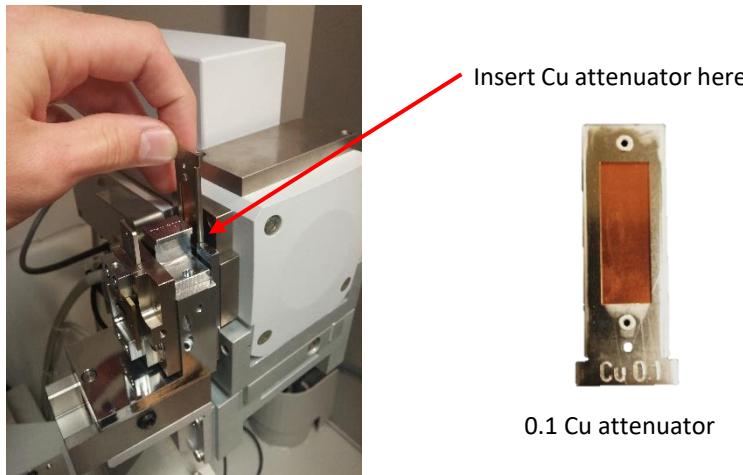


### X'Pert Data Collector Powder S.O.P.

1. Open Data Collector
  2. Login to Data Collector:
    - a. Login: AMSEC (powder) or XRR (reflectivity)
    - b. PW: AMSEC
  3. Under Instrument select Connect
    - a. A dialog box will appear, select Default
    - b. A second dialog box will appear, click "Ok"
    - c. If it asks to apply offsets, click "Yes"
  4. Once the instrument control pane appears on the left of the screen, insert your sample into the diffractometer (Be very careful with the diffractometer doors. They open/close very easily and can be easily broken)
    - a. To mount a powder sample on a glass slide do either i or ii below:
      - i. Place a small amount of powder on a slide and wet the sample with either methanol or hexanes and tap with a spatula to evenly disperse the sample on the slide, let dry
      - ii. Smear a small amount of petroleum jelly onto the slide, evenly disperse the sample onto the jelly
  5. In the control pane under the Instrument Settings tab adjust the following parameters by double clicking on them. This is to collect a standard polycrystalline pattern on a glass slide (for other types of samples/sample mounts please contact the AMSEC lab manager):
    - a. Chi, Phi and Omega offset = 0.000 mm
    - b. Z-height = See computer monitor
    - c. X-ray generator Tension = 45 kV
    - d. X-ray generator Current = 40 mA
  6. If you already have a program, skip to the next step. Under File select New Program → Absolute Scan
    - a. Under Experiment Type select 2Theta-Omega → set offset=0.00, fill in the necessary parameters on the right side of the dialog box, some example parameters are given here for reference:
      - i. Start angle = 20.000°
      - ii. End angle = 80.000°
      - iii. Step size = 0.0200°
      - iv. Time per step = 5.0 s
    - b. Save the program with a descriptive file name to the proper folder under C:\Data\Your Folder
  7. Under Measure select Program
    - a. In the dialog box that appears select browse and locate the appropriate program
    - b. A second dialog box will appear, click the Folder icon or enter in a file path to auto save the data (C:\XRD Data\Research Group\Your File)
    - c. Enter any comments for the sample
  8. Make sure the doors are fully closed and then click "Ok", the diffractometer will automatically start the scan
- Once the scan is completed adjust the X-ray generator current down to 20 mA and carefully remove your sample, clean the glass slide used to mount the sample
9. Under Instrument select Disconnect
    - a. In the dialog box that appears select "Disconnect and close instrument control window"
    - b. Click "Ok"
  10. Log out of FOM
    - a. Take any container with remaining sample with you from the AMSEC lab or it will be disposed of

### Powder Sample Alignment S.O.P.

1. Ensure the X-ray shutter is closed and make sure the XRD is safe to open
2. Get the 0.1 Cu beam attenuator from the cabinet under the sample prep station (in the box labeled "Panalytical Attenuators and Mirror Masks")
3. Insert it on the incident side (right side or X-ray source side) of the XRD with the label facing towards the sample stage. **FAILURE TO INSERT THE Cu ATTENUATOR WILL RESULT IN DAMAGE TO THE DETECTOR.**



4. Check that the diffracted side (left side) optics are correct (refer to the laminated sheet)
5. Insert a blank microscope slide into the sample holder, shut the XRD doors (carefully)
6. Set the Z-height, 2-theta, and omega to zero (0)
7. Perform height alignment scans (all located under Measure, Program)
  - a. Select and run the **2-theta alignment** program
    - i. Locate the center of the peak that resulted from the scan, move the 2-theta position to the center of the peak (or peak maximum)
  - b. Select and run the **z-alignment** program
    - i. Locate the height position where the intensity is ~50% of maximum achieved counts, move the z-height to that position
  - c. Select and run the **omega alignment** program
    - i. Locate the center of the peak that resulted from the scan, move the omega position to the center of the peak (or peak maximum)
  - d. Select and run the **z-alignment** program
    - i. Locate the height position where the intensity is ~50% of maximum achieved counts, move the z-height to that position
  - e. Under User Settings, select Fine Calibration Offsets
    - i. In the dialog box that appears select "set new = 0"
8. Remove the Cu attenuator and replace it back in the box labeled "Panalytical Attenuators and Mirror Masks."

## Instrument Hazards and Best Practices: XRD

This document will cover the inherent hazards when utilizing this piece of equipment as well as the best practices and procedures to avoid danger. These hazards will not include basic things that may be included in the basic safety training document that each user has attested to have reviewed at [fom.wwu.edu/documents](http://fom.wwu.edu/documents).

**Lab coats are to be provided by the user unless special hazards exist in which case they are located at the PPE station.**

### Hazards:

- Chemical exposure
- X-ray exposure

### 1. Required PPE

Appropriate laboratory attire is required at all times in the AMSEC laboratories. Whenever chemicals