SOP: SOPABSSTR
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Absolute Structure Determination

PURPOSE:

This document proposes procedures to facilitate data collection for absolute structure determination on the Bruker GADDS 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.

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 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.
2. Toggle ON/OFF switch on instrument control module. Allow 2 mins for boot-up.
3. Point to FRAMBO 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. 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 GADDS X-ray three-circle diffractometer was employed for crystal screening, unit cell determination and data collection.
5. The goniometer is controlled using the FRAMBO software suite, version.
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 5.0cm from the crystal sample.
8. 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).
9. A “matrix” data collection consisting of sixty data frames are taken at widths of 0.5° with an exposure time of 10 seconds.
10. The reflections harvested from the “matrix” data collection are exported to the program cell_now for unit cell determination.
11. A suitable cell is found and refined by nonlinear least squares and Bravais lattice procedures.
12. After careful examination of the unit cell, a absolute structure data collection procedure is initiated.
13. Data collection consists of full hemisphere of data collected using omega scans, involving the collection ~6400 0.5° frames at fixed angles for ϕ, 2θ,

and χ (2θ = -28°, χ = 54.73°, 2θ = +28°, χ = 54.73°, 2θ = -60°, χ = 54.73°, 2θ = +60°, χ = 54.73°. 2θ = -90°, χ = 54.73°), while varying omega.

14. After data collection, the crystal is measured carefully for size, morphology and color.
15. Data is reduce with the SAINT program to render the *hkl* file with directional cosines.
16. Data is scaled (absorption corrected) with the SADABS program to generate the corrected *hkl* file.
17. The structure is solved and refined and the FLACK parameter is determined employing the TWIN routine of SHELXL.
18. A Flack parameter of 0.1 with an esd of less the 0.05 is considered proof of determination of the correct handiness of the structure and indicates successful absolute structure determination.

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 locked file cabinets.
3. The instrument log should be updated after each project and will be kept at the instrument control station.

Document control

- The goal of the laboratory document control program is to assure that all documents for a specified project will be accounted for when the project is completed.
- Accountable documents used shall include, but not be limited to, logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses.