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Environmental Programs Directorate
Engineering & Technology

Standard Operating Procedure

for **X-RAY FLUORESCENCE ANALYSIS**

APPROVAL SIGNATURES:

Subject Matter Expert:	**Organization**	**Signature**	**Date**
David Vaniman	EES-14	Signature on File	9/2/2011
Quality Assurance Specialist:	**Organization**	**Signature**	**Date**
Paul Lowe	QA-IQ	Signature on File	9/2/2011
Responsible Line Manager:	**Organization**	**Signature**	**Date**
Danny Katzman	ET-EI	Signature on File	9/2/2011

1.0 PURPOSE AND SCOPE

The purpose of this procedure is to provide instructions for operation of the Rigaku ZSX Primus II X-Ray Fluorescence Spectrometer by the Engineering & Technology Environmental Investigations (ET-EI) division of 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 X-Ray Fluorescence Spectrometer Instruction Manual and the X-Ray Fluorescence Spectrometer Document for Maintenance, both of which are stored in the X-Ray Laboratory, TA-3, SM 494, room 103.

2.2 Precautions

None.

3.0 EQUIPMENT AND TOOLS

- Rigaku ZSX Primus II Sequential X-Ray Fluorescence Analyzer
- Haskris RW175 Heat Exchanger
- DELL Optilplex 780 PC

4.0 STEP-BY-STEP PROCESS DESCRIPTION

4.1	**Intensity Calibration**	
Task Leader	1.	Standards with known concentrations will be analyzed prior to each run and calibration curves will be derived to determine unknown concentrations.
4.2	**Detector Resolution Checks**	
Task Leader	1.	The Rigaku ZSX Primus II contains two X-ray counters, one flow proportional and one scintillation. The resolution checks are performed daily as described in the X-ray Fluorescence Instruction Manual.

4.3	**Standards**	
Task Leader	1.	Certified standards include standards purchased from NIST and standards acquired from other analysts that have been widely distributed and for which composition and homogeneity have been documented in technical publications. An updated list of certified standards can be found in the X-ray Laboratory.
4.4	**Environmental Conditions**	
Task Leader	1.	Normal interior building temperature and humidity are acceptable for the operation of the Rigaku ZSX Primus II. The X-ray tube is cooled by the Haskris RW175 Heat Exchanger.
4.5	**Sample Preparation**	
Task Leader	1.	Samples will be prepared as fused disks, using a weighed mixture of sample powder and lithium tetra/meta borate flux. Sample powders will typically be prepared by pulverizing samples in a shatterbox or ball mill, although other powders may be used. A split of each powder will be used to obtain loss-on-ignition (LOI) weights whenever sufficient powder is available. Records of sample preparation will be maintained in the Sample Preparation Laboratory
4.6	**Loss on Ignition and Supplemental Ferrous/Ferric Determinations**	
Task Leader	1.	Typical loss-on-ignition (LOI) values reflect loss of water from rock samples; in iron-rich samples the LOI determination may be positive as a result of oxidation of ferrous iron. In such samples the LOI data can be supplemented by determination of ferrous iron by titration (Husler, 1989).
4.7	**Sample Control**	
Task Leader	1.	Samples will be tracked by fusion log numbers that can be correlated with unique EP project geologic sample identifications.
4.8	**Sources of Error and Uncertainty**	
Task Leader	1.	Potential sources of instrumental error or uncertainty beyond those of fixed physical and instrumental limitations will be indicated by the certified operator's inability to generate quantitative analyses of certified standards within accepted two-sigma tolerance limits. Potential errors arising from presence of unexpected or unaccounted for elements will be indicated by inability to achieve totals within a range of 98.5-101.5%.
4.9	**Safety Considerations**	
Machine Custodian	1.	Normal operating conditions as performed by certified operators present no safety hazard. Safe operations are covered IWD 1135-Rigaku, Operation of Rigaku XRF.

4.10 Records Management

Project Participants	1.	Submit records and/or documents generated to the Records Processing Facility according to EP-DIR-AP-10003, Records Management Procedure for ADEP Employees: • Results of analysis relevant to production of XRF data.

5.0 PROCESS FLOW CHART

Flow chart is to be included at a later date.

6.0 ATTACHMENTS

None.

7.0 REVISION HISTORY

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	2/9/07	Reformatted and renumbered, supersedes SOP-9.13	E
0	9/7/2011	New document control number assigned; Supersedes EP-ERSS-SOP-5047, R0; Revised Acquisition of Rigaku ZSX Primus II; updated organizational changes	T/E