during collection, transportation, and storage for analysis as described in the Work Plan or as otherwise specified. Additional guidance on data management is provided in the Quality Assurance Project Plann (QAPP) and the Data Management Plan (DMP).

Sample personnel should be aware that a sample is considered to be in a person's custody if the sample meet the following conditions:

- It is in a person's actual possession.
- It is in view after being in a person's possession.
- It is locked up so that no one can tamper with it after having been in physical custody.

When samples leave the custody of the sampler, the cooler must be custody-sealed and possession must be documented.

2.0 EQUIPMENT AND MATERIALS

- Computer or tablet with Scribe or Scribe compatable software loaded.
- Laser printer with paper (8.5 × 11 inch) and ink cartridge (black).
- Sample nomenclature guidance.
- Scribe or scribe compatable generated sample identification.
- Digital sampling forms.
- Digital C-O-Cs.
- Field book.

3.0 PROCEEDURE AND GUIDLINES

For the USS Lead Site Remedial Action, the following sample management tasks will be completed for each sampling task:

- **Pre-construction residential soil sampling:** Scribe or similar will be pre-populated by the sample manager. The field team leader will update Scribe with the sample dates and times and will prepare the COCs and tags.
- **Air sampling during construction:** Scribe or similar will be pre-populated to the extent possible by the sample manager. The resident inspector collecting the samples will update Scribe with the sample dates and times and will prepare the COCs and tags.
- **Borrow source and stockpile sampling during construction:** Scribe or similar will be pre-populated to the extent possible by the sample manager. The resident inspector collecting the samples will update Scribe with the sample dates and times and will prepare the COCs and tags.

- **XRF Screening:** XRF readings will be entered in to the Field Reconciliation Form for each property. The XRF tool will also be downloaded to a site computer by the resident inspector each day and will be entered in to an application. The data will then automatically be uploaded into the project database.
- **Preliminary Data:** preliminary EDDs will automatically be loaded into the project database and screened by the project chemist. The project chemist will send the data via email to the project team.

3.1 Chain-of-Custody Forms

The chain-of-custody form must contain the following information:

- CASE NUMBER/CLIENT NUMBER
- EPA REGION: Enter Region "5".
- CERCLIS ID: FOR USS LEAD, USE "IND047030226".
- SPILL ID: FOR USS LEAD, USE "TBD".
- SITE NAME/STATE: "USS LEAD", "IN".
- PROJECT LEADER: ENTER THE PARSONS SITE MANAGER.
- ACTION: FOR USS LEAD, CHOOSE "REMEDIAL ACTION".
- SAMPLING CO.: "PARSONS" or Subcontractor.
- SAMPLE NO.
- MATRIX: DESCRIBES THE SAMPLE MEDIA (E.G., SOIL, WATER, ETC.).
- SAMPLER NAME: THE NAME OF THE SAMPLER OR SAMPLE TEAM LEADER.
- CONCENTRATION: LOW (L), LOW/MEDIUM (M) OR HIGH (H).
- SAMPLE TYPE: "GRAB" OR "COMPOSITE".
- ANALYSIS: THIS INDICATES THE ANALYSES REQUIRED FOR EACH SAMPLE.
- TAG NO.: This number appears on the bottom of the sample tag and includes a prefix ("5") followed by a series of numbers. The entire number must appear on the chain-of-custody form.
- PRESERVATIVE: Document what preservative has been added to the sample (e.g., "HCl", "Ice Only", "None").
- STATION LOCATION: THIS IS THE PARSONS STATION LOCATION IDENTIFIER.
- SAMPLE COLLECT DATE/TIME: USE MILITARY TIME.
- QC TYPE: THIS IS FOR FIELD QC ONLY, AND INCLUDES FIELD DUPLICATE, FIELD BLANKS, EQUIPMENT BLANKS, AND TRIP BLANKS.
- DATE SHIPPED: THE DATE THAT SAMPLES ARE RELINQUISHED TO THE SHIPPING CARRIER.
- CARRIER NAME: (E.G., "FEDEX").
- • AIRBILL: AIRBILL NUMBER USED FOR SHIPPING.
- • SHIPPED TO: THIS IS THE LABORATORY NAME AND FULL ADDRESS, INCLUDING THE LABORATORY CONTACT. IF THE CONTACT IS NOT
- KNOWN, USE "SAMPLE CUSTODIAN".
- CHAIN-OF-CUSTODY RECORD FIELDS: THE SAMPLER'S SIGNATURE MUST APPEAR IN THE "SAMPLER Signature" and the Relinquished By" fields. The date and

