SOP: SOPPOWGAD
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Powder Data Collection on GADDS

PURPOSE:

This document proposes procedures to facilitate data collection on the Bruker GADDS Powder X-ray Diffractometer.

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.
- 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.

RESPONSIBILITY:

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

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

MATERIALS:

- Bruker GADDS powder X-ray Diffractometer
- 10X stereo microscope

PROCEDURE:

- The instrument custodian is responsible for both alignment and calibration of the diffractometers and the training of any potential users of the diffractometers.
- The instrument will be aligned monthly. A powder 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.
- 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)

Procedural Deviations

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

Instrument Operation

1. 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.

2. Toggle ON/OFF switch on instrument control module. Allow 2 mins for boot-up.
3. Point to GADDS icon and start the data collection software
4. Point to VIDEO icon and start the video camera capture software.

Enclosure Door Operation

1. Move enclosure doors to the closed position and lock in place.

Instrument Control

1. The BRUKER D8 GADDS general-purpose three-circle X-ray diffractometer is employed for sample screening and data collection.
2. The goniometer is controlled by the GADDS software suite (Microsoft Win 2000 operating system).
3. Typically a 0.7mm nylon loop is dipped in the sample.
4. The loop is then placed on the diffractometer in a cold nitrogen stream (Oxford) maintained at 110K.
5. The sample is optically centered with the aid of a video camera such that no translations are observed as the sample was rotated through all positions.
6. The detector is set at 5 or 12cm from the sample (MWPC Hi-Star Detector, 1024x1024 pixel).
7. The X-ray radiation employed is generated from a Cu sealed X-ray tube (K_α = 1.54184Å with a potential of 40 kV and a current of 40 mA) and filtered with a graphite monochromator in the parallel mode (175 mm collimator with 0.5 mm pinholes).
8. The beam intersection coordinates are compared to the corundum (Al_2O_3) standard coordinates and changes are made accordingly.
9. A single data frames are taken at widths of -179° in θ (continuous rotation on φ: Gandolfi Scan) with an exposure time of 5 seconds. Three frame sets at 20,55 and 90^o 2-θ settings are collected.
10. The data is reduced by area integration methods to produce a single powder diffraction pattern for each frame.
11. Merging the individual powder diffraction patterns in the program EVA will produce a single one-dimensional pattern, which is reported.

Documentation

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1. All raw data stored on magnetic or optical media shall periodically be backed up onto compacted disks or and stored on the Linux RAID server.
2. Records that are readily regenerated from the raw data may be placed in labeled folders and stored in file cabinets.
3. The instrument log should be updated after each project and will be kept at the instrument control station.

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