



# National Human Exposure Assessment Survey (NHEXAS)

## Arizona Study

# Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona Tucson, Arizona 85721

Cooperative Agreement CR 821560

## **Standard Operating Procedure**

**SOP-UA-L-10.1** 

**Title:** Operation, Calibration, and Routine Use of the Spectrace 9000

Field Portable X-Ray Fluorescence Analyzer

**Source:** The University of Arizona

U.S. Environmental Protection Agency Office of Research and Development Human Exposure & Atmospheric Sciences Division Human Exposure Research Branch

Notice: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), partially funded and collaborated in the research described here. This protocol is part of the Quality Systems Implementation Plan (QSIP) that was reviewed by the EPA and approved for use in this demonstration/scoping study. Mention of trade names or commercial products does not constitute endorsement or recommendation by EPA for use.

### Operation, Calibration, and Routine Use of the Spectrace 9000 Field Portable X-Ray Fluorescence Analyzer

#### 1.0 Purpose and Applicability

The purpose of this SOP is to describe the procedures to be followed in operating and calibrating the Spectrace 9000 field portable X-ray fluorescence analyzer. This procedure applies to the determination of metal concentrations in samples for the EPA NHEXAS project of the University of Arizona/Battelle/Illinois Institute of Technology consortia. Note; references to actions on the unit's keyboard are noted in **Bold** surrounded by quotation marks (ex. "cont/pause").

#### 2.0 Definitions

- 2.1 keV = Kilo electron volt
- 2.2 SOP = Standard operating procedure
- 2.3 Spectrace 9000 = Portable XRF analyzer
- 2.4 XRF = X-Ray florescence

#### 3.0 References

- 3.1 Spectrace 9000 Portable XRF Analyzer Operating Instructions, revision 0.3, January 1992.
- 3.2 M. Bernick, P. Berry, G. Voots, G. Prince, et al., A High resolution Portable XRF HgI<sub>2</sub> Spectrometer for Field Screening of Hazardous Metal Wasters, Pacific-International Congress on X-Ray Analytical Methods, August 1991.
- 3.3 Spectrace 9000 Field Portable X-Ray fluorescence Operating Procedure, Standard Operating Procedures, Response Engineering and Analytical Contract, U.S. EPA Environmental Response Team, SOP No.: 1713, 33 pp., Revision 1.0, December 21, 1992.

#### 4.0 Discussion

The Spectrace 9000 portable XRF analyzer employs three radioactive isotope sources (Fe-55, Cd-109 and Am-241) for the production of primary X-rays. Each source emits a specific set of primary X-rays which excite a corresponding range of elements in a sample. The sample is positioned in front of the source-detector window and measurement is initiated. Fluorescent and backscattered X-rays from the sample enter through the beryllium (Be) detector window and are counted in the high resolution mecuric iodide (HgI<sub>2</sub>) detector.

Elemental concentrations are computed using a fundamental parameter algorithm of the form:

Concentration =  $R \times S \times (1 + SUM (A_n \times C_n))$ 

where

R = the measured analyte S-ray intensity relative to the pure element.

S = a calculated sensitivity coefficient.

SUM{} = a summation of n-element absorption-enhancement terms containing calculated alpha-coefficients and iteratively computed element concentrations.

The Spectrace 9000 uses fundamental parameter XRF calibrations derived from theoretical considerations. Each application contains a complete analysis configuration which includes elements to be measured, interfering elements in a sample, and a set of fundamental parameter calibration coefficients. A comparison between the Spectrace 9000 and a "Kevex" XRF from the AREAL Laboratory is contained in Appendix A.

Samples collected within the homes of participants in the NHEXAS project will be tested for their elemental content. The elements of interest include Primary Metals; Lead (Pb), Arsenic (As), Cadmium (Cd), Nickel (Ni), and Chromium (Cr); and Secondary Metals; Barium (Ba), Manganese (Mn), Selenium (Se), Vanadium (V), Copper (Cu), and Zinc (Zn). The sample types include soil from the yard and next to the foundation, dust from carpeting, dust from windowsills, and air filters. XRF analysis will constitute the primary analysis in the initial stages of sampling and will be coupled with other more precise analytical methods during a subset of samples.

