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Environment & Remediation Support Services

Standard Operating Procedure

for **X-RAY FLUORESCENCE ANALYSIS**

APPROVAL SIGNATURES:

Subject Matter Expert:	**Organization**	**Signature**	**Date**
David Vaniman	ERSS	Signature on File	11/30/06
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1.0 PURPOSE AND SCOPE

The purpose of this procedure is to provide instructions for the operation of the Rigaku 3064 X-Ray Fluorescence Spectrometer by the Environment & Remediation Support Services (ERSS) Division of the Los Alamos National Laboratory (Laboratory).

2.0 BACKGROUND AND PRECAUTIONS

2.1 Background

This procedure is to be used in conjunction with the approved Site Specific Health and Safety Plan (SSHAP).

The XRF System uses an intense beam of X-Rays of specified energy that strike and interact with constituent elements of the target specimen to produce characteristic X-Rays of those elements. The characteristic X-Rays are detected with a wavelength spectrometer and scaled. The scaled signal is corrected for absorption and fluorescence effects and compared with standard specimens of known elemental concentration. Unknown compositions ranging from trace to major amounts can be quantitatively determined.

Detailed operating instructions for the XRF System are given in the Rigaku Instruction Manuals 1 and 2, the Rigaku Dataflex Software Manual, and the XRF-11 User's Guide, all of which are stored in the XRF Laboratory.

2.2 Precautions

None.

3.0 EQUIPMENT AND TOOLS

- Rigaku 3064 Sequential X-Ray Fluorescence Analyzer
- Rigaku ASC-24 Sample Changer
- Digital Equipment Microvax III (controller)
- Rigaku Dataflex (commercially acquired software package)
- XRF-11 (commercially acquired software package)

4.0 STEP-BY-STEP PROCESS DESCRIPTION

4.1 Intensity Calibration

Task Leader	1.	Ensure samples to be examined with the SX-50 are in a form or size that can be inserted into, or attached to, an SX-50 stage mount. [NOTE: Quantitative microanalysis routines assume that all samples for analysis will be relatively flat and that analysis sites will be normal to beam incidence.]
	2.	Determine whether or not non-conductive samples will be given an electrically conductive coating before being placed in the SX-50.

Task Leader (Continued)	3.	Use carbon unless specific applications benefit from other types of conductive coatings. [NOTE: Carbon coating procedures are outlined in the Ladd Vacuum Evaporator Instruction Manual.]

4.2 XRF-11 Standard Reference

Task Leader	1.	Calibrate quantitative X-Ray analysis routines by using standards traceable to the National Institute of Standards and Technology (NIST) or well characterized materials published in credible technical journals and widely used by micro-analysts.
	2.	Document within notebooks the basis for usage of standards from other sources for specification applications.

4.3 Detector Resolution Checks

Task Leader	1.	Review detailed operating instructions for the SX-50 system which are given in the SX-50, IMIX, and VISILOG (VISIVIEW) Manuals stored in the laboratory near the instrument.
	2.	Refer to the manuals when questions arise or consult with the Machine Custodian to solve specific problems.

4.4 Standards

Task Leader	1.	Process image and elemental information following the instructions in the SX-50, IMIX, and VISILOG Manuals.
	2.	Photograph and/or print on paper image and elemental information following instructions in the SX-50 Reference Manual, if desired. [NOTE: The Cameca software package “QuantiView” comprises software used for acquisition and reduction of quantitative elemental data. “QualiView” is the Cameca software package used for acquisition of qualitative elemental information, and “VisiView” is the image processing software package. Data reduction is based on the methods of Pouchou and Pichoir (1985).]

4.5 Environmental Conditions

Task Leader	1.	Base sample identification on the unique identifier marked on the sample. [NOTE: This will typically be an etched identification on the thin section that has been coated for analysis.]

4.6 Sample Preparation

Task Leader	1.	Determine criteria for recognizing and evaluating potential sources of error and uncertainty. [NOTE: These criteria will be indicated by the Procedure User's inability to obtain a quality image or to generate a semi-quantitative or quantitative analysis within tolerance limits.]
	2.	Base acceptance criteria for quantitative analysis of samples on acceptable analyses of appropriate standards. [NOTE: Use as a general guide a value of 2 sigma (i.e., if standard analysis are within 2 sigma based solely on counting statistics of the list or published values, then the analysis is acceptable). Procedure User's may define different acceptance criteria (e.g., charge balance ratios).]

4.7 Loss on Ignition and Supplemental Ferrous/Ferric Determinations

[NOTE: Malfunction of the SX-50 System is readily detectable by the Machine Custodian during operation of the instrument.]

Task Leader	1.	Contact the Machine Custodian when concerns are raised regarding possible equipment malfunction during operation.

4.8 Sample Control

[NOTE: Normal operating conditions as performed by trained Procedure Users present no safety hazards.]

4.9 Sources of Error and Uncertainty

[NOTE: Normal interior building temperature and humidity are acceptable for the operation of the SX-50 System. Cooling water for the SX-50 diffusion pump and electronic chassis is supplied by the building chilled-water system maintained in the range of 55 to 65 degrees Fahrenheit.]

Task Leader	1.	Monitor to ensure ambient air temperature for the SX-50 System ranges between 60 and 80 degrees Fahrenheit.
	2.	Take extra precaution to ensure system stability by checking standards often if environmental conditions move out of range during operation of the SX-50 System in WDS mode.

4.10 Safety Considerations

Machine Custodian	1.	Check the accuracy of the computer-generated micrometer marker on the annually against NIST Reference Material 484, SEM Magnification Standard. [NOTE: Tolerance is +/- 10%.]
	2.	Arrange for repair of the instrument when it is found to be out of tolerance.

4.11 Records

ERSS Project Participants	1.	Submit the following records generated by this procedure to the Records Processing Facility: • Notebook records of the sample handling and results of analysis relevant to production of XRF data; and • Data submittals for the ER electronic database.

5.0 PROCESS FLOW CHART

Flow chart is to be included at a later date.

6.0 ATTACHMENTS

None.

7.0 REVISION HISTORY

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Revision No. *[Enter current revision number, beginning with Rev.0]*	Effective Date *[DCC inserts effective date for revision]*	Description of Changes *[List specific changes made since the previous revision]*	Type of Change *[Technical (T) or Editorial (E)]*
0.0	2/9/07	Reformatted and renumbered, supersedes SOP-9.13	E

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