SOP: SOPSAXSLA
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Small Angle X-ray Scattering, Rotating Anode

PURPOSE:

This Standard Operating Procedure (SOP) states the responsibilities and describes the methods, procedures, and documentation used to obtain Small Angle X-ray Scattering data from the Bruker X-ray SAXS at the Department of Chemistry, Texas A & M University

POLICY:

Data must be collected in a manner to provide maximum coverage and optimal quality to produce the best possible results.

BACKGROUND AND PRECAUTIONS

1. SAXS is a method by which investigators can identify the materials and obtain qualitative and quantitative information on their abundance's and physical properties.
2. The diffractometer produces ionizing radiation using high voltage sources. The diffractometers are safety interlocked such that if the panels are all in place, risk to the operator is negligible.
3. The person requesting XRD analyses will record of sample submittals and analysis results in the instrument notebook

TRAINING

- All users must be trained as specified by the Environmental Health and Safety Office (EHSO at Texas A & M University) guidelines pertaining to radiation producing devices.

Operation of Siemens D-500 X-ray Diffractometers

- The **X-ray Diffraction Laboratory manager** will monitor the proper implementation of this procedure and ensure that users have completed all applicable training assignments in accordance the EHSO guidelines.

Approved :JHR 1/8/2009

RESPONSIBLE PERSONNEL

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

- X-ray Laboratory Manager
- X-ray Laboratory Assistant Manager
- The X-ray Powder Diffraction User

MATERIALS:

- Bruker D8 NANOSTAR small angle X-ray scattering instrument
- NANOFIT Program for Data Analysis
- Topas. Program for Retvield Refinement

PROCEDURE:

Instrument Custodian

The instrument custodian is responsible for both alignment and calibration of the diffractometers and the training of any potential users of the diffractometers.

Calibration

The instrument will be aligned monthly. A Silver Behenate standard will be employed as specified by the Bruker Operation Manual. The results of the calibration will be available to all users and posted on the instrument.

Control of Samples

Samples will be tracked, stored, shipped, and handled by the user. Samples that are investigated by the X-ray Diffraction Laboratory Staff will be tracked, stored, handled and shipped in accordance with the Sample Handling and Security SOP (SOP –SAMP)

Diffractometer Operation

1. Turn on rotating anode generator.
2. Insert sample as described by the Sample Mount SOP (SOP-MOUNT)
3. The instrument is operated by the COMMANDER software.

Data Analysis

1. The data are regressed and displayed using the NANOFIT software package.

2. Crystalline phases are identified by comparing their patterns with patterns of pure standards, patterns from the ICDD files, or with calculated patterns.

Procedural Deviations

Deviations from this procedure and the effects it may have on the resulting work shall be documented.

Instrument Control

1. A BRUKER NANOSTAR SAXS (small angle x-ray scattering) instrument is employed for data collection.
2. The X-ray radiation employed is generated from a Cu Bruker FR591 rotating anode fine-focus X-ray source (K_α = 1.54184Å with a potential of 40 kV and a current of 90 mA).
3. The X-rays are filtered through cross-coupled Gobel mirrors and collimated (collimator distance = 146mm) by three 0.4mm pin-holes.
4. The detector [MWPC hi-star area detector] is set at 175mm from the sample and the sample chamber and x-ray paths are evacuated.
5. The detector distance and beam intersection is calibrated employing a silver Behenate standard.
6. The instrument is controlled with the SAXS software suite (Microsoft Win 2000 operating system) and the data is collected in the still (add) mode.
7. A 1mm quartz capillary is filled with the liquid sample and placed in the sample chamber at room temperature.
8. The sample chamber and x-ray beam paths are evacuated and a 3600sec scan is preformed.
9. The glassy carbon standard is then inserted between the sample and the detector and a 300 sec standard scan is collected.
10. The sample is then replaced with distilled de-ionized water, the chamber and x-ray beam path was evacuated and a blank scan of 3600sec is completed, which is followed by insertion of the glassy carbon and a 300sec standard scan.
11. Finally the capillary is removed from the chamber and beam path evacuated and a 3600sec background scan which is followed by insertion of the glassy carbon and a 300 sec standard scan are preformed.
12. Transmission factors are then calculated for the sample and the blank. The blank is then subtracted from the sample employing the scale factor calculated by dividing the sample transmission factor by the blank transmission factor.
13. An area integration (GADDS NT software reference manual pp 1-11 to 1-15) is employed to reduced the data to a one-dimensional q ($4\pi\sin\theta/\lambda$) versus ln(Intensity) trace.

Documentation

- All raw X-ray data stored on magnetic or optical media shall periodically be backed up onto compacted disks or and stored on the Linux RAID server.
- Records that are readily regenerated from the raw data such as hard copy plots and peak search data sheets may be placed in labeled folders and stored in file cabinets.