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Environment & Remediation Support Services

Standard Operating Procedure

for **OPERATION OF SIEMENS D-500 X-RAY DIFFRACTOMETERS**

APPROVAL SIGNATURES:

Subject Matter Expert:	**Organization**	**Signature**	**Date**
David Vaniman	ERSS	Signature on File	11/30/07
Quality Assurance Specialist:	**Organization**	**Signature**	**Date**
Jackie Kolakowski	ERSS	Signature on File	12/6/06
Responsible Line Manager:	**Organization**	**Signature**	**Date**
Craig Eberhart	ERSS	Signature on File	12/06/06

1.0 PURPOSE AND SCOPE

The purpose of this procedure is to describe the methods, procedures, and documentation used to obtain S-Ray powder diffraction data from the Siemens D-500 X-Ray Powder Diffractometers used by the Environment & Remediation Support Services (ERSS) Division of the Los Alamos National Laboratory (Laboratory).

2.0 BACKGROUND AND PRECAUTIONS

2.1 Background

Powder X-Ray Diffraction is a method by which investigators can identify the minerals present in a rock and obtain quantitative information on their abundance's and physical properties.

2.2 Precautions

Malfunctions of the equipment are readily apparent. For the diffractometers, one either obtains counts or no counts.

The diffractometers produce ionizing radiation using high voltage sources. However, the diffractometers are interlocked such that if the panels are all in place, risk to the operator is almost nonexistent.

Only two possibilities (rare) for data corruption exist: 1) loss of X-Ray flux; or 2) occurrence of noise spikes. The first problem can be identified by the lack of any diffracted intensity (i.e., counts = 0). Such data should be discarded. The occurrence of the second possibility can be noted by "peaks" only one 2T step wide. Such data are generally usable and only need to be discarded when a noise spike overlaps on an important peak.

The person requesting XRD analyses will record of sample submittals and analysis results in their research notebook.

3.0 EQUIPMENT AND TOOLS

- Siemens D-500 X-Ray Powder Diffractometers;
- Materials Data, Inc. Jade X-Ray Data Package; and
- Materials Data, Inc. DataScan Instrument Control Package

4.0 STEP-BY-STEP PROCESS DESCRIPTION

4.1	**Calibration**	
Team Leader	1.	Calibrate and align the equipment using procedure EP-ERSS-SOP-5043, Calibration and Alignment of the Siemens Diffractometers.
4.2	**Control of Samples**	
Team Leader	1.	Track, store, ship, and handle samples in accordance with procedure EP-ERSS-SOP-5057, Handling, Packaging, and Shipping of Samples.
	2.	Exercise care to label all X-Ray runs with correct sample name, checking sample output against names on the sample bottles.

4.3	**Diffractometer Operation**	
Team Leader	1.	Turn on diffractometer as outlined in the Siemens D-500/501 Operating Instructions.
	2.	Insert sample and turn on X-Rays either by pressing the shutter-open button or by placing shutter in automatic mode.
	3.	Operate the instrument and conduct data analyses using the MDI software packages.
4.4	**System Shut-Down**	
Team Leader	1.	Shut down the diffractometer in accordance with the instruction manual or the instructions listed on the front of the diffractometer.
4.5	**Data Analysis**	
Team Leader	1.	Regress and display data using the MDI software package.
	2.	Identify crystalline phases by comparing their patterns with patterns of pure standards, patterns from the ICDD files, or with calculated patterns.
	3.	Conduct quantitative X-Ray diffraction analysis in accordance with the methods of FULLPAT (ref. Chipera and Bish, 2001).
4.6	**Documentation**	
Team Leader	1.	Ensure all raw X-Ray data stored on magnetic or optical medial is periodically backed up onto magnetic tape and stored in a fireproof safe.
	2.	Place records that are readily regenerated from the raw data (e.g., hard copy plots and peak search data sheets) in labeled three-ring binders.
4.7	**Records**	
Team Leader	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 X-Ray diffraction data; and • Data submittals for the ERSS 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

Author: David Vaniman

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	02/09/07	Reformatted and renumbered, supersedes SOP-09.03	E

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