

Standard Operating Procedure for:

X-MET3000TXS+ Handheld XRF Analyzer
(XRF.doc)

Missouri State University

and

Ozarks Environmental and Water
Resources Institute (OEWRI)

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1 Identification of the test method

Operation of the X-MET3000TXS+ handheld elemental analyzer for heavy metal screening and measurement of contaminated soil. The instrument is classified as a portable hand-held open-beam X-ray tube based analytical X-ray device.

2 Applicable matrix or matrices

This instrument can be used for natural soil samples.

3 Detection Limit

The detection limits for this instrument are: Antimony = 19ppm, Arsenic = 7ppm, Barium = 60ppm, Cadmium = 23ppm, Cobalt = 18ppm, Copper = 15ppm, Chromium = 40ppm, Gold = 16ppm, Lead = 15ppm, Mercury = 13ppm, Nickel = 16ppm, Selenium = 4ppm, and Zinc = 8ppm.

4 Scope of the test method

- 4.1 This procedure will be used as a field and laboratory reference guide for the collection of heavy metal data from soil samples.
- 4.2 The instrument measures antimony, arsenic, barium, cadmium, cobalt, copper, chromium, gold, lead, mercury, nickel, selenium, and zinc concentrations in natural soil sediments from cutbank profiles, soil cores, or prepared samples in a laboratory setting.
- 4.3 The procedures described here present principal components of the instrument, pre-operating instruction, actual sample analyses, instrument settings, safety components associated with ionizing radiation, and reporting procedures. The user should review the instrument manuals for further information about the instrument.

5 Summary of test method

- 5.1 The X-MET3000TXS+ handheld elemental analyzer is based on energy dispersive X-ray fluorescence technology and uses an X-ray tube as the source of X-rays. The X-ray tube has a Ru target, 40kV HV supply and a high resolution high count-rate PentaPIN[®] SiPIN detector system which allows for fast and accurate soil analyses. The instrument is operated by a HP iPAQ PDA with a Windows Mobil 5.0 operating system.
- 5.2 X-rays produced by the instrument, bombard the atoms of the target sample. Photons collide with electron shells and electrons move. The movement of the electrons decreases the atom's energy and an X-ray photon is emitted. The energy of the photon being emitted is approximately equal to the decrease in the atom's energy and the X-rays fluoresce. Each element produces uniquely defined energy changes and the quantities of electrons in various shells are proportional to the number of atoms of the element in the sample. The detector system measures the fluorescent X-rays and the energies that are produced from each X-ray. The net intensities of the X-rays are converted into element concentrations using empirical coefficients and linear and polynomial multi-parameter regressions derived from calibration standards.

- 5.3 The instrument comes pre-calibrated from the manufacturer. The Universal Soils Fundamental Parameters calibration for assay of 24 common alloying elements was used. This calibration was based on Certified Reference Materials and Reference Materials as defined in ISO 30:1981 standard.
- 5.4 All technicians using the X-MET3000TXS+ must have read this SOP, have been trained in the appropriate use of the instrument by the responsible party or other designated authority, have signed an acknowledgement form indicating that they have read and understand the SOP, and must provide necessary information for record keeping associated with use of this instrument.
- 5.5 Charged batteries are inserted into the instrument or the instrument is plugged into an outlet, the PDA is locked into position, the power key is turned to the "ON" position to activate the instrument, the yellow indicator light comes on, the peltier cooler and X-ray tube stabilize, the PDA is turned on, and the software is used to set up sample data storage information and to operate the instrument.
- 5.6 Timed Assay's are used to increase accuracy and precision of soil analyses. The instrument is activated by a single press of the trigger and measures the sample for a preprogrammed time which decreases error associated with analysis duration.
- 5.7 In the field soil profiles are screened in-situ by creating a divet in the soil that encompasses the entire analysis window, covering the soil divot with pre-measured plastic film, positioning the analysis window into the divot, ensuring a correct measurement angle of 90°, activating the trigger, and performing a timed assay. Soil samples screened and collected in the field are dried, ground, and sieved and placed in plastic freezer bags in the laboratory. The instrument is positioned in the bench top stand to ensure a correct measurement angle of 90° and is plugged into an outlet to reduce wear on battery supply. The soil samples are analyzed in the laboratory by placing the entire bag of sample in the sample bag holder, closing the door of the holder, activating the trigger, and performing a timed assay.
- 5.8 The sample analysis spectra and results are stored on the PDA, the software program is exited, the PDA is powered off, the power key is turned to the "OFF" position to deactivate the instrument, and analysis spectra and results are downloaded to a PC for further processing.

6 Definitions

- 6.1 Acute Dose: A large amount of radiation received in a short period of time that results in physical reactions due to massive cell damage.
- 6.2 Analytical batch: The set of samples processed at the same time.
- 6.3 Blank: The plastic film used to cover the sample analysis divet during field determinations or the exact brand of plastic freezer bag containing the samples during laboratory analysis. Only the plastic film or plastic bag is measured to

determine additions or interferences present from field procedures, storage procedures, apparatus, or the laboratory environment.

- 6.4 Backscattering: Deflection of radiation in a scattering process through an angle greater than 90°. X-rays that reflect back to the detector and scatter in the direction of the instrument operator.
- 6.5 Bremsstrahlung: X-rays or “braking” radiation produced by the deceleration of electrons. X-ray tubes produce bremsstrahlung as accelerated electrons interact with the target material.
- 6.6 Chain of Custody (COC): Used to describe the written record of the collection, possession and handling of samples. Chain of custody forms should be completed as described in the Chain of Custody SOP # 1030R01. Chain of custody (COC) forms are located on a board in Temple Hall 125.
- 6.7 Characteristic X-rays: A form of electromagnetic radiation with a wavelength in the range of 10 to 0.01 nanometers, corresponding to frequencies in the range 30 PHz to 30 EHz. X-rays are a form of ionizing radiation, have no mass or electrical charge, and are emitted from electrons during electron shell transfers. X-rays can travel several hundred feet in air, have the highest penetrating power of the types of ionizing radiation, and are best shielded by using concrete, lead, or steel.
- 6.8 Check Standard: Standard sediment sample of known concentration and purity that produce consistent concentrations of analytes. These standards are used to check instrument performance.
- 6.9 Chronic Dose: A small amount of radiation received continually over a long period of time which can be from natural background sources.
- 6.10 Controlled Area: An area where access, occupancy, and working conditions are monitored by the responsible person to control radiation exposure to personnel.
- 6.11 Fundamental Parameters (FP): An Assay & Grade method used for analyses performed using this SOP that measures the elemental concentrations of unknown samples. Specifically, “Soil FP” will be used for analyzing heavy element concentrations in soil.
- 6.12 Ion: An atom that has lost or gained an electron.
- 6.13 Ionizing Radiation: Energy in the form of waves or moving subatomic particles that has enough energy to remove electrons from neutral atoms or molecules. Ionizing radiation has the potential to alter the chemical structure of living cells.
- 6.14 Laboratory Duplicate (LD): Two samples taken at the same time and placed under identical circumstances and that are treated identically throughout field and laboratory procedures. Analysis of laboratory duplicates indicates the precision associated with sample collection, preservation, and storage as well as laboratory procedures.

- 6.15 Method detection limit (MDL): The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.
- To calculate the MDL:
 - Prepare triplicates of two sediment samples with low organic matter. The laboratory director or supervisor will choose appropriate samples to use to determine MDL.
 - Analyze all samples.
 - Include all sample processing steps in the determination.
 - Calculate the standard deviation (s).
 - From a table of the one-sided t distribution select the value of t for $7 - 1 = 6$ degrees of freedom at the 99% level. This value is 3.14
 - The product 3.14 times s is the desired MDL.
- 6.16 Minimum Quantification Interval: The lowest level that can be quantitated accurately and is generally defined as four times the method detection limit = $4(\text{MDL})$.
- 6.17 Primary Beam: Ionizing radiation from an X-ray tube that is directed through an aperture in the radiation source housing for use in conducting X-ray fluorescence measurements.
- 6.18 Radiation: Energy in the form of waves or moving subatomic particles that is classified as non-ionizing and ionizing.
- 6.19 Relative Percent Difference (RPD): calculated as the difference between a sample and duplicate results, divided by the average of the sample and duplicate results, multiplied by 100%.
- 6.20 Sievert (Sv): Current unit of measure for radiation and the SI unit for dose equivalence that takes into account the energy absorbed (dose), the quality of radiation, and the biological effect of different types of radiation in the body. $1\text{Sv} = 100\text{rem}$.
- 6.21 System Barrier: The portion of an area that clearly defines the transition from a controlled area to a radiation area and that provides the necessary shielding to limit the dose rate in the controlled area during normal operation.

7 Interferences

- 7.1 Window distance from the sample and angle of measurement are important aspects of these analyses. In field determinations the analysis window will be placed directly on the plastic film that is placed directly on the soil sample. The appropriate measurement angle of 90° and timed assays will be used.
- 7.2 Penetration depth is important for some elemental determinations. Precise determination of cadmium requires at least 6mm of soil depth.

- 7.3 The sample should be as smooth and clean as possible. Rust, oil, and dust buildup in and around the analysis window will interfere with element analyses.
- 7.4 The sand fraction of a sample can affect its homogeneity which can alter elemental determinations. To determine the homogeneity of the sample, it should be dried and measured at least three times and each element should be averaged. Particles with diameter >2mm should be removed from the sample and the samples should be re-analyzed at least three times and each element should be averaged. If the average of the 2 sets of measurements differ <20% the sample is considered homogeneous and “semi-quantitative”. If the average of the 2 sets of measurements differ >20% the sample is considered heterogeneous and particle size is affecting elemental determination. Additional sieving should take place and will be dictated by the project manager as well as the laboratory supervisor.
- 7.5 Soil moisture can interfere with elemental determinations. Sample moisture should not be higher than 10-20%. Field determinations will produce less accurate analyses if moist samples are analyzed. All determinations in the laboratory will be performed using oven-dried sediments.
- 7.6 X-ray spectra can be very close in energy and can interfere by giving overlapping peaks. Standards used during manufacturer calibration enhance the isolation of the specific peak of each element. In addition, Oxford Instruments has a unique spectrum analysis algorithm that corrects for interfering absorption coefficients and their effects on calculated concentrations by monitoring the X-ray intensity from the interfering elements and applying correction factors derived during calibration.
- 7.7 Compton or back scattering peaks can interfere with a peak of an element. The sample bag holder will be used in the laboratory and the background plate should be used when ever possible in the field to minimize interference.

8 Health and safety

- 8.1 Researchers, faculty members, staff members, and students will use X-ray producing equipment in accordance with their departments’ established procedures and the requirements of this standard practice. Only trained personnel are allowed to operate this instrument.
- 8.2 The responsible person for this instrument is the director of the Ozarks Environmental and Water Resources Institute.
- 8.3 The manufacturer, Oxford Instruments, designed the X-MET3000TXS+ to conform to ANSI N43.2-2001 and the 21CFR 1020.40 safety requirements for cabinet X-ray systems with the exception of using a totally enclosed beam. Switches, required keys, indicator lights, and an infrared beam safety sensor were designed to prevent operators from being exposed to an open X-ray beam. The instrument was tested by TUV Rheinland against safety requirement of IE 61010-1, “Safety Requirements for Electrical Equipment for measurement, Control, and Laboratory

use, Part 1 General Requirements". The instrument passes the ionizing radiation leakage requirements in IEC61010-1, section 12.2.1 of <1 µSv/hour at 100mm.

- 8.4 Ionizing radiation can damage chromosomes of a cell and incomplete repair of that cell may result in the development of cancerous cells. A red LED indicates that the instrument is producing X-rays. The instrument's X-ray beam is collimated through an aperture that is approximately 0.14 inch in diameter.
- 8.5 The instrument's safety system consists of a key lock, three lights, a trigger to activate X-rays, and an infrared sensor. The key lock controls power to all components and must be turned on before any actions can take place. The yellow light indicates that the HV supply is energized when illuminated. The trigger must be activated for the instrument to produce X-rays. The infrared beam interrupter sensor must be covered with a sample for the instrument to produce X-rays. The red light indicates that X-rays are being generated when illuminated.
- 8.6 The X-ray tube and high voltage power supply are sealed in a fluid filled sub-assembly. The X-ray tube is shielded by a variety of materials to minimize any stray X-ray radiation. The X-ray tube and shielding material are mounted in the instrument housing and the housing is closed using tamper-proof fasteners.
- 8.7 Radiation dose from an x-ray device must not exceed 20mSv per year averaged over five years as developed by the International Commission of Radiological Protection (ICRP-1990), the Biological Effects of Ionizing Radiation (BEIR) Committee, the US Environmental Protection Agency (EPA), and the National Council of Radiation Protection (NCRP).
- 8.8 All technicians will practice As Low As Reasonably Achievable (ALARA) program guidelines that were designed to prevent unnecessary exposures to humans or the environment. Technicians will limit the amount of time spent in a radioactive area because the less time that a person is exposed the smaller dose is received. Technicians will maintain the maximum possible distance from the radiation source because as distance increases exposure rate decreases. Technicians will follow all precautions to shield themselves from exposure. All persons not directly involved in operating the instrument will remain three feet away from the instrument during analyses. The sample bag holder attachment shields others in the laboratory and beyond the walls of the laboratory from exposure which is why **samples will only be analyzed using the sample bag holder while in the laboratory.** Walls and partitions are not safe shields. Radiation does bounce off surfaces and extends around corners. The beam of radiation, when looking at the instrument from the top, exits the instrument at approximately 45° to the left. Never point the instrument at any person when the probe is activated or otherwise.

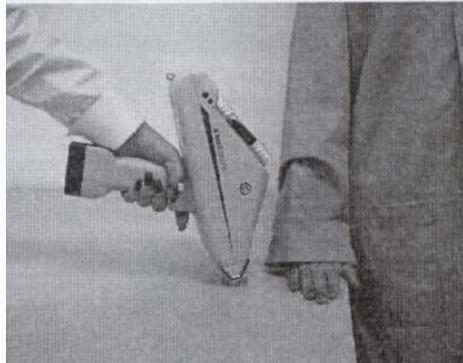


Figure 1. Incorrect Procedure: All persons not directly involved in operating the instrument will remain three feet away from the instrument during analyses.

- 8.9 Small sample volume that do not allow the entire measurement window to be covered present additional risk because part of the primary radiation could go through the sample un-attenuated. The safety shield for small samples will be used during analyses of samples that do not cover the entire measurement window.
- 8.10 The figure below illustrates the radiation profile of the instrument that was obtained by measuring doses from backscattering using a low scintillation probe [NaI(Tl)]. No sample was in place and an artificial instrument condition was used that did not contain the safety interlocks.

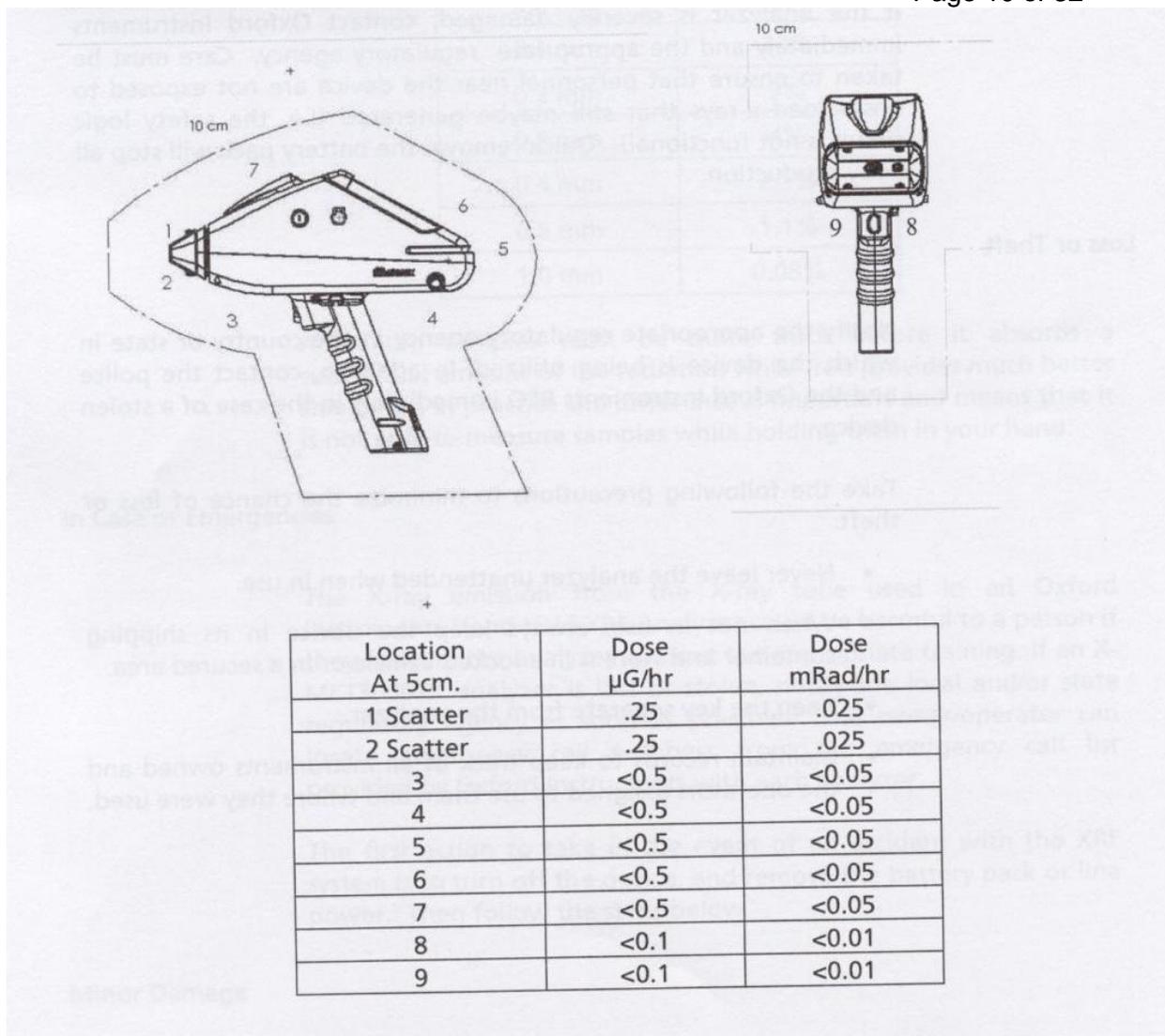


Figure 2. Background Radiation Measurements: These measurements indicate that the air kerma rate at 5cm from any accessible surface was lower than 4.38 μGy/hr.

- 8.11 The bone in the finger will absorb radiation about 3-5 times more than soft tissue, so DO NOT HOLD A SAMPLE IN FRONT OF THE MEASUREMENT WINDOW WITH THE FINGERS IN THE DIRECT BEAM OR DIRECT THE BEAM AT ANY PART OF THE HUMAN BODY. Always use the background plate.

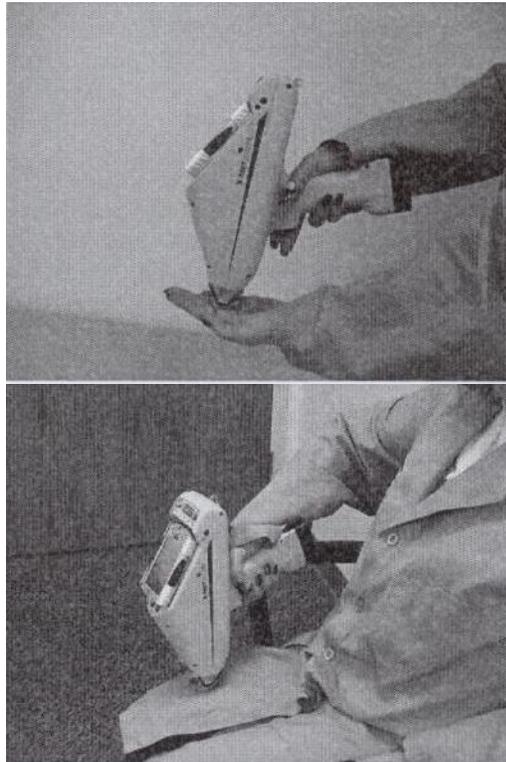


Figure 3. Incorrect Procedure: Do not hold a sample in front of the measurement window with the fingers in the direct beam or point the beam at any part of the body.

- 8.12 Always place the instrument in contact with the sample to minimize excess backscatter. Never hold the instrument away from the sample.

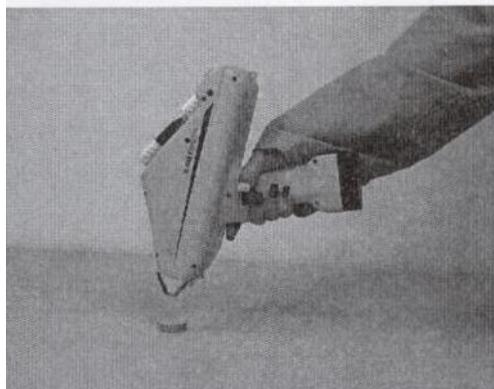


Figure 4. Incorrect Procedure: Never hold the instrument away from the sample.

- 8.13 The infrared beam safety sensor located at the aperture and detector end of the instrument (nosepiece) will not permit X-rays to be generated unless the infrared beam is covered by a solid object. Never defeat the IR sensor, defeating this safety feature could result in over-exposure of the operator.



Figure 5. Incorrect Procedure: Never defeat the IR sensor, defeating this safety feature could result in over-exposure of the operator.

- 8.14 The appropriate measurement angle of 90° will be used at all times to prevent additional risk associated with primary beam angle and backscattering.

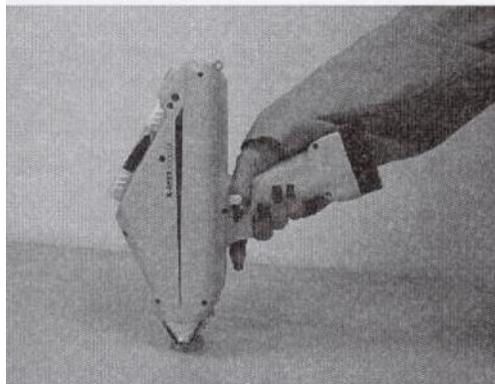


Figure 6. Correct Procedure: Use of the appropriate 90° angle of measurement.

