 X-ray Diffraction Laboratory: Department of Chemistry Texas A & M University	Doc. No:	SOPPOWD8
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Standard Operating Procedure Title: X-ray Powder Diffraction: D8 Advanced		

SOP: SOPPOWD8

Last date revised: December 23 2009

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X-ray Powder Diffraction: D8 Advanced

PURPOSE:

This Standard Operating Procedure (SOP) states the responsibilities and describes the methods, procedures, and documentation used to obtain X-ray powder diffraction data from the Bruker X-ray powder diffractometer 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. Powder X-ray diffraction 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.

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- 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

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 Advanced X-Ray Powder Diffractometer.
- Eva. Program for Data Analysis
- Topas. Program for Rietveld 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 quartz 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 diffractometer by depressing the red on/off button on the right panel of the diffractometer. Wait for the amber “busy” led to deactivate.

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2. Turn on the X-rays by turning the key on the transformer face from O to I and then press the standby button. Wait 60 secs and then press the X-ray on button. The AMBER bar should be illuminated.
3. Insert sample as described by the Sample Mount SOP (SOP-MOUNT)
4. The instrument is operated by the COMMANDER software.

Data Analysis

1. The data are regressed and displayed using the EVA 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.

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.