SIEMENS X-RAY DIFFRACTION PROCEDURE

Effective Date 2/3/89

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Preparer

1/18/89
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1/23/89
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1-24-89
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SIEMENS X-RAY DIFFRACTION PROCEDURE

1.0 PURPOSE

The purpose of this procedure is to describe the methods, procedures, and documentation used to obtain x-ray powder diffraction data from the Siemens D-500 x-ray powder diffractometers.

2.0 SCOPE

This procedure applies to all ambient or controlled-temperature x-ray diffraction analysis of any rock or mineral for the Yucca Mountain project using the Siemens D-500 x-ray powder diffractometers.

3.0 PRINCIPLES

N/A

4.0 DEFINITIONS

4.1 Machine Custodian

The person who is in charge of an instrument and is responsible for the maintenance and safety of the instrument. This person's name shall be posted on the front of the diffractometers.

5.0 RESPONSIBILITIES

The Machine Custodian is responsible for both alignment and calibration of the diffractometers and the training of any potential users of the diffractometers. The principal investigator (PI) has the responsibility to assure implementation of this procedure for YMP work. The PI may delegate performance of the procedure to any properly trained and certified individual.

6.0 PROCEDURE

6.1 Overview

6.1.1 Equipment and Software Used

- o Siemens D500 X-Ray Powder Diffractometers
- o DIFFRAC500 - Siemens Commercial X-Ray Data Package.
- o Anton Paar TTK Temperature Attachment. (This attachment is capable of controlling the sample temperature between liquid nitrogen (-195.8°C) and ~300°C under a vacuum).
- o Oxford Instruments ITC4 Temperature Controller.
- o Roughing Vacuum Pump.

6.1.2 Critical Laboratory Setup Parameters

The diffractometer is set up in accordance with the Standard Operating Procedure (SOP) posted on the front of the diffractometer enclosure. One may also refer to the instruction manual, reference 8.1.

6.1.3 Special Environmental Conditions

None.

6.2 Traceability

6.2.1 Complete records shall be maintained on every sample run. Diffraction patterns and peak-search outputs may be placed in labeled binders. All x-ray runs shall be recorded in a controlled YMP logbook and shall include the date of the run, sample identification, signature of the operator, diffractometer used, and any unusual run conditions.

6.2.2 Great care shall be exercised to label all x-ray runs with correct sample name, checking sample output against names on the sample bottles.

6.3 Diffractometer Operation

6.3.1 Turn on diffractometer as stated in 6.1.2.

6.3.2 Insert sample and turn on x-rays either by pressing the shutter-open button or by placing shutter in automatic mode.

6.3.3 The instrument is operated and data analyses conducted using the Siemens software package, DIFFRAC 500. Refer to the DIFFRAC 500 users manual, reference 8.2.

6.3.4 If the samples are to be x-rayed at variable temperatures using the Anton Paar TTK attachment, the TTK attachment is operated and maintained as outlined in the TTK instruction manual (ref. 8.7), and the Oxford ITC4 temperature controller is operated as outlined in the ITC4 Operators Manual (ref. 8.8).

6.4 System Shut-Down

The diffractometer is shut-down in accordance with the SOP posted on the front of the diffractometer enclosure. One can also refer to the instruction manual, reference 8.1.

6.5 Data Analysis

Crystalline phases are identified by comparing their patterns with patterns of pure standards, patterns from the JCPDS files (ref. 8.3, ref. 8.6), or with calculated patterns. Quantitative x-ray diffraction analysis is conducted in accordance with TWS-ESS-DP-116 (ref. 8.5).

7.0 QUALITY ASSURANCE

7.1 Personnel

Only those persons YMP-certified in accordance with the Personnel Certification Procedure shall perform YMP-related analysis on this equipment.

7.2 Calibration

There is a separate procedure, Procedure: Alignment of the Siemens Diffractometer (TWS-ESS-DP-24) for alignment and calibration. Temperature calibration of the TTK attachment shall be performed at several temperatures, preferably at liquid nitrogen temperature and in boiling water, as outlined in reference 8.8. Calibration of the sample temperature may be performed using an independent temperature measuring device such as the thermocouple probe operated in accordance with TWS-ESS-DP-105 (Thermal Calibration Procedure) or using known melting point(s) of a crystalline phase.

7.3 Records

7.3.1 All x-ray runs shall be recorded in a controlled YMP logbook, records of which shall include date of run, sample identification, analyst's signature, diffractometer used, and any unusual run conditions.

7.3.2 A master catalog of all automatic x-ray runs shall be maintained and periodically updated and stored on magnetic or optical media.

7.3.3 All rav x-ray data stored on magnetic or optical media shall periodically be backed up onto magnetic tape and stored in a fireproof safe.

7.3.4 Records that are readily regenerated from the rav data such as hard copy plots and peak search data sheets may be placed in labeled three ring binders.

7.4 Accept/Reject Criteria

7.4.1 Only two possibilities (rare) for data corruption exist:
1) loss of x-ray flux;
2) occurrence of noise spike.

Number 1 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 2θ step wide. Such data are generally usable and only need to be discarded when a noise spike overlaps on an important peak.

7.4.2 During a variable-temperature x-ray run, data shall be rejected if, after a run is completed and the TTK attachment is opened, the sample is found to have fallen or blown (due to vacuum) out of the sample holder.

7.4.3 The dated entry into the YMP Siemens logbook for each x-ray run shall constitute evidence that the procedure has been implemented and satisfactorily accomplished for the x-ray run.

7.5 Procedural Deviations

Deviations from this procedure shall be fully documented in the Siemens YMP logbook or in the analyst's YMP notebook, whichever is most appropriate, explaining the deviation and the effects if may have on the resulting vork.

7.6 Storage, Shipping, and Handling

7.6.1 Samples will be tracked, stored, shipped, and handled in accordance with the procedure for Sample Identification and Control for Mineralogy-Petrology Studies (Ref 8.4).

7.6.2 Special equipment handling, shipping, and storage requirements do not apply since the diffractometers remain operational and are never moved.

8.0 REFERENCES

8.1 Siemens D500/501 Operating Instructions, C72000-B3463-A42, Siemens Corporation, Cherry Hill, New Jersey.

8.2 Siemens DIFFRAC 500 User's Manual, Siemens Corporation, Cherry Hill, New Jersey

8.3 Powder Diffraction File, Search manual, Fink Method, Inorganic, Publication SMF-26 (Joint Committee on Powder Diffraction Standards, Swarthmore, Pennsylvania, 1976, pp. 1017.

8.4 TWS-ESS-DP-101: Sample Identification and Control for Mineralogy-Petrology Studies.

8.5 TWS-ESS-DP-116: Quantitative X-Ray Diffraction Data Reduction Procedure.

8.6 Mineral Powder Diffraction File, Data Book (Joint Committee on Powder Diffraction Standards, Swarthmore, Pennsylvania, 1986) 1396 pp.

8.7 Anton Paar Instruction Manual for TTK-Temperature Attachment for low and medium Temperature, Anton Paar KG., Karntnerstrasse 322, A-8054 Graz/Austria.

8.8 Oxford Instruments Operating Manual for Model ITC4 Temperature Controller, May 1987 Issue 3, Oxford Instruments Limited, Osney Mead, Oxford OX2 ODX, England.

8.9 TWS-ESS-DP-105: Thermal Calibration Procedure.

9.0 ATTACHMENTS

None.

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