- 8.15 A beryllium window is used in the radiation detector inside the instrument. Corrosion of beryllium may occur if it is exposed to moisture, particularly when ions such as chlorine, sulphates, copper, or iron are present. Do not store the instrument in high humidity areas or in circumstances where atmospheric condensation may occur. Beryllium and its compounds are toxic, but the solid form of beryllium which is used in the detector window of this instrument, poses no health hazard.
- 8.16 The instrument will be tested every six months to ensure that the ON/OFF mechanism is working properly. If the instrument fails, an OIA representative will be contacted immediately for instructions.
- 8.17 Instrument use and instrument service records will be maintained for two years or until the ownership of the instrument is transferred or the instrument is decommissioned. If the instrument is transferred to non-MSU persons, that transfer must be reported to regulatory agencies and the OIA representative. The QA/QC manager will maintain records and report necessary information regarding the instrument.

- 8.18 Any damage to shielding will be immediately recorded and that information will be given to the QA/QC manager.
- 8.19 NEVER REMOVE LABELS FROM THE INSTRUMENT. Removal of labels will void all warranties.
- 8.20 Never attempt to override any safety feature.
- 8.21 The battery can be pulled from the instrument during an emergency to terminate power.
- 8.22 Timed Assay measurements will be used for all sample analyses. These measurements allow the user to activate the trigger by pressing the trigger once versus keeping the trigger pressed during the entire measurement window. The instrument measures the sample for a specified time each time and increases precision and safety.
- 8.23 Dosimetry is available to any student using the instrument or in the laboratory while the instrument is being operated.
- 8.24 The primary operator is responsible for the instrument and should always be in the operators possession either in direct sight or in a secure area. NEVER LEAVE THE KEY IN AN UNATTENDED ANALYZER. Notify the QA/QC manager at 417-836-3198 if the instrument is stolen or lost. The QA/QC manager will contact the police and appropriate regulatory agencies.
- 8.25 The instrument will be stored in a locked cabinet in the laboratory. The key will be stored by the QA/QC manager and in a separate location to avoid unauthorized usage.

9 Emergency Procedures and Call List

- 9.1 The first action to take in the event of an accident involving the instrument is to stop all X-ray production by turning the instrument off and removing the battery pack or unplugging the instrument.
- 9.2 If any hardware item is damaged, even if the instrument remains operable, contact the Oxford Radiation Safety Officer (RSO) at 1-847-439-4404. Use of a damaged instrument may lead to unnecessary radiation exposure and inaccurate analyses.
- 9.3 Questions and additional instruction should be directed to the QA/QC manager at 417-836-3198.
- 9.4 The QA/QC manager will contact university emergency personnel or off-campus contacts as necessary.

10 Personnel qualifications

Soil parameters will be collected by Missouri State University (MSU) graduate assistants who have received appropriate training, prior coursework, and field experience regarding the collection of soil parameter data, and who are familiar with all of MSU's sample handling and labeling procedures. All operators of the X-MET3000TXS+ will be trained and will know all safety and operation procedures before using the instrument.

11 Equipment and supplies

- 10.1 X-MET3000TXS+ Handheld Elemental Analyzer, Oxford Instruments, 945 Busse Road, Elk Grove Village, IL, 60007, Telephone: 847-439-4404, www.oxford-instruments.com in shipping case
- 10.2 Power Supply:
 - Field - all three batteries charged as indicated by a green status light while plugged into the charger
 - Laboratory - power cord, PDA AC adapter and plug
- 10.3 Charged PDA (as indicated by a solid amber light when the PDA is plugged into the charger) with Stylus and protection cover
- 10.4 Sample Holder:
 - Field – sample film and bags
 - Laboratory – sample bag holder accessory, safety shield for small samples
- 10.5 Background Plate
- 10.6 Soil Check Sample “2 A1” Oxford Instruments
- 10.7 Power Key and PDA lock key
- 10.8 Field or Laboratory Book, Pen
- 10.9 Instrument Use Record

12 Reagents and standards

- 11.1 Soil Check Sample “2 A1” Oxford Instruments
- 11.2 Blank: Sample Film or Sample Bag
- 11.3 Background Plate

13 Sample collection, preservation, shipment and storage

The analyses are conducted *in situ* and only samples containing a predetermined level of contaminants will be collected and transferred to the laboratory. Samples will be placed into plastic bags and labeled. There are no special provisions for shipment.

Sample bags should be opened and placed into 60° C oven immediately upon return to laboratory.

14 Quality control

- 14.1 Quality control program: The minimum requirements of the quality control program for this analysis consist of an initial demonstration of laboratory capability and the periodic analysis of blanks and standard check samples as a continuing check on performance. The laboratory must maintain performance records that define the quality of the data that are generated.
- a. Analyses of laboratory blanks are required to demonstrate freedom from contamination.
 - b. The laboratory shall, on an ongoing basis, demonstrate through calibration verification and analysis of the ongoing precision and recovery sample that the analysis system is in control.
 - c. The laboratory should maintain records to define the quality of data that is generated.
- 14.2 Initial demonstration of performance. The following must be satisfied before the analytical procedure may be used for samples and before a new analyst may analyze samples.
- a. Method Detection Limit (MDL) – To establish the ability to detect the analyte, the analyst shall determine the MDL by carrying through 7 or more blanks (sample bags and plastic film) through the analytical procedures. The average value, \bar{X} , and the standard deviation of the values, s , shall be calculated. The MDL is equal to $3s$ (3 x standard deviation). MDL's will vary with element and batch.
 - b. Initial Precision and Recovery – To establish the ability to generate acceptably precise and accurate results, the manufacturer performed an extensive calibration of the instrument using various masses of solids which illustrate the instrument's response to analyte concentration. The instrument calibration should remain stable for the life of the instrument.
- 14.3 Soil Check Standard "2A1" and blank will be measured at the start of the analytical cycle and after every twenty samples or at least twice during a batch analysis. A laboratory duplicate (LD) will be analyzed after every twenty samples or will be analyzed at least twice during a batch analysis. The criteria for these checks are noted in Table 1.

Table 1. Quality control samples and acceptance criteria.

Check	Acceptance Criteria
Blank	\leq MDL
MD	\pm 20% of each element
Soil Check Stnd	\pm 20% of each element

15 Calibration and standardization

- 15.1 Errors are inherent in the measurement of X-rays due to the nature of radioactive disintegration of the ray source.
- 15.2 Randomness of the emission of X-rays from the sample is random but predictable.
- 15.3 Calibration was completed by the manufacturer, Oxford Instruments. The calibration certificate is kept with original operating and safety manuals in the OEWRI office.
- 15.4 The background plate will be used to minimize accuracy errors and reduce backscattering.
- 15.5 Any damage to the instrument will be reported to the QA/QC manager. The instrument will be maintained as defined in the user manual.
- 15.6 The detection limits for this instrument are: Antimony = 19ppm, Arsenic = 7ppm, Barium = 60ppm, Cadmium = 23ppm, Cobalt = 18ppm, Copper = 15ppm, Chromium = 40ppm, Gold = 16ppm, Lead = 15ppm, Mercury = 13ppm, Nickel = 16ppm, Selenium = 4ppm, and Zinc = 8ppm.