time (military time) must also be included. If additional personnel were involved in sampling, their signatures should appear in the "Additional Sampler Signature(s)" field. Although the samples are "relinquished" to the shipping carrier, the shipping carrier does not have access to the samples as long as the shipping cooler is custody sealed. Consequently, the shipping carrier does not sign the chain-of-custody form.

- SAMPLE(S) TO BE USED FOR LABORATORY QC: THIS IDENTIFIES WHICH SAMPLES ARE TO BE USED FOR MATRIX SPIKE/MATRIX SPIKE DUPLICATE ANALYSES.
- INDICATE IF SHIPMENT FOR CASE IS COMPLETE: USE "Y" OR "N".
- CHAIN-OF-CUSTODY SEAL NUMBER: RECORD THE CUSTODY SEAL NUMBERS THAT APPEAR ON THE Region 5 custody seals that can be found on the shipping container. There is usually a minimum of two per shipping container.

3.2 Labeling Proceedure

Each sample container will be identified with a uniquely numbered sample tag consistent with EPA Region 5 data management guidlines. Each tag will contain the following information:

- Case/SAS number (if required).
- The unique sample number for sample tracking.
- Parsons station location (i.e., the sample identifier).
- Date of sampling.
- Time the sample was collected (in military time).
- All parameters for which the sample will be analyzed.
- Preservative used (if any).
- Sample type (grab or composite).
- Sample concentration (low, medium, high).
- Sample matrix (soil, water, etc.).
- The signature of sample team leader.
- Identification when sample is intended to be used by the lab for matrix spike/matrix spike duplicate.

4.0 KEY ITEMS

- All sample containers must be properly tagged.
- Each cooler must have a chain-of-custody form and the samples in the cooler (as identified by the sample tags) must match what is on the chain-of-custody form.
- Each chain-of-custody form must be properly relinquished (signature, date, time).
- The custody seal numbers must be written on each chain-of-custody form.
- The shipping cooler must be custody sealed in at least two places.

STANDARD OPERATING PROCEDURE NO. 08
AIR SAMPLING

1.0 PURPOSE OF PROCEDURE

Standard Operating Procedure (SOP)-08 describes the guidelines for air sampling for arsenic and lead particulates.

2.0 GENERAL REQUIREMENTS

This SOP is applicable to perimeter and personal air sampling for Parsons staff, conducted during excavation activities. Air sampling refers to the use of an air sampling pump and collection media to obtain air samples of contaminants of concern. The air sampling pump causes air to be drawn through the collection media at a constant, predetermined flow rate. Samples must later be run through a separate analysis process in order to determine contaminant concentrations. Results are typically reported as a time-weighted average that is used to specify engineering controls and personal protective equipment requirements and to demonstrate compliance with hazard (exposure) limits.

Air samples will be collected and analyzed in accordance with the National Institute of Occupational Safety and Health (NIOSH) Method 7303 and analyzed by a laboratory that is accredited by the American Industrial Hygiene Association for that method.

