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environmental restoration project

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Environmental Restoration Project
Standard Operating Procedure

for:

Calibration and Alignment of the Siemens Diffractometers

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Revision Log

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CALIBRATION AND ALIGNMENT OF THE SIEMENS DIFFRACTOMETERS

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CALIBRATION AND ALIGNMENT OF THE SIEMENS DIFFRACTOMETERS

1.0 PURPOSE

This Standard Operating Procedure (SOP) states the responsibilities and describes the methods, procedures, and documentation required to align and calibrate the Siemens D500 X-ray powder diffractometers at the Los Alamos National Laboratory (Laboratory) ER Project.

2.0 SCOPE

This SOP is a manadatory document and shall be implemented by all ER Project participants when aligning and calibrating the Siemens D500 X-ray powder diffractometers for the ER Project

3.0 TRAINING

3.1 All users of this SOP are trained by self-study, and the training is documented in accordance with QP-2.2.

3.2 The Geology Task Leader (TL) will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with QP-2.2.

3.3 Due to safety considerations, the machine custodian is solely responsible for performing machine alignment. The ER user and the machine custodian performing calibration have the responsibility to adhere to this procedure.

4.0 DEFINITIONS

4.1 *Machine Custodian* — The person who is in charge of an analytical X-ray instrument and who is responsible for the maintenance and safety of the instrument.

4.2 *Shorting Plug or Key* — The plug (θ-2θ machine) or key (θ-θ machine) used to bypass the safety circuits to allow alignment. To be used only by the machine custodian.

4.3 *NIST* — National Institute of Science and Technology.

4.4 *Site-Specific Health and Safety Plan (SSHASP)*—A health and safety plan that is specific to a site or ER-related field activity that has been approved by an ER health and safety representative. This document contains information specific to the project including scope of work, relevant history, descriptions of hazards by activity associated with the project site(s), and techniques for exposure mitigation (e.g., personal protective equipment [PPE]) and hazard mitigation.

5.0 BACKGROUND AND PRECAUTIONS

Note: This SOP is to be used in conjunction with an approved Group environment, safety, and health plan.

Calibration and, if necessary, alignment of the Siemens X-ray diffraction equipment must be performed on a periodic basis in order to assure that all data collected on the instruments yield accurate d-spacings. The calibration described here involves only d-spacings. Calibration may use any well-characterized d-spacing standard that is traceable to NIST, such as their standard reference materials. It is not necessary that the diffractometer have no error in 2θ. Calibration is performed to determine the magnitude and direction of any error in 2θ so that appropriate calibration curves may be used when obtaining d-spacings. Alignment is performed only when calibration data reveal a significant error in alignment, usually after modification of the instrument (e.g., installation of new X-ray tube, installation of alternate sample holders, installation of the TTK).

5.1 Certain aspects of the alignment procedure require that one bypass the interlocks to work on the instrument with the X-ray beam on. The use of extreme caution and safety equipment such as lead-lined gloves is mandatory.

5.2 Highly accurate d-spacing measurements are not crucial to qualitative phase identification or quantitative phase analysis. However, it is useful and good laboratory practice to have a well- aligned diffractometer. In general, errors less than 0.02° 2θ are desirable, but errors up to 0.10° 2θ are acceptable in many cases.

5.3 There are few sources for error that would not be readily detected. A potential situation where minor error could occur is when the linear absorption coefficient or transparency of a sample being run is not similar to that of the standard used to calibrate the instrument. In this case, if accurate d-spacings were desired, the instrument should be calibrated with a standard of appropriate linear absorption coefficient or an analytical correction should be performed to correct for transparency effects.

5.4 The calibration sticker paced on the instrument shall constitute evidence that the procedure has been implemented and satisfactorily accomplished.

6.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

6.1 Focus Area Leader

6.2 Team Leader

6.3 Quality Program Project Leader

6.4 Geology Task Leader

6.5 ER Project personnel

7.0 EQUIPMENT

7.1 NIST SRM 640a or 640b Si Powder d-spacing Standard.

7.2 Granite surface plate or precision flat plate.

7.3 Height Gauge.

7.4 Dial Indicator Gauge.

7.5 Precision Right-Angle Block.

7.6 Assorted Slits.

7.7 Strip Chart Recorder.

7.8 Assorted Screw Drivers and Wrenches.

7.9 Shorting Plug or Key.

7.10 MDI software packages (MDI DataScan 3.2 and MDI Jade 5).

7.11 LCLSQ, lattice parameter refinement program.

8.0 PROCEDURE

Note: ER Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

Note: Deviations from SOPs are made in accordance with QP-4.2, Standard Operating Procedure Development and documented in accordance with QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

Note: Only NIST traceable standards such as NIST SRM 640b shall be used to calibrate or check the calibration of the Siemens X-ray powder diffractometers. Deviations from this procedure shall be documented as required in QP-4.2.

8.1 Calibration of Diffractometers

As required, either by time elapsed since last annual calibration check or by a change in the instrument or sample holder geometry, the instrument shall be calibrated against a certified standard reference material (SRM), such as NIST SRM 640a or 640b (Si powder), or against a secondary reference standard traceable to an NIST standard.

8.1.1 The calibration standard may be mounted either in a cavity or as a thin film to minimize specimen transparency effects.

8.1.2 The calibration standard shall be examined on the instrument over a 2θ range practical for the instrument and standard. A recommended procedure is to examine NIST SRM 640a or 640b from ~18° 2θ to~150° 2θ using 0.01-0.02°-2θ steps and counting for at least 1.0 sec per step.

8.1.3 Determine peak positions for all diffraction maxima using the profile fitting routine in the Jade software package, typically using the Pearson VII profile. The results of the profile refinement may be compared with the certified values for the standard to assess the magnitude of the 2θ error.

8.2 Alignment of the Diffractometer

If a calibration check reveals significant errors in alignment (2θ errors generally greater than 0.02° 2θ), an alignment of the diffractometer should be performed. If only some aspects of the instrument have been modified, only an abbreviated alignment will be required. For example, if a new X-ray tube has been installed, only the tube height will require alignment. When the TTK stage is installed, both sample height (6.2.3) and θ zero point (6.2.5) must be checked and adjusted if necessary.

6.2.1 Specimen holder pin height. This step is performed only if the specimen stop-screws have been modified. Thus, in practice it is seldom if ever performed. 6.2.1 is not applicable to the TTK stage.

8.2.1.1 Check the three specimen stop-screws on the specimen holder for the same height. The two outer pins should be coplanar with the inner reference pin. Tools Needed are as follows:

- Granite surface plate or precision flat plate;
- Height gauge;
- Dial indicator gauge; and,

- Precision right-angle block.

8.2.1.2 Set the level of the specimen holder using the bar by the inner stop-screw.

8.2.1.3 Set the dial indicator height using the bar or inner screw.

8.2.1.4 Before adjusting stop-screws, the locking screws should be loosened. There is one for each stop-screw.

8.2.1.5 When the height of the three stop-screws is set, retighten the locking screws.

8.2.2 Install the specimen holder on the specimen carrier using the push rod that is built into the carrier. Use the flat surface of the rod and try to make the holder parallel to the rod. This step is performed only if the specimen holder has been removed from the specimen carrier.

8.2.3 (With shorting plug or key in). The axis of rotation of the goniometer is on the specimen surface when the maximum intensities appear at θ = 0° (front side) and 180° (back side). Note that the maximum intensities are approximately the same. This step is done only with the θ-2θ diffractometer and only if the specimen height has been modified. In practice, this is seldom if ever performed.

8.2.3.1 Slit positions: I II III IV Slit: 1°1° --- .018° (These are final slits - can start with wider slits, particularly #IV.)

8.2.3.2 Install glass alignment slit.

8.2.3.3 Insert an absorber plate into the direct beam to limit counts to below ~40,000 counts/sec. Set tube power to minimum (~7kV and ~3mA).

8.2.3.4 Find the maximum intensities by adjusting θ and 2θ independently and alternately on front (θ = θ°). Record 2θ and full scale counts.

8.2.3.5 Find the maximum intensities as above on back side (θ at 180°). This maximum intensity should be about the same value as the front side (θ = θ°). If intensity is different, move holder up and down - if you can't get it, the three pins are not in the same plane and 6.2.1 should be performed.

8.2.3.6 It is not important at this time that the θ and 2θ counting mechanisms read zero, but note only the 2θ values.

8.2.3.7 Ignore the θ value alltogether. The 2θ values only need to be to be the same, for θ front and back.

8.2.3.8 Compare the 2θ numbers found on front and back sides. The difference should be no greater than 0.02°. Do a θ-2θ scan on both front and back to insure that you are aligning on the main peak.

8.2.3.9 If the difference is greater than 0.02°, the specimen holder has to be moved closer or farther away from the push rod. If the front 2θ value is larger than the back 2θ value, the holder needs to moved towards the pin, i.e., down.

8.2.3.10 If the intensities (front and back sides) are not within ~10% of each other, then step 6.2.3.8 should be rechecked.

8.2.4 Adjust tube height for maximum intensities when 2θ is reading zero. Use only the 0.018 slit in position IV and the glass slit in the sample position. Use no other slits.

8.2.4.1 Loosen the nut at top of tube slightly with small wrench in the D500 accessory kit. Loosen the allen screw at the lower back of the tube.

8.2.4.2 Move θ to front side.

8.2.4.3 Adjust 2θ off of maximum intensities, slightly toward zero.

8.2.4.4 Adjust tube height to get back toward maximum intensity.

8.2.4.5 Adjust θ so maximum intensity is restored.

8.2.4.6 Repeat above steps until you get zero 2θ with maximum intensity.

8.2.4.7 Tighten the nut and screw loosened in 6.2.4.1 above.

8.2.4.8 You should not have to set the height again unless a new tube is installed.

8.2.4.9 If the Kevex Psi detector is installed, steps 6.2.4.1 through 6.2.4.8 cannot be performed. Instead, tube-height alignment is performed using a high-angle reflection of a NIST standard such as Si. The tube height is incrementally adjusted until the high-angle reflection is located at the correct 2θ value obtained using profile refinement. Turning the allen screw on top of the tube clockwise (from above) lowers the observed peak positions.

8.2.5 With the glass slit in the sample position, adjust θ for zero with maximum intensity by means of the eccentric screw (Item 183 in Fig. 20 of reference B). As in previous 2θ adjustment, move toward zero slowly. Peak the eccentric in small steps until you get to zero. Recheck after adjusting slits (slits 0.1, 3, --, 0.018 in slit positions I, II, III, and IV, respectively). This alignment cannot be performed with the Kevex Psi detector installed, and the scintillation detector must be installed. This step needs to be performed only if the specimen carrier has been removed.

8.2.6 Adjust slit holder for slit positions I and II. Put in the smallest slits. If there is no intensity change, the slits are properly aligned. Do the same for slit positions III and IV.

8.2.6.1 Insert the following slits into the slit holder: Slit position: I II III IV Slit: 0.1°3°--- 0.018°.

8.2.6.2 Remove the sheet metal shield of the slit holder and, if necessary, the Soller slits.

8.2.6.3 Loosen the locking screws.

8.2.6.4 Reposition the sheet metal shield (radiation protection).

8.2.6.5 Displace the slit holder by means of the two adjusting screws until the ratemeter or chart recorder indicates maximum intensity. In order to turn one adjusting screw in, it is necessary to loosen the opposite screw because these are pressure screws.

8.2.6.6 Insert smallest (0.1°) slit in position II, and you should get the same intensity or you must readjust.

8.2.6.7 Loosen two bottom screws for slit III and IV holder (allen screws are right behind slit III in white painted metal). Adjust slit holder for III and IV with the smallest slits in positions III and IV.