16 Procedure

Field

- 16.1 All three batteries for the XRF should be recharged prior to field deployment. Plug each battery into the charger located in Temple 125. Each battery could take up to 2 hours to fully recharge. Each fully charged battery will operate the instrument for approximately 4 hours.
- 16.2 The instrument will be reserved and signed out by the primary operator for field activities for that day. The reservation sheet is located on the cabinet where the instrument is stored in the OEWRI office. When reserving the instrument, the primary user will provide their name and the date of use. When the primary operator actually removes the instrument from the cabinet, they must list a secondary operator if applicable, field team members, and the date and time that the instrument was removed from the cabinet. The primary operator is responsible for the instrument for the entire time that it is out of the cabinet. See Section 8.23 for additional security information.
- 16.3 Seat and lock the PDA into the instrument:
 - a. Remove the stylus from the PDA computer.
 - b. On the right side of the XRF, unlock the PDA computer lock with the key. Slide the PDA computer into the cradle and take care to seat it on the connector correctly.

- c. Lock the PDA computer into place and remove key. Store key in the key container in the case.
- 16.4 Insert a fully charged battery into the instrument by pushing it into the handle of the instrument until it clicks into place. Place the other 2 fully charged batteries into the performed area in the case that is specifically for the batteries.
- 16.5 Place the instrument into the XRF case. Arrange all attachments, instrument safety and power keys, and supplemental materials in the case in a manner that ensures that they stay in place during transport. Close the case and lock it making sure that the key for the outside lock is outside of the XRF case. The instrument will be stored in a cool, dry location within the case during transport to and from the field. The instrument should never be left unattended and should be in direct sight of the primary operator for the field activities or in a secure area as designated by the primary operator. See Section 8.23 for additional security information.
- 16.6 Once on location in the field, unlock the case and secure the lock and key within the case. Insert the instrument power key and turn it to the "ON" position. The yellow indicator light will illuminate. Wait 1-2 minutes for the peltier cooler and X-ray tube to stabilize.
- 16.7 Push the PDA power switch "ON". It is located in the upper right corner just above the screen.
- 16.8 To adjust the backlight on the PDA use the stylus to tap the on the small circular slider below the "light bulb" icon located on the right side of the screen and move it back and forth. When plugged in, the PDA draws power from the battery in the XRF, so the brighter the backlight, the more power it pulls from the XRF battery.
- 16.9 Activate the software by tapping on the "Start" menu and then on "X-MET". The "X-MET3000" screen will appear and will contain a "Waiting for Connection" message. A screen with a white background will appear shortly thereafter. If the software is started before the instrument power is turned on the program cannot communicate with the instrument. If this occurs, refer to Section 24 for troubleshooting instructions.
- 16.10 Fundamental Parameters (FP), shown as "[soil_fp]" in the upper left corner of the screen, is the method used. This XRF is only set to use the FP method, do not change the method. If "[soil_fp]" is not on the screen, contact the Laboratory Director.
- 16.11 The active results format is illustrated to the right of "[soil_fp]". For OEWRI field work, this should read: [OWERI-field].
- 16.12 Tap anywhere within the white background to bring up the X-MET 3000 Main Menu. The only menus that can be accessed by users are: "Name Sample", "Set Measurement Time", "Result Format", and "Display Spectra" (that follows

measurements, if desired). Do not change any settings in “Select Method” or “Settings” menus.

- 16.13 Tap anywhere outside of the menu and within the white space to exit the menu.
- 16.14 Tap “Output Settings” to define the data storage file. All field data collected must be saved in the “FieldData” folder located in My Documents on the Storage Card. Do not save the data files anywhere else.
- 16.15 The “Results Format” menu chooses the output configuration for the measured data. Select “[OWERI-field]”. Result formats can vary and will only be modified by the project manager and the Laboratory Director prior to field deployment.
- 16.16 Naming the samples and additional information:
 - a. Tap “Name Sample” and input the sample name which should be a combination of location and site number using the keyboard displayed on the screen. If the name consists of a continuous phrase, a space then a number (example: “Site 1”), the number is automatically increased after every measurement (Site 2, Site 3, etc.). Samples cannot be named after it has been measured, so input sample name prior to sample measurement.
 - b. Tap “OK” to proceed to the next screen “Additional Information” and input any other descriptive information such as the project title.
 - c. The maximum number of characters for the sample name is 20. The maximum for the additional information is 80 characters.
- 16.17 The time span in which the sample is measured is based on project requirements and will be set by the project manager. Tap the button labeled “Set Measurement Time:” to change the measurement time span. The minimum measurement time for [OEWRIField] is set to 5 seconds. When screening in the field, a minimum of 20 seconds is suggested. This measurement time provides relative elemental concentration peaks, not exact values, which is adequate for screening samples in the field.
- 16.18 All samples will be analyzed by using the “Timed Assay” trigger operating mode which the instrument is set to use. The timed assay allows the operator to push the trigger once and release versus applying pressure to the trigger for the entire measurement time span.
- 16.19 Carefully place the standard in the safety shield. The safety shield is the small metal box stored in the upper left corner of the XRF case. The standard should be positioned with the plastic wrap side up taking care to not tear or damage the plastic wrap on the top of it. Input the sample name as “Standard”. Press the trigger on the instrument handle to analyze the standard for the set measurement time span. The red x-ray indicator light will turn on indicating that radiation is being emitted and the instrument is working properly. The standard has been analyzed and it is safe to remove the standard when the red x-ray indicator light is

not illuminated. The results of the measurement will be shown on the white background in table form. Return the standard to its case and place it back into the XRF case. The standard will be the first analysis for each field outing.

- 16.20 Remove the background plate from the XRF case. Place an empty sample bag on the background plate. Input the sample name as "Bag Blank". Press the trigger on the instrument handle to analyze the sample bag for the set measurement time span. The red x-ray indicator light will turn on indicating that radiation is being emitted and the instrument is working properly. The analysis is complete and it is safe to remove the standard when the red x-ray indicator light is not illuminated. The results of the measurement will be shown on the white background in table form. The empty sample bag, or "Bag Blank" will be the second analysis for each field outing.
- 16.21 Before analyzing samples during screening processes or from the bag, remove any leaves, grass, or stones from the measurement surface area.
- 16.22 When screening samples in situ, cover the measurement surface area with a sample bag to protect the measurement window.
- 16.23 When screening bagged samples, place the background plate underneath the sample to prevent interference from materials behind the sample and to reduce x-ray scattering. The background plate may be used when analyzing soil cores as dictated by field conditions and project management. Field notes should indicate whether a background plate was used or not.
- 16.24 Place the instrument measurement window directly over the sample and hold the instrument perpendicular to the sample surface during the entire length of the measurement. Ensure that the thickness of material being sampled is at least 1 cm for best results.
- 16.25 Press the trigger once to start the analysis and measure elements for the set measuring time. The red x-ray indicator light will turn on indicating that radiation is being emitted and the instrument is working properly.
- 16.26 The results of the measurement will be shown on the white background in table form.
- 16.27 Continue analyzing samples either in situ, from cores, or in sample bags, inputting appropriate sample names. In addition to the PDA, a field notebook should record sample analyses and their locations. Global positioning points should be recorded in the field notebook or incorporated into the sample name if various locations are being analyzed for the project.
- 16.28 The XRF can be left on between analyses or while moving to different sites. Turn the PDA off to save battery power. To begin analyzing again, press the power button in the upper right corner on the PDA.