3.0 EQUIPMENT AND SUPPLIES

- 0.8-micrometer cellulose ester membrane filter
- Air sampling pump
- Tygon or equivalent flexible tubing
- Primary calibration standard or a precision-rotameter calibrated against a primary calibration standard

4.0 PROCEDURES

Sampling Requirements:

- Pump flow rate: 1.95 to 2.05 liters per minute

4.1 Calibration

- Calibrate personal sampling pumps before and after each day of sampling, either using a primary calibration standard (bubble or graphite piston meter) or a precision-rotameter calibrated against a primary calibration standard.
- Pre-sampling Calibration:
    - Connect the sampling train to the calibration instrument.
    - When the flow rate is within 5 percent of the flow rate specified by the analytical method, repeat the procedure at least 3 times.
    - If all 3 readings are within 5 percent of the recommended flow rate, then average the readings to obtain your pre-calibration flow rate.

  - Document the calibration process on the digital Air Sampling Data Form.
- Post-Sampling Calibration: After the sampling period is completed, a post-calibration is required prior to the pump battery being recharged. The process is similar to the pre-calibration process, except the flow rate is not to be adjusted.
  - Connect the sampling train to the calibration instrument and record 3 flow rate readings and average.
  - Record the average flow rate on the Air Sampling Data Form as the post-calibration flow rate.

4.2 Sample Collection

- Perform the pre-calibration to determine the flow rate as discussed above.
- For sample collection, the pump should be turned on immediately before placing on the worker or once mounted to a perimeter air monitoring station. Personal air samples are collected in the breathing zone of a worker, which is defined as a half-sphere around the head of the employee being monitored and forward from the plane of the shoulders.
- Record the start time.
- If the sampling pump is turned off during breaks or lunch, the start and stop times must be recorded on the Air Sampling Data Form.
- At the completion of the day or sampling period, stop the pump and record the time. The total sampling time, in minutes, should be determined from the start and stop times.
- Perform the post-sampling calibration as discussed above to determine the post-calibration flow rate.
- The next step is to compare the pre- and post-calibration flow rates to determine the sample flow rate.
  - If the pre- and post-calibration flow rates are within 5 percent of each other, then average the two to determine the sample flow rate.
  - If the difference between the pre- and post-calibration is greater than 5 percent but less than 10 percent, then the lower of the two flow rates will be used as the sample flow rate.
  - If the comparison of the pre- and post-calibration flow rates indicates a difference of greater than 10 percent, then the difference is too great to accurately estimate a flow rate and the sample must be voided.

4.3 Field Blanks

- The blanks should be opened but not used to take samples. They should be handled in the same manner as any sampling media used in sampling air contaminants, with the exception that no air is drawn through them.
- One blank should be submitted with each 10 samples for any given analysis/sampling period.

4.4 Sample Handling and Shipping

- Record sample information on the filter cartridge. Place the labeled filter cartridge in a clean zip-top plastic bag labeled with the sample information.

- Record the sample information on the chain of custody form provided in Attachment 1.
- Arsenic and lead samples are not required to be shipped on ice.

APPENDIX D
VANTA XRF DOCUMENT

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Using a Vanta XRF for decision-making at Zone 2, USS Lead

USEPA FIELDS Group

John Canar

Chuck Roth

Lead

Using Olympus Vanta (SN 803028 or SN 803032)

Lead

Decision criteria: median of five XFR readings < 261ppm

A median XRF Lead value of 261ppm has at most a 5% chance of being at or above 400ppm had that sample been sent to the laboratory.