#### 5.0 Responsibilities

- 5.1 The Project Director will be responsible for:
  - 5.1.1 Final review and approval of this procedure.
- 5.2 The Project Lab Supervisor will be responsible for:
  - 5.2.1 Insuring SOP procedures are followed by the Project Lab Staff.
  - 5.2.2 Notifying the appropriate technicians with needed repairs. In cases when the item can not be fixed in-house, Project Field Coordinator will generate the appropriate paperwork, notify the appropriate vendor or company, and ship the disfunctional item.
- 5.3 The Project Lab Staff will be responsible for:
  - 5.3.1 Knowing and following the procedures described in this SOP.
  - 5.3.2 Recording the information as directed in this SOP.
  - 5.3.3 Notifying the Project Lab Supervisor with down equipment and repair supplies needed (where applicable).
  - 5.3.4 Providing the Project Lab Supervisor with down equipment label and isolating the down equipment into the down equipment area.

Page: 3 of 14

- 5.3.5 Insuring proper labeling techniques of down equipment.
- 5.3.6 Repairing the item (where applicable) in a timely manner.

#### 6.0 Equipment and Materials

- 6.1 Distilled Deionized Water
- 6.2 Iron Standard (#9800014)
- 6.3 NIST Standard Montana Soil #2710 (#9800005)
- 6.4 NIST Standard Vacuum Dust SRM #2583 (#9800006)
- 6.5 Quartz Blank (#9800002)
- 6.6 Spectrace 9000 XRF
- 6.7 Teflon Blank (#9800001)
- 6.8 Teflon Filter, 37 mm, Blank (#9800004)
- 6.9 Teflon Blank from a 6x6 Filter Sheet (#9800003)
- 6.10 XRay Mylar Film
- 6.11 XRay Fluorescence Cups

#### 7.0 Procedure

- 7.1 Preparation
  - 7.1.1 Siting Criteria
    - A Field Use:

The Spectrace 9000 is capable of being portable using a battery pack which installs into the back of the unit. The unit currently draws power using a converter plugged into the wall and routed through a "UPS" (uninterrupted power supply). Field use is currently not included in the study design of the NHEXAS project.

B. Laboratory Use:

The Spectrace 9000 will be located in the Laboratory (Room 130A), Health and Related Professions Bldg., University of Arizona. The Unit will be set up in a semi-permanent configuration for sample analysis. The unit meets all requirements of the University of Arizona siting criteria for radioactive material.

7.1.2 Reagents

Not applicable

- 7.1.3 Setup and Calibration
  - A. Work Area
    - 1. Wipe down the work area with DDW and paper towels.
    - 2. Locate the samples to be analyzed, and check that "XRF Analysis" forms (UA-10-1) are filled out for each sample, including the technician code, date, Sample ID#, source times and the application used.
    - 3 If the Spectrace 9000 is off turn it on and let it warm up for at least 30 minutes. Often the machine is left on continuously and the LED face is turned

down to prevent damage to the screen.

#### B. Calibration of Spectrace 9000

- 1. Energy Calibration Check
  - a. This procedure is performed at the beginning of each week. The calibration updates the energy files in the Spectrace 9000 to keep abreast of the decaying isotopes energy levels.
  - b. Place the safety shield, a thick cover lined with lead, on the probe and initiate a 600 second analysis to update the X-ray energy calibration.
  - c. The results and spectra are recorded in the "Spectrace 9000" logbook.
  - d. Locate and record, from the x12 horizontal magnification spectrum screen, the central keV value for each of the following peaks:

Theoretic Source Pe	al Specification ak (keV)		(keV)	
Cd-109	Pb L-alpha	10.54	<u>+</u> 0.040	
	Pb L-beta	12.61	±0.040	
	Pb L-gamma	14.76	<u>+</u> 0.040	
	Emission Peak	22.10	<u>+</u> 0.040	
Fe-55	S K-alpha	2.31	<u>+</u> 0.010	
	Emission Peak	5.89	<u>+</u> 0.010	
Am-241	Pb L-alpha	10.54	<u>+</u> 0.050	
	Pb L-alpha	12.61	<u>+</u> 0.050	
	Emission Peak	59.5	<u>+</u> 0.20	

- e. If any of the peaks fail to meet specification, i.e. fall outside of the specified level, do another energy calibration check.
- f. Failure of the second energy calibration check results in the machine being labled as "Down Equipment" and the company is consulted to resolve the problem.

#### 2. Resolution Check

- a. The resolution check examines the detector's ability to resolve X-ray energies and is to be performed at the beginning of each week.
- b. Select the application.
- c. Place the Iron standard (#9800014) on the unit and select Cd-109 for an acquisition time of 200 seconds.
- d. Save the results and spectra by entering the Iron Standard ID number

(#9800014) on the keypad and push "Cont/Pause".

- e. Display the Cd-109 spectrum. Move the cursor to the center of the large peak on the left hand side of the display using the "<=" key. The "Coarse"/"Fine" toggle switch allows movement to the center. Use the "Hmag" key then "12x" to display a larger image of the iron peak.
- f. Locate and record in the spectrac logbook the peak counts (must be >1000 counts) for the center of the iron K-alpha peak (6.4 keV). Divide the maximum peak counts by two and record the results.
- g. Use the "=>" key to move the cursor to the right side of the peak. Record the counts and keV of the channel with counts less than or equal to one-half the maximum peak count value recorded in step f.
- h. Repeat for the left side of the peak using the "<=" key.
- i. Subtract the left-side from the right-side keV. The difference should be less than 0.300 keV. If the unit fails to meet this specification, call Spectrace Instruments for assistance.
- i. Return to the main menu.

#### 3. Check of the isotope intensities

- a. The check of the isotope intensities uses the values obtained during the resolution check (7.1.3; B; 2).
- b. Select from the main menu 5 "See Raw Data" then 6 "Intensities".
- c. Record the values and standard deviations for Mg, Fe, and Co.
- d. To be valid the intensities must be >0.95 for Fe, Mg <0.003, and Co <0.003.
- e. Return to the main menu.

#### 4. Blank Sample Check

- a. The blank (zero) sample check is performed to monitor the instrument's zero drift in the selected application. The check is performed at the beginning of each analysis session, and after not more than 10 analysis of a given sample type.
- b. Select the application to be used.
- c. Wipe the probe window and teflon blank with distilled deionized water and/or lens cleaner with a kimwipe.
- d. Measure the plug, using an acquisition time of 200 seconds for each source.
- e. Save the results and spectra by entering the sample ID# of the teflon plug (#9800001).
- f. The elemental results should fall within 3 standard deviations of zero to be valid.
- g. If elements are significantly outside these limits, check the probe window

SOP# UA-L-10.1 Revision #1 15 May 1999

Page: 6 of 14

and the blank sample for contamination and re-analyze. If the standard is not met in the second analysis perform "Acquire Background Data".

- h. Failure to achieve the standard set in 7.1.3.B.3.f is remedied by performing the "Acquire Background Data" operation. From the "Main Menu" press 1 "Measure" then 5 "Options", and 2 "Acquire Background Data". The machine will prompt for the alternate placement of "nothing", the "teflon plug", or "quartz plug" onto the window for analysis using each isotopic source.
- i. When finished re-run the teflon plug to assure that the analysis criteria are met (7.1.3.B.3.f).

#### 7.2 Analyses/Procedures

#### 7.2.1 Standards and Blanks

#### A. Machine Standards

1. The manufacturer provides a set of blank standards, iron (#9800014) quartz (#9800002) and teflon (#9800001), to be used in the calibration process.