- 16.29 When all analyses are complete, select “Exit” from the Main Menu to exit the program. The program will prompt for a confirmation in case you mistakenly tap the “Exit” button.
- 16.30 Push the PDA power switch “OFF”.
- 16.31 Turn the instrument power key to the “OFF” position and remove the key.
- 16.32 Return the XRF back to the case provided for transport. Make sure to verify that the PDA lock and instrument power keys, standard, and background plate are in the case. Close the case and lock it making sure that the key for the outside lock is outside of the XRF case.
- 16.33 Upon returning to the University, the primary operator will:
- a. Return the batteries to the bench top and plug one in to charge.
 - b. Remove the PDA from the instrument and place it in the PDA cradle for charging and data extraction.
 - c. Secure the instrument within the locked cabinet located in the OEWRI office.
 - d. Sign the instrument reservation sheet providing the time and date of return and verifying that that instrument was returned complete and clean.
- 16.34 Return the instrument case lock key and the instrument use record to the QA/QC manager.

Laboratory

- 16.35 The instrument will be reserved and signed out by the primary operator for laboratory activities for that day. The reservation sheet is located on the cabinet where the instrument is stored in the OEWRI office. When reserving the instrument, the primary user will provide their name and the date of use. When the primary operator actually removes the instrument from the cabinet, they must list a secondary operator if applicable, others in the laboratory during analyses, and the date and time that the instrument was removed from the cabinet. The primary operator is responsible for the instrument for the entire time that it is out of the cabinet. See Section 8.23 for additional security information.
- 16.36 Position the instrument in the bench top stand as shown below. Ensure that the grooves on the side of the XRF main body and handle are lined up with the stand. Slip the black rubber band attached to the peg below the handle over the end of the handle to ensure proper instrument stability.

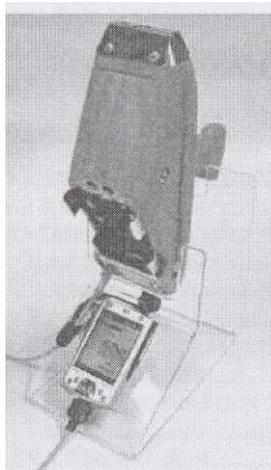


Figure 7. Instrument position in bench top stand.

- 16.37 Attach the auxiliary power cord to the instrument by removing the battery and plugging the cord into the bottom of the handle. The outlet is located to the right of the work station and the auxiliary power supply cord should not be removed from the bench at any time.
- 16.38 Connect the PDA to the instrument with the remote extension cable using the port on the front of the instrument. Ensure that the red mark on the round end of the cable is pointing downward when inserting it into the instrument. The opposite end of the cord plugs into the bottom of the PDA only one correct way. Do NOT force the cord into the PDA.
- 16.39 Place the sample bag holder (large metal box) on the instrument and tighten it to the instrument. It is a snug fit so be sure that it is fully set on the instrument before tightening the black knobs. **Never analyze any sample in the laboratory without using the sample bag holder.**
- 16.40 Insert the instrument power key and turn it to the “ON” position. The yellow indicator will illuminate. Wait 1-2 minutes for the peltier cooler and X-ray tube to stabilize.
- 16.41 Push the PDA power switch “ON”. It is located in the upper right corner just above the screen.
- 16.42 To adjust the backlight on the PDA use the stylus to tap the on the small circular slider below the “light bulb” icon located on the right side of the screen and move it back and forth.
- 16.43 Activate the software by tapping on the “Start” menu and then on “X-MET”. The “X-MET3000” screen will appear and will contain a “Waiting for Connection” message. A screen with a white background will appear shortly thereafter. If the software is started before the instrument power is turned on the program cannot communicate with the instrument. If this occurs, refer to Section 24 for troubleshooting instructions.

- 16.44 Fundamental Parameters (FP), shown as “[soil_fp]” in the upper left corner of the screen, is the method used. This XRF is only set to use the FP method, do not change the method. If “[soil_fp]” is not on the screen, contact the Laboratory Director.
- 16.45 The active results format is illustrated to the right of “[soil_fp]”. For OEWRI laboratory work, this should read: [OWERI-lab].
- 16.46 Tap anywhere within the white background to bring up the X-MET 3000 Main Menu. The only menus that can be accessed by users are: “Name Sample”, “Set Measurement Time”, “Result Format”, and “Display Spectra” (that follows measurements, if desired). Do not change any settings in “Select Method” or “Settings” menus.
- 16.47 Tap anywhere outside of the menu and within the white space to exit the menu.
- 16.48 Tap “Output Settings” to define the data storage file. All laboratory data collected must be saved in the “LabData” folder located in My Documents on the Storage Card. Do not save the data files anywhere else.
- 16.49 The “Results Format” menu chooses the output configuration for the measured data. Select “[OWERI-lab]”. Result formats can vary and will only be modified by the project manager and the Laboratory Director prior to field deployment.
- 16.50 Naming the samples and additional information:
- a. Tap “Name Sample” and input the sample name which should be a combination of location and site number using the keyboard displayed on the screen. If the name consists of a continuous phrase, a space then a number (example: “Site 1”), the number is automatically increased after every measurement (Site 2, Site 3, etc.). Samples cannot be named after it has been measured, so input sample name prior to sample measurement.
 - b. Tap “OK” to proceed to the next screen “Additional Information” and input any other descriptive information such as the project title.
 - c. The maximum number of characters for the sample name is 20. The maximum for the additional information is 80 characters.
- 16.51 The time span in which the sample is measured is based on project requirements and will be set by the project manager. Tap the button labeled “Set Measurement Time:” to change the measurement time span. The minimum measurement time for [OWERI-lab] is set to 90 seconds.
- 16.52 All samples will be analyzed by using the “Timed Assay” trigger operating mode which the instrument is set to use. The timed assay allows the operator to push the trigger once and release versus applying pressure to the trigger for the entire measurement time span.

- 16.53 Carefully place the standard in the sample bag holder over the sampling window. Place a small piece of paper over the proximity sensor that is located left of the measurement window. The standard should be positioned with the plastic wrap side down taking care to not tear or damage the plastic wrap on the top of it. Input the sample name as "Standard". Press the trigger on the instrument handle to analyze the standard for the set measurement time span. The red x-ray indicator light will turn on indicating that radiation is being emitted and the instrument is working properly. The standard has been analyzed and it is safe to remove the standard when the red x-ray indicator light is not illuminated. The results of the measurement will be shown on the white background in table form. Return the standard to its case and place it back into the XRF case. The standard will be the first analysis for each laboratory batch.
- 16.54 Place an empty sample bag into the sample bag holder. Input the sample name as "Bag Blank". Press the trigger on the instrument handle to analyze the sample bag for the set measurement time span. The red x-ray indicator light will turn on indicating that radiation is being emitted and the instrument is working properly. The analysis is complete and it is safe to remove the standard when the red x-ray indicator light is not illuminated. The results of the measurement will be shown on the white background in table form. The empty sample bag, or "Bag Blank", will be the second analysis for each laboratory batch.
- 16.55 Before analyzing samples, remove any leaves, grass, or stones from the measurement surface area.
- 16.56 Organize the project sample bags to minimize confusion when preparing samples and to ensure that sample information is entered into the database correctly.
- 16.57 Insert each sample bag into the sample bag holder directly over the measurement window. The thickness of material being sampled should be at least 1 cm, if it is not, note the approximate thickness in the laboratory notes or incorporate the information into the sample name.
- 16.58 Press the trigger on the instrument handle to analyze the sample bag for the set measurement time span. The red x-ray indicator light will turn on indicating that radiation is being emitted and the instrument is working properly. The analysis is complete and it is safe to remove the standard when the red x-ray indicator light is not illuminated. The results of the measurement will be shown on the white background in table form.
- 16.59 Continue analyzing each sample.
- 16.60 Include a laboratory duplicate for every 20 samples analyzed. Input the sample name as the original sample name adding "-LD" at the end of the sample name. Laboratory duplicates for this method are a re-analysis of a sample. Analyze the sample initially, remove the sample bag, shake it, and reanalyze it. There must be 2 LD's for each batch analyzed.

