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Standard Operating Procedure Title: Data Collection on APEXii D8-1		

SOP: SOPDATAP1

Last date revised: December 26 2008

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Data Collection on APEXii D8-1

PURPOSE:

This document proposes procedures to facilitate data collection on the Bruker APEXii Single-crystal 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. Single-Crystal X-ray diffraction is a method by which investigators can identify the materials and elucidate crystalline structure.
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.

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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 APEXii single-crystal 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 crystal 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.

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2. Toggle ON switch on instrument side-panel. Allow 2 mins for boot-up.
 - Green led will be lit
3. Point to BIS icon and start the Bruker Instrument Control software.
 - Set distance to 6.0cm when prompted
4. Point to APEX icon and start the data collection software.


Enclosure Door Operation

1. Move both enclosure doors to close position and lock in place.

Instrument Control

1. A polarizing microscope is used to identify a suitable specimen from a representative sample of crystals of the same habit.
2. The crystal is coated in a cryogenic protectant and then fixed to a nylon loop which in turn is fashioned to a copper mounting pin.
3. The mounted crystal is placed in a cold nitrogen stream (Oxford) maintained at 110K.
4. A BRUKER APEXii X-ray three-circle diffractometer is employed for crystal screening, unit cell determination and data collection.
5. The goniometer is controlled using the APEX software suite
6. The sample is optically centered with the aid of a video camera such that no translations were observed as the crystal was rotated through all positions.
7. The detector is set at 6.0 cm from the crystal sample
8. The X-ray radiation employed is generated from a Mo sealed X-ray tube ($K_{\alpha} = 0.70173\text{\AA}$ with a potential of 50 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).
9. Dark currents are obtained for the appropriate exposure time
10. A "matrix" data collection consisting of thirty data frames are taken at widths of 0.5° with an exposure time of 10 seconds.
11. The reflections harvested from the "matrix" data collection are used in the auto-indexing procedure to determine the unit cell.
12. A suitable cell is found and refined by nonlinear least squares and Bravais lattice procedures.
13. After careful examination of the unit cell, a standard data collection procedure is initiated.
14. Data collection consists of one hemisphere of data collected using omega scans, involving the collection 2400 0.5° frames at fixed angles for ϕ , 2θ , and χ ($2\theta = -28^{\circ}$, $\chi = 54.73^{\circ}$), while varying omega.

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15. After data collection, the crystal is measured carefully for size, morphology and color.

Documentation

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.