#### B. Sample Standards

- 1. A soil sample, of the 2700 series provided by NIST, is used as the Calibration Standard (#9800005) for Soils analysis (figure 1), whereas a new vacuum dust standard is being developed by NIST, SRM #2583, and will be tested (#9800006).
  - a. The soils calibration standard consists of 2000.7 mg of material placed in an XRF sample cup and reserved as sample #9800005 during the life of the project. The standardized soil sample consist of moderately contaminated soil that was oven-dried, sieved, and blended to achieve a high degree of homogeneity. NIST standards are valid for 5 years from the date of shipment. A sample standard is run in conjunction with the analysis of any sample type to provide cross sample comparisons.
  - b. The vacuum dust calibration standard consists of 3.8101 g of material placed in an XRF sample cup and reserved as sample #9800006 during the life of the project. The standardized vacuum dust sample consist of moderately contaminated material that was oven-dried, sieved, and blended to achieve a high degree of homogeneity. NIST standards are valid for 5 years from the date of shipment. A sample standard is run before analysis of any sample type to provide cross sample comparison.
- 2. There are currently no commercially available standards for filter material, thus blank, unexposed filter material is used to determine a zero reading. These include a blank 37 mm filter (#9800003) used during Teflo filter analysis (25 and 37 mm) and the Teflon filter coating from a 6 x 6 inch filter sheet

(#9800004) used during Sentinel Filter analysis. A sample standard is run before analysis of any sample type is begun to ensure valid data.

#### 7.2.2 Actions

#### A. Soil Samples

- 1. From the main menu press 2 "Chose an application". Scroll to the "Soil Analysis" program and press "OK". The machine will prompt for confirmation. Press "Yes" to continue. The "Soil Analysis" program is used to analyze soil samples and carpet dust samples.
- 2. Clean the probe window by wiping with a kimwipe wetted with DDW and/or lens cleaner. Samples are placed in XRF cups and have mylar film stretched over the top during characterization (UA-L-11.1, 7.6 for soil; UA-L-12.1, 7.3.2.G for vacuum dust).
- 3. Place the sample with the mylar film window facing the x-ray source on the measurement gun. Close the cup shield over the sample.
- 4. Check the display to insure that each isotope is set for 200 seconds of exposure time. Check that readings are the result of "Real" seconds rather than "Live" seconds.
- 5. Press "Cont/Pause" to initiate the program.
- 6. The machine beeps when analysis is completed. Enter the sample ID on the keypad by pressing #1 "number" and the desired number for each digit of the sample ID. Press "Cont/Pause" upon completion.
- 7. Readings appear on the screen with their standard deviation, reported as "ppm". Record these measurements on the XRF analysis form as a backup to the electronic data (figure 3).
- 8. Repeat steps 3 through 7 for each sample.
- 9. Run the teflon plug (#9800001) as a standard after no more than 10 analysis to check for machine drift. The results must be within 3 standard deviations of zero values for the analysis to be valid. If values are outside of three standard deviations re-analyze the teflon plug. A second failure constitutes machine failure and calibration must be performed, using the "Acquire Background Data" program, and the samples re-analyzed.
- 10 The soils program analyzes for 25 elements which excludes Hg and Ir. A second program "Hg and Ir in Soils" analyzes for Hg and Ir by recalling each sample spectrum and calculating the elemental values (see 7.2.2.C.1-7).
- 11 Download data (7.2.2.2.D.1-4) after each batch of no more than 10 samples or before changing sample types.

#### B. Thin films

1. From the main menu press 2 "Chose an application". Scroll to the "Thin

Page: 8 of 14

films" program and press "**OK**". The machine will prompt for confirmation. Press "**Yes**" to continue. The "Thin Films" program is used to analyze all filters and sticky films.