- 16.61 Reanalyze the standard after every 20 samples and at the end of the batch analysis. Input the sample name as "Check Standard". Reanalyze an empty sample bag after every 20 samples and at the end of the batch analysis. Input the sample name as "Bag Blank".
- 16.61 The XRF can be left on between analyses.
- 16.62 When all analyses are complete, select "Exit" from the Main Menu to exit the program. The program will prompt for a confirmation in case you mistakenly tap the "Exit" button.
- 16.63 Push the PDA power switch "OFF".
- 16.64 Turn the instrument power key to the "OFF" position and remove the key.
- 16.65 Return the XRF back to the case. Make sure to verify that the PDA lock and instrument power keys, standard, and background plate are in the case. Close the case and lock it making sure that the key for the outside lock is outside of the XRF case.
- 16.66 Remove the PDA from the instrument and place it in the PDA cradle for charging and data extraction.
- 16.67 Secure the instrument within the locked cabinet located in the OEWR office.
- 16.68 Sign the instrument reservation sheet providing the time and date of return and verifying that that instrument was returned complete and clean.
- 16.69 Return the instrument case lock key and instrument use record to the QA/QC manager.

17 Data acquisition, calculations, and reporting

- 17.1 To download data from the instrument to a computer:
 - a. Ensure that the Microsoft ActiveSync software is installed on the destination computer.
 - b. Insert the PDA into the PDA cradle and plug the USB cord into the computer.
 - c. Navigate to Tools>Explore Pocket PC on the toolbar of the ActiveSync window that opens. A new Windows Explorer window will open.
 - d. Navigate to: Mobile Device> My Windows Mobile-Based Device> Storage Card> My Documents. If the Storage Card is not visible, turn off the PDA, gently remove the storage card in the back and reinsert it carefully. Turn the PDA back on and try to navigate to the destination again.
 - e. Choose the data location: FieldData or LabData and then copy and paste the "*.log.txt" files to any folder on a different drive (local, network, etc.). All

OEWRI raw data files must be copied to the appropriate project removable drive. It is recommended that an additional copy the file be saved to another location for backup.

- f. Open the pasted files to ensure they were properly copied.
- g. Delete all files that were copied from the source folder (FieldData or LabData) on the PDA. All OEWRI raw data files must be copied to the appropriate project removable drive before deleting data files from the PDA.
- h. “.log.txt” files can be opened using Microsoft Excel. Save the “.log.txt” files as Workbooks using the “Save As” option in Microsoft Excel.

17.2 Files will be removed from the PDA regularly. All data should be downloaded immediately upon returning from the field.

17.3 The X-ray intensity which is measured from the size of the spectrum peaks is directly proportional to the concentration of the elements in the sample.

$$I = \frac{N}{t} = \frac{(k)(I_0)(T)(C)}{H}$$

Where:

- I = X-ray Intensity (counts per second)
- N = Net count (minus background and overlap)
- t = Measurement time (seconds)
- k = Geometrical constant (sensor-sample geometry)
- I₀ = Source strength (photons per second/steradian)
- T = Excitation cross section for the element in question
- C = Weight fraction of the element
- H = Matrix absorption coefficient.

17.4 Reporting results: Results should be reported to 0.1 mg TN/L precision.

17.5 Relative percent difference (RPD): $\left[\frac{(A-B)}{\text{AVERAGE}(A,B)} \right] 100$

- A = original sample concentration
- B = duplicate sample concentration

17.6 Standard Deviation: The evaluation of MDL and precision require calculation of standard deviation. Standard deviations should be calculated as in equation 2.

$$s = \left\{ \frac{\sum x^2 - [(\sum x)^2/n]}{n - 1} \right\}^{1/2}$$

Where: n = number of samples,
 x = concentration in each sample.

Note: This is the sample standard deviation calculated by the STDEV function in Microsoft Excel.

- 17.7 Coefficient of Variation (Cv%): The evaluation of accuracy require the calculation of coefficient of variation which is the standard deviation of the multiple sample measurements divided by the mean of those measurements.

$$Cv\% = (s/\bar{x})100$$

18 Computer hardware and software

- 18.1 X-Met software on PDA provided for the instrument by Oxford Instruments.
- 18.2 This document is created using Microsoft Word. The Word file name for this SOP is: XRF.doc
- 18.3 Microsoft Excel is used for recording and reviewing the final data from the instrument.

19 Method performance

- 19.1 The desired performance criteria for this measurement are:
- Detection limit: varies by element and batch, but the instrument detection limits are: Antimony = 19ppm, Arsenic = 7ppm, Barium = 60ppm, Cadmium = 23ppm, Cobalt = 18ppm, Copper = 15ppm, Chromium = 40ppm, Gold = 16ppm, Lead = 15ppm, Mercury = 13ppm, Nickel = 16ppm, Selenium = 4ppm, and Zinc = 8ppm.
 - Precision: $\pm 20\%$, precision data will be reported as relative percent difference and project managers will determine what data is used.
 - Accuracy: $\pm 20\%$, accuracy data will be reported as relative percent difference and project managers will determine what data is used.
 - Minimum Quantification Interval: 1.0 ppm
- 19.2 Zinc, iron, chromium, and calcium concentrations are routinely found in blanks during batch analysis due to materials used to manufacture the sample holder.

20 Pollution prevention

All wastes from these procedures shall be collected and disposed of according to existing waste policies within the MSU College of Natural and Applied Sciences.

21 Data assessment and acceptable criteria for quality control measures

- 21.1 The analyst should review all data for correctness.
- 21.2 Relative percent difference (RPD) should be calculated for pairs of duplicate analyses to determine precision. The desired precision is $\pm 20\%$. Laboratory duplicate (LD) measurements will be randomly selected from each set of 20 samples in the batch and analyzed. There must be at least two LD analyses

during each batch analysis. Precision data will be reported as relative percent difference and project managers will determine what data is used.

- 21.3 The soil check standard will be used to determine accuracy associated with each batch. The soil check standard is measured after each set of 20 samples or at least twice during each batch analysis. The RPD between the average of the measurements of the check standard for each batch and the initial soil check standard report will be determined for each element and recorded with each laboratory report. The soil check standard report is included in Section 26. The desired accuracy is $\pm 20\%$. Accuracy data will be reported as relative percent difference and project managers will determine what data is used.
- 21.4 The completed Excel spreadsheet is reviewed by the analyst's supervisor or the OEWRI QA officer.

