 <b>X-ray Diffraction Laboratory: Department of Chemistry</b> <b>Texas A &amp; M University</b>	Doc. No:	SOPALIGNA
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<b>Standard Operating Procedure</b> <b>Title: X-ray Instrument Alignment – APEXII</b>		

SOP: SOPALIGNA

Last date revised: June 10, 2009

Date approved: June 10, 2009

## Instrument Alignment - APEXII

### PURPOSE:

This document proposes procedures to facilitate the alignment of the APEXII single crystal X-ray diffractometer.

### POLICY:

The instrument must be aligned and maintained in such a manner as to provide the maximum safety to the user while providing the most accurate data possible.


### 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.

### 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
- X-ray Technician

## MATERIALS:

- Bruker APEXII X-ray Diffractometer
- PIN alignment instrument
- Alignment tools
- Ruby or Sapphire Standard Crystal
- Steel Alignment Pin


## 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.

### Procedural Deviations


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

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### Instrument Alignment

1. Inspect instrument and enclosure for unsafe/unusual conditions.
2. PURGE LN2 temperature attachment.
3. Check Water/Water heat exchanger
  - a. Add water if necessary
4. Check Generator
  - a. Check Flow Rate
5. Check LYTRON/NESLAB temperature
6. Stop all programs and restart all programs.
7. Test all safety features
8. Zero all angles
  - a. Check zero and update/home axis if necessary
9. Raise LT nozzle so that it is out of the way of the instrument.
10. Move detector back to 12 cm.
11. Remove beam stop.
12. Check Collimator
13. Attach PIN alignment tool
14. Tweak alignment for max intensity.
15. Record final MV and collimator size.
16. Replace beam stop.
17. Move Detector to 5.0 cm
18. Attach Steel Goniometer/alignment pin.
19. Center scope with Optical Alignment.
20. Attach alignment crystal and center.
21. Run Matrix
  - a. Find offsets and record/update files
  - b. Save as default
22. Collect new dark currents 10, 20, 30 and 60.
23. Save and exit program
24. Back-up computer
  - a. Delete old files
  - b. Defrag
25. Record events in LOG book.

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### 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.