- 2. Clean the probe window by wiping with a kimwipe wetted with DDW. Place the sample with the exposed surface facing the x-ray source on the measurement gun. Close the cup shield over the sample.
- 3. Check the display to insure that each isotope is set for 200 seconds of exposure time. Check that readings are the result of "Real" seconds rather than "Live" seconds.
- 4. Press "Cont/Pause" to initiate the program.
- 5. The machine beeps when analysis is completed. Enter the sample ID on the keypad by pressing #1 "number" and the desired number for each digit of the sample ID. Press "Cont/Pause" upon completion.
- 6. Readings appear on the screen with their standard deviation, reported as  $\mu g/cm^2$ . Record these measurements on the XRF analysis form as a backup to the electronic data (figure 3).
- 7. Repeat steps 3 through 7 for each sample.
- 8. Run the 37 mm teflon filter (#9800003) or sheet filter (#9800004) as a standard after no more than 10 analysis to check for machine drift. The results must be within 3 standard deviations of zero for the analysis to be valid. If values are outside of three standard deviations re-analyze the teflon plug. A second failure constitutes machine failure and calibration must be performed, using the "Acquire Background Data" program, and the samples re-analyzed.
- 9. The "Thin Films" program analyzes for 25 elements which excludes Hg and Ir. A second program "Hg and Ir in Thin Films" analyzes for Hg and Ir by recalling each sample spectrum and calculating the elemental values (see 7.2.2.C.1-7).
- 10 Download data (7.2.2.2.D.1-4) after each batch of no more than 10 samples or before changing sample types.

## C Calculation of Hg and Ir from analysis spectra

- 1. Each of the analysis programs calculate values for 25 elements. Due to programming constraints this leaves Hg and Ir out of the initial analysis. Separate programs for both soils (Hg and Ir in Soils) and thin films (Hg and Ir on Filters) exist to re-analyze spectra to obtain these values.
- 2. To recalculate values start from the main menu and press 2 "Choose an application". Scroll to the program to be chosen, "Hg and Ir in Soil" for soils and "Hg and Ir on Filters" for thin films, and press "OK". The machine prompts confirmation.
- 3. Disable the automatic spectra save option, so previously saved spectra are not saved again filling the machines memory. Return to the main menu and press 5

Page: 9 of 14

- "More", then 3 "Set store/send modes". Select 4 "Store", scroll to "Spectra" and choose 3 "Mod". Spectra should change from "On" to "Off". Exit and return to the main menu.
- 4. To recalculate Hg and Ir, Start from the main menu and press 1 "Measure". When the "Measure Screen" appears press 5 "Opts", and 5 again "Recall Spectra and recalc". Scroll to the desired ID number and press 3 "OK".
- 5. The machine will prompt for a new ID number. Enter the original sample ID.
- 6. Record the values on the sample's XRF back-up form (figure 3).
- 7. Return to the measure menu and repeat steps 2 through 6 for each spectra.

#### D. Downloading Data

- 1. Data is stored in the Spectrace 9000 for each set of samples analyzed. Once analysis is complete data is downloaded into a subdirectory and file on the NHEXAS network. Quality checks are made by the lab supervisor and the data is then copied to a subdirectory for processing by the data coordinator.
- 2. From the main menu of the Spectrace 9000 choose #3 "Review stored results", then #5 "All". A list of the ID numbers and the time of analysis should appear on the screen.
- 3. Go to the computer in room 130A and from the "C:" prompt type "R" and then "cd\spectrac" to get to the "spectrac" subdirectory on the "R" drive. Type "get G:\holding\:filename" and press "enter". The files are named according to the following format "mm/dd/yffc.d", where; "mm" is the month, "dd" is the day, "y" is the last digit of the year (i.e. 6 for 1996), "ff" is the sample prefix, "c" is an alphanumeric counter (i.e. A,B,C,...) for multiple files created on the same day, and "d" consist of the suffix which is either "dat" for data or "spc" for spectra. For example 6 floor dust samples analyzed on September 28, 1995, would be listed by sample number in the file named 0928571a.dat for the data and 0928571a.spc for the spectra.
- 4. Once downloaded, the "XRF Backup forms" (figure 3) are given to the lab supervisor for QC/QA check. The file is opened and the recorded values checked against the electronic data. The values reported on the Spectrac 9000 screen are rounded, thus the exact values will not match exactly on both the electronic and paper backup forms. There should be agreement between the two concidering the preceding contraint. Checked files are moved to "G:\XRF\_dat" for further processing by the data coordinator.
- 5. The "G:\holding" and "G\XRF\_dat" subdirectory is access limited to members of the lab staff, the lab and field directors, and the data coordinator.