8.2.7 Run a scan on the glass slit through zero.

8.2.7.1 Set up for scan: Slit position: I II III IV Slit: 1°1°1°0.018°

8.2.7.2 Set generator to minimum power (~7kV/3mA for FK60-04 Cu tube), ratemeter to ~2x10^4 counts/sec full scale, time constant = ~0.2, absorber in direct beam, diffractometer set to step at 1/5°/min upscale, the chart recorder set to feed at 2 cm/min, have θ and 2θ coupled.

8.2.7.3 Start scan at -999.500° 2θ (scan range is dependent on tube type).

8.2.7.4 This scan must be run automatically on the θ-θ machine, using the same conditions, starting 2θ position, and a 0.01° 2θ step size.

8.2.7.5 A good alignment will fulfill the following conditions:

- The maximum of the primary beam must be within 0.006° of 0°.
- The amplitudes of the two side maxima should not differ from one another by more than ~30%.
- The amplitude of the main maximum should be about
- three times as high as those of the side maximum.

8.2.8 Optimization of 2θ calibration. This is done using a certified d-spacing standard such as the NIST SRM 640 Si series and compensates for systematic errors such as sample transparency and 2θ offset.

8.2.8.1 Mount the calibration standard as in 6.1.1.

8.2.8.2 With θ and 2θ synchronized, drive to high angles (typically over 100° 2θ) and locate a peak of appreciable intensity (one that can be easily detected). Set the X-ray tube kV and mA to typical operational values (45 kV, 35 mA).

8.2.8.3 Perform a quick automatic scan over the range of the peak and determine the observed peak position using the profile fitting routine in the MDI Jade software.

8.2.8.4 If the position of maximum intensity is not at the certified value, adjust the peak position by adjusting the tube height as in 6.2.4. When this step is complete, the position of maximum intensity should be at the certified value.

8.2.8.5 Refine the adjustments made in 6.2.5 and 6.2.6.

8.2.8.6 For a quick check of the quality of alignment, one or two high-angle reflections may be measured as in 6.1. After determining that alignment is sufficient, perform complete calibration as in 6.1.

8.3 Optimization and calibration of the solid-state Si detectors.

8.3.1 Perform detector energy calibration as described in Kevex 4911 user's manual.

8.3.2 Set the detector bias to the appropriate value listed on the front of each diffractometer.

8.4 Data Analysis

The MDI Jade software package is used to regress all data produced or required by this procedure. The program LCLSQ used to determine specimen displacement errors.

8.5 **Lessons Learned**

During the performance of work, ER Project personnel shall identify, document and submit lessons learned in accordance with QP-3.2, Lessons Learned. This QP can be located at: http://erinternal.lanl.gov/home_links/Library_proc.htm.

9.0 REFERENCES

ER Project personnel may locate the ER Project Quality Management Plan/ER Project QP requirements crosswalk at http://erinternal.lanl.gov/home_links/Library_proc.htm.

The following documents have been cited within this procedure.

MDI (Materials Data Incorporated), DataScan 3.2, An Automated Control and Data Acquisition System for X-ray Diffractometers, 1995-1999.

MDI (Materials Data Incorporated), Jade 5, XRD Pattern Processing, 1991-1999.

Kevex 4911 Single Channel Spectrum Analyzer User's Manual, #7257-4911.

QP-2.2, Personnel Orientation and Training

QP-3.2, Lessons Learned

QP-4.2, Standard Operating Procedure Development

QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities

10.0 RECORDS

Calibration records shall include date of calibration, standard used, signature of the person performing calibration, the peak positions observed for the standard and the necessary corrections to 2θ. Alignment records shall include the date of alignment, the procedures performed, and the signature of the machine custodian performing the alignment. A new calibration sticker shall be placed on the front of the diffractometer following each calibration identifying when the instrument was calibrated, when the next calibration is due, and the name of the person who did the calibration.