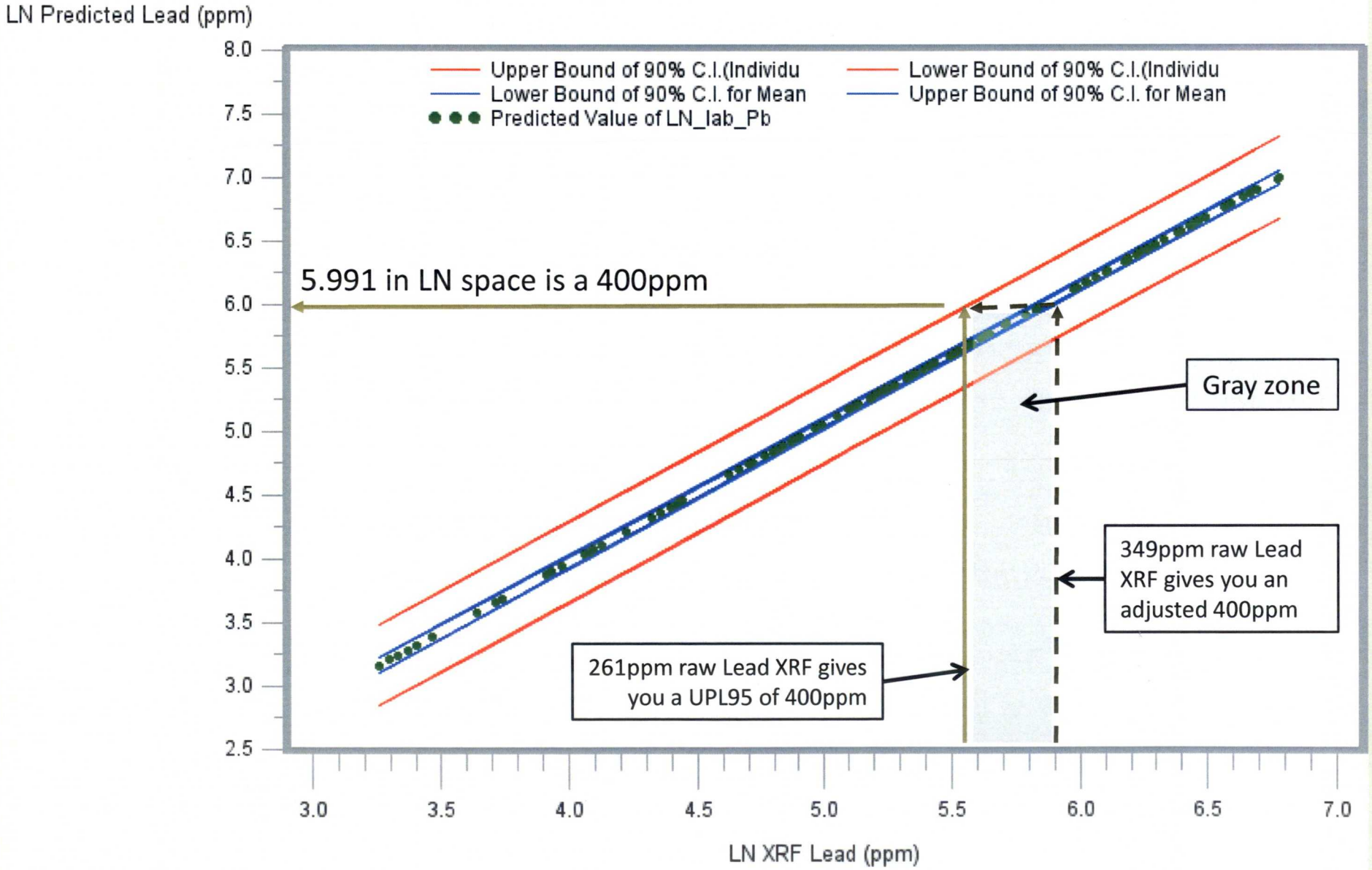
Potential options:

- dig an addition 3-6 inches if a confirmation composite sample has a median XRF Lead value of 261ppm or greater. Then collect a new composite sample.

- send the sample to the laboratory with a 24-hour turn-around if a confirmation composite sample has a median XRF Lead value between 261ppm and 349ppm. (Where 349ppm is the median XRF Lead value that adjusts to a 400ppm via regression.)

Regression of the Natural Log of Lead Lab and XRF values

Regression diagnostics
Vanta XRFs (803028 and 803032)
USS Lead Superfund Site: Zone 2



Upper Prediction Limits and Gray zone explained (Lead):

The UPL (upper prediction limit) provides a very good estimate of what the lowest XRF Lead value can be in order to be 95%* confident that in future sampling that value would be less than 400ppm had that sample been sent for Laboratory analysis. The current UPL value for the median Lead XRF is 261ppm. Hence, we'd expect only 5%* of samples with an XRF value less than 261ppm to be 400ppm or above in the lab.