#### 7.3 Calculations

7.3.1 Resolution Check Calculation

SOP# UA-L-10.1 Revision #1 15 May 1999 Page: 10 of 14

A. Run the Cd-109 isotope on the Iron Standard for 200 seconds (see 7.1.3.B.2 for procedure)

```
Record "KeV" and "Counts/2 "=

"Left side (< Counts/2)" = "KeV"

"Right side (< Counts/2) = "KeV"

"Left side KeV"-"Right side KeV" = value < .3 to be valid
```

#### 7.4 Quality Control

- 7.4.1 Tolerance Limits
  - A. Spectrace 9000
    - 1. Before use the Spectrace 9000 must satisfactorily pass the Resolution Check (7.1.3 B.2), and Blank Sample Check (7.1.3 B.3).
    - 2. The Spectrace 9000 will only be used if the results of blank samples fall within 3 standard deviations of reported sample values (figure 1).

#### 7.4.2 Detection Limits

- A. Spectrace 9000
  - 1. The minimum detection limits for the three sources at various times are listed in figure 2. Observed values may vary depending upon the age of the isotope sources. Variation with the amount of time of isotope exposure is shown in figure 3. Generally the detection limit is reduced by 50% for every fourfold increment in source measuring time.

### 7.4.3 Maintanence and Corrective Actions

#### A Spectrace 9000

- 1. If the Spectrace 9000 fails calibration or shows excessive zero drift, the manufacturer is to be consulted.
- 2. The x-ray sources last approximately 18 months. Once low energy levels are reached the machine will prompt for an "Energy Calibration Check" and fail the criteria outlined in 7.1.3.B. The manufacturer services the instrument during isotope replacement. Any service performed upon the machine is recorded in the "Spectrace 9000" logbook.
- 3. During operation a red light flashes on the Probe gun. If the light changes to yellow, this signals a low battery condition. Follow instructions in the "Operating Manual" to replace the battery.
- 4. The probe window may be broken during regular operation. Replacement instructions are also in the "Operation Manual".
- 5. If the battery pack is used, it is to be changed every 3 months. Outside power is

SOP# UA-L-10.1 Revision #1 15 May, 1999 Page: 11 of 14

turned off and the machine is allowed to drain the battery to the point that it turns itself off. The battery is exchanged with a secondary battery, and the replacement noted in the "Spectrace 9000" logbook. The original battery is reenergized to await the next battery change.

#### 8.0 Records

- 8.1 Data Collected by this Procedure
  - 8.1.1 Spectrace 9000
    - A. XRF analysis
      - 1. The results and spectra are downloaded from the machine onto the lab computer after each sample type is completed or upon the completion of not more than 10 analysis. See section 7.2.2.D for the procedure.
      - 2. The results of the XRF analysis are listed on the "XRF Analysis" form as a backup to electronic data (figure 3, L-10.0-1.0), along with the type of application used, sample ID#, technician code, date, time, and length of time for each isotope exposed.
    - B. Logbook
      - 1. All calibration information and maintanence performed on the machine is recorded in the "XRF Calibration Logbook".
- 8.2 Location/Placement of Forms
  - 8.2.1 Sample Forms
    - A. The completed "XRF Analysis" forms (figure 3) are filed by date in the Lab Supervisor's office.
    - B. A sign-out sheet is kept on the file cabinet.
    - C. Any individual removing the files records their name and the date on the sign out sheet.

Figure 1. Example of Certificate of Analysis for Standard Reference Soils.