22 Corrective actions for out-of-control or unacceptable data

- 22.1 The results for precision and accuracy are compared to the acceptable value of $\pm 20\%$ for this analysis for all analytes. The initial soil check standard (2A1) report contains the concentrations of each element within the soil check standard and is included in Section 26.
- 22.2 Zinc, iron, chromium, and calcium concentrations are routinely found in blanks during batch analysis due to materials used to manufacture the sample holder.
- 22.3 If data are unacceptable for any reason, the analyst should review their analytical technique prior to conducting this analysis again.
- 22.4 When measurement time is increased the signal, N, increases. The time of analysis can be increased to increase precision and accuracy. The count rate should be increased by a factor of four to reduce the standard deviation by one-half.
- 22.5 If the sensor is not against the sample at a direct and consistent angle, the geometrical constant (k) will vary. Maintain direct and consistent angle of measurement.
- 22.6 The excitation cross section, T, varies with atomic number of the element and with source energy. The closer the element's absorption energy is to the source energy, the higher the excitation cross section. Certain elemental energies may further away from the source energy and will result in lower excitation efficiency.
- 22.7 The instrument may require trouble shooting techniques if the data are unacceptable. All instrument maintenance will be conducted by the QA/QC manager or sent to the manufacturer.

23 Waste management

There are no wastes generated using this method. All samples remain in sample bags and no other consumables are used.

24 Troubleshooting

- 24.1 If the software program is started before the instrument power is turned on, the program cannot communicate with the instrument. If this occurs, follow the steps below to reset the PDA.
- Exit the software program and turn off power to the instrument.
 - Remove the PDA from the instrument and use the stylus to press the reset button on the bottom left corner of the PDA. The loading screen will appear, turn the PDA off.
 - Remount the PDA onto the instrument.
 - Switch the instrument power "ON", turn the PDA on, and start the software program.
- 24.2 If the instrument will not perform an analysis of a sample, first, make sure that the instrument was powered on before the software program was started. If that does not initiate analyses, follow the steps below.
- Make sure that the infrared sensor is covered completely with sample. The safety sensor must be activated before analyses can begin.
 - Make sure that the yellow power light is on. If the yellow light is dim or is blinking, change the battery or connect the instrument to the AC adaptor.
 - If the instrument was powered before the software program and the PDA has been reset and the PDA is still not communicating with the instrument, make sure that the PDA is seated properly on the instrument. Exit the software, remove the PDA, reseal the PDA, and restart the software program.
 - If the sample surface is too darkly colored to reflect light, the infrared sensor may not activate. If the sample is clearly dark and the instrument will not perform an analysis, insert a piece of white paper between the sample and the infrared sensor.
- 24.3 If the X-MET software locks up, PDA memory may be the cause. Close all other programs by tapping on "Start", then "Settings", then "System" tab, then "Memory", then "Running Programs", then "Stop All".
- 24.4 If the PDA touchscreen completely "freezes" and the reset button did not fix the problem, perform a hard reset by following the steps below.
- Take the memory card out of the PDA.
 - Press and hold down the left and right function buttons at the bottom of the front panel and simultaneously press the reset button at the bottom of the PDA until the display turns dark.
 - Use the stylus to press the reset button on the bottom left corner of the PDA.
 - Follow screen instructions to realign the PDA screen and set the time and date settings.
 - If this does not fix the problem, recharge the PDA and perform a hard reset. If recharging the PDA does fix the problem, contact the QA/QC manager.

25 References

- 25.1 19 CSR 20, "Radiation Protection", Missouri Department of Health and Senior Services.
- 25.2 29 CFR Part 1910, Occupational Safety and Health Administration.
- 25.3 N 43.2-2001, "Radiation Safety for X-ray Diffraction and Fluorescence Analysis Equipment", American National Standard Institute (ANSI)/Health Physics Society (HPS).
- 25.4 Missouri Statutes Section 192.400, "Radiation Control", Department of Health and Senior Services.
- 25.5 US EPA Method 6200, "ITRV: XRF Technologies for Measuring Soil and Sediment (Niton XLt 700 Series XRF)", EPA publication EPA/540/R-06/004, February 2006.
- 25.6 User's Manual. 2006. Eureka Environmental Engineering, Austin Texas.
- 25.7 Safety Manual. 2004. Eureka Environmental Engineering, Austin Texas.

26 Appendices

X-MET 3000TXS+ Check Sample Report													
Serial Number: K50080													
Date: September 7, 2007													
Software: 2.2.1													
Method: Soil FP													
Type: Soil FP													
Time: 60 seconds													
Units: PPM													
Alloy: Soil Check Sample													
NAME	Fe	Ca	Ti	Zn	Zr	Sr	Mo	Pb	Cu	Cd	Hg	As	Se
Run 1	10090	9943	659	590	291	250	159	98	73	70	57	52	45
Run 2	10212	9537	650	586	279	256	155	0.00	67.00	51.00	50.00	56.00	42.00
Run 3	10112.00	10312.00	668	592	279	233	162	0.00	81.00	86.00	62.00	56.00	43.00
Average	10138.00	9930.67	659.00	589.33	283.00	246.33	158.67	32.67	73.67	69.00	56.33	54.67	43.33
STDEV	65.02	387.65	9.00	3.06	6.93	11.93	3.51	56.58	7.02	17.52	6.03	2.31	1.53

Figure 8. Soil Check Standard (2A1) Report.



Missouri State
UNIVERSITY

XRF SOP ACKNOWLEDGMENT FORM

Name _____ SS # _____

Address _____
Street/Box City State Zip

Phone (local): _____(permanent) _____

Name of Guardian:

Guardian's Address _____ Phone: _____

ACTIVITY INFORMATION

Type of Activity: Operation of the X-MET3000TXS+ handheld elemental analyzer for heavy metal screening and measurement of contaminated soil.

ACKNOWLEDGMENT

"I, the undersigned participant, being the age of 18 or above, desire to participate in the Activity described above, which includes transportation to and from Springfield. The Activity is potentially hazardous and I have read and understand the standard operating procedures (SOP) associated with this instrument. I should not engage in the Activity unless I am alert and observant, which I represent myself to be. I assume any and all risks associated with the Activity including but not limited to, fall, personal injury, collision with other persons, the effects of weather, including high heat and humidity and motor vehicle transportation, all such risks being known and appreciated by me. I attest that I am sufficiently physically fit to participate in this Activity.

Knowing the risks, and in consideration of being permitted to participate in the Activity described above, I for myself and my personal representatives, heirs, and assigns, do hereby hold harmless and release, waive discharge and covenant not to sue the board of Governors of Missouri State University from any and all claims or liability on account of death or injury to person or property of the undersigned of any kind of nature whatsoever arising out of or in any way connected with, the undersigned's participation in the Activity and transportation activities, even though the claim or liability may arise out of the negligence or carelessness on the part of Missouri State University, or any third person, whether foreseen or unforeseen, known or unknown.

The undersigned hereby expressly agrees that this release and waiver is intended to be as broad and inclusive as permitted by the laws of the State of Missouri and that if any portion hereof is held invalid, it is agreed that the balance, notwithstanding, continues in full legal force and effect.

The undersigned further states that he/she has carefully read the foregoing Release and Waiver of Liability, knows the contents thereof, and has agreed to sign this Release and Waiver of Liability as his/her own free act and deed.

I also declare that I will take all necessary and/or recommended precautions to insure my own person against physical and/or mental injury and property loss or damage. This includes, but is not limited to, following printed or verbal instruction given by the activity leader(s).

I further declare that I assume responsibility for my actions or behaviors that may conflict with accepted standards. University requirements for participants, common sense or the instructions, I receive from Activity leader(s) either before or during the activity.

I do hereby affirm that I am covered under my guardian's medical policy or otherwise have adequate medical insurance.

I have read the above release and agree to the provisions contained herein. I have also informed my Guardian of this trip.

For the participant:

If 18 years of age:

Participant's Signature Date

Guardian's Signature Date

This form should be returned to the QA/QC manager in Temple Hall Room 326.