The range of XRF values between the UPL and the adjusted Lead XRF value is the gray zone. This is the zone in which a project manager may choose to send samples to the Laboratory to determine if the sample is above or below 400ppm. For the current regression results, this zone has XRF Lead values between 261ppm and 349ppm, where 349ppm is the median Lead XRF value that adjusts to a 400ppm via regression.

* We use 95% and 5% because we're splitting the 10% for both sides of the UPL and LPL lines.

False negatives:

- 0% false negatives (0 of 130 samples) based on 261ppm
- a false negative occurs when the median Lead XRF is <261ppm but the laboratory value is 400ppm or greater

Arsenic

Using Olympus Vanta (SN 803028 or SN 803032)

Arsenic

Decision criteria: median of five XFR readings < 14.5ppm

A median XRF Arsenic value of 14.5ppm has at most a 5% chance of being at or above 26ppm had that sample been sent to the laboratory.

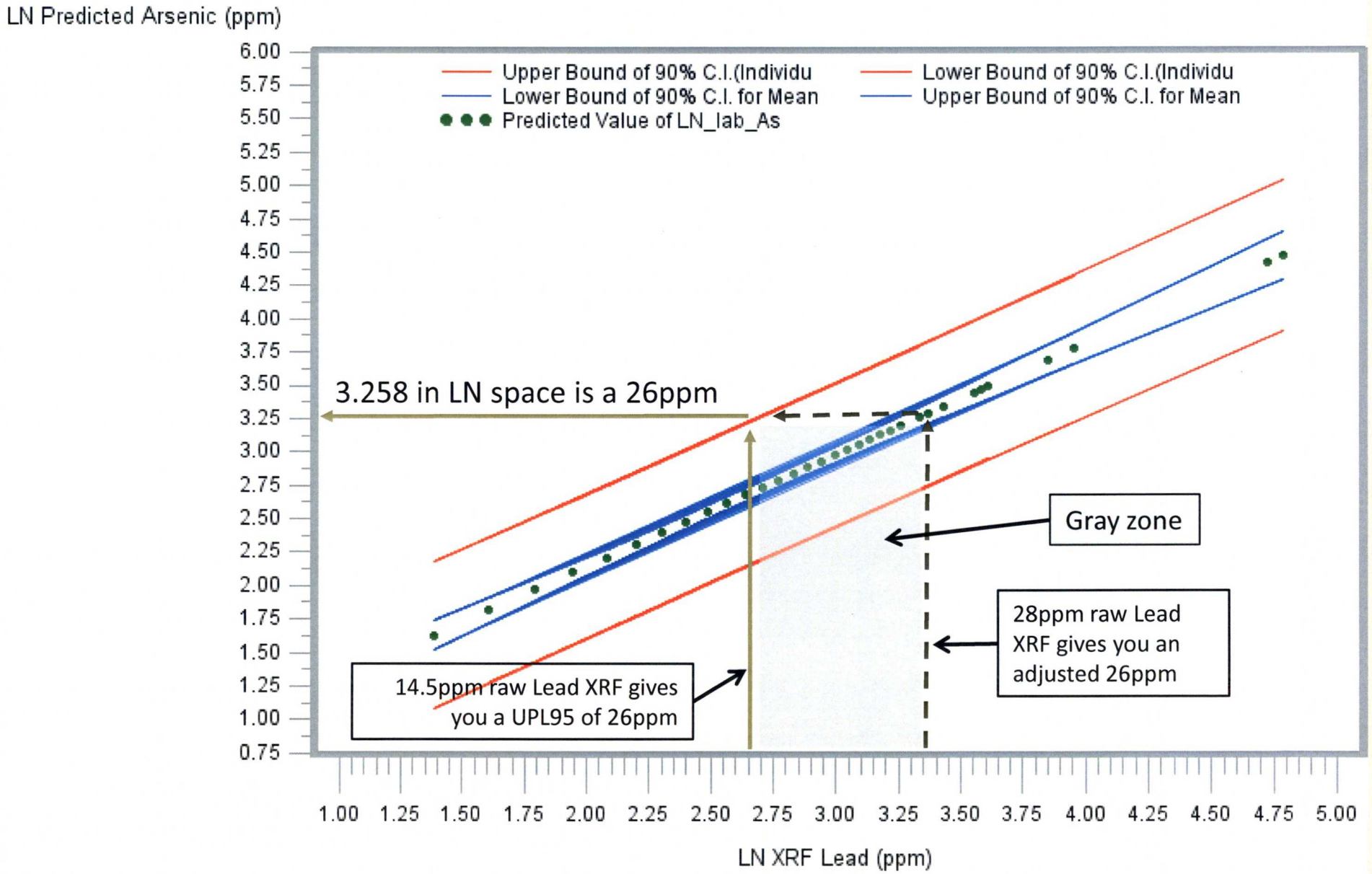
Potential options:

- dig an addition 3-6 inches if a confirmation composite sample has a median XRF Arsenic value of 14.5ppm or greater. Then collect a new composite sample.

- send the sample to the laboratory with a 24-hour turn-around if a confirmation composite sample has a median XRF Arsenic value between 14.5ppm and 28ppm. (Where 28ppm is the median XRF Arsenic value that adjusts to a 26ppm via regression

Regression of the Natural Log of Arsenic Lab and XRF values

Regression diagnostics
Vanta XRFs (803028 and 803032)
USS Lead Superfund Site: Zone 2



Upper Prediction Limits and Gray zone explained (Arsenic):

The UPL (upper prediction limit) provides a very good estimate of what the lowest XRF Arsenic value can be in order to be 95%* confident that in future sampling that value would be less than 26ppm had that sample been sent for Laboratory analysis. The current UPL value for the median Arsenic XRF is 14.5ppm. Hence, we'd expect only 5%* of samples with an XRF value less than 14.5ppm to be 26ppm or above in the lab.

The range of XRF values between the UPL and the adjusted Arsenic XRF value is the gray zone. This is the zone in which a project manager may choose to send samples to the Laboratory to determine if the sample is above or below 26ppm. For the current regression results, this zone has XRF Arsenic values between 14.5ppm and 28ppm, where 28ppm is the median Arsenic XRF value that adjusts to a 26ppm via regression.

* We use 95% and 5% because we're splitting the 10% for both sides of the UPL and LPL lines.

False negatives:

- 0% false negatives (0 of 111 samples) based on 14.5ppm
- a false negative occurs when the median Arsenic XRF is <14.5ppm but the laboratory value is 26ppm or greater

XRF standards

XRF 2711a standard and blank:

Ranges for the XRF standard (2711a) for the Olympus Vanta units #803028 and #803032 were developed from 9 samples taken over three days on the 2711a standard. The +/-20% range, +/-2 σ, and 25th-75th percentiles were calculated for each unit. The differences between the two units are fairly small for both Lead and Arsenic. Hence, a single error range can be employed for the two XRF units. The FIELDS Group recommends using the +/-20% of the mean. Those values are shown below:

Lead +/-20% error = 1,191 ppm to 1,776 ppm
Arsenic +/-20% error = 84 ppm to 121.5 ppm

The FIELDS Group recommends reading the 2711a standard three times at the beginning of the "day" and reading the standard again, three times, at the end of the "day". The Lead and Arsenic concentrations from reading the 2711a standard must be within the above ranges to demonstrate that the Vanta XRF units are working properly. Additionally, a blank should also be read three times at the beginning of the day and at the end of the day. XRF concentration values from the blank will demonstrate that the Vanta unit is not "contaminated" and, hence, is not reading metal concentration values. Additionally, a "cal" check should be done at the beginning of each "day".