## National Institute of Standards & Technology

## Certificate of Analysis

#### Standard Reference Material 2710

#### Montana Soil

#### Highly Elevated Trace Element Concentrations

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

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

#### NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification is valid for 5 years from the date of shipment from NIST. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST. Return of the attached registration card will facilitate notification.

Stability: This material is considered to be stable; however, its stability has not bean rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser.

<u>Use:</u> A minimum sample weight of 250 mg (dry weight - see Instructions for Drying) should be used for analytical determinations to be related to the certified values on this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to effect complete dissolution. If volatile elements (i.e., Hg. As, Sc) are to be determined, procesutions should be taken in the dissolution of SRM 2710 to avoid volatilization losses.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and R.L. Watters, Ir., of the NIST liberganic Analysical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Materials were coordinated through the Standard Reference Materials Program by T.E. Gills and J.S. Kane.

Gaithersburg MD 20899 October 30, 1992 William P. Reed, Chief Standard Reference Materials Program

(over)

Figure 2. Minimum detection limits for a number of different acquisition times and different sample types.

Soil Sample		nple	Thin Sample
N	1easuring	MDL	Measuring MDL
	ime (Sec)	(mg/kg)	Time (Sec) $(\mu g/cm^2)$
Fe-55			
Potassium (K)	60	325	200 0.40
Calcium (Ca)	60	150	200 0.20
Titanium (Ti)	60	110	200 0.15
Chromium (CrLo	60	180	200 0.40
Cd-109			
Chromium (CrHi		525	200 0.90
Manganese (Mn)	60	410	200 0.65
Iron (Fe)	60	225	200 0.65
Cobalt (Co)	60	205	200 0.50
Nickel (Ni)	60	125	200 0.30
Copper (Cu)	60	90	200 0.65
Zinc (Zn)	60	70	200 0.40
Mercury (Hg)	60	60	200 0.45
Arsenic (As)	60	50	200 0.40
Selenium (Se)	60	35	200 0.15
Lead (Pb)	60	30	200 0.50
Rubidium (Rb)	60	10	200 0.10
Strontium (Sr)	60	10	200 0.10
Zirconium (Zr)	60	10	200 0.15
Molybdenum (M	(o) 60	10	200 0.10
Am-241	•		
Cadmium (Cd)	60	180	800 2.50
Tin (Sn)	60	100	800 2.50
Antimony (Sb)	60	65	800 1.50
Barium (Ba)	60	20	800 0.70

SOP# UA-L-10.1 Revision #1 15 May 1999 Page: 14 of 14

Figure 3. XRF analysis Form.	XRF ANALYSIS
Technician Init Code [ ][ ]	
Sample ID# Source Time [ ][ ][ ][ ][ ][ ]	e: Fe-55 [ ][ ][ ]sec. Application: Thin Film [ ] Cd-109 [ ][ ][ ]sec. Soils [ ] Am-241 [ ][ ][ ]sec.

Element	Reading (µg/cm²; ppm)	Standard Deviation
Pb		
As	[ ][ ][ ][ ][ ][ ][ ][ ]	
Cd	[ ][ ][ ][ ][ ][ ][ ][ ]	
Ni		
Cr	[ ][ ][ ][ ][ ][ ][ ][ ]	
Ba		
Mn	[ ][ ][ ][ ][ ][ ][ ][ ]	
Se	[ ][ ][ ][ ][ ][ ][ ][ ]	
V		
Cu		
Zn		
K		
Ca		
Co		
Fe		
Mo		
Tl		[ ][ ][ ][ ].[ ][ ][ ][ ][ ]
Ag		[ ][ ][ ][ ][ ][ ][ ][ ][ ][ ]
Sr		[ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][
U		[ ][ ][ ][ ][ ][ ][ ][ ][ ]
Th		[ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][
Sn		
W		[ ][ ][ ][ ][ ][ ][ ][ ][ ][ ]
Ti		[ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][ ][
Rb		[ ][ ][ ][ ][ ][ ][ ][ ][ ][ ]
Ir		
Hg		
	QA/QC Signature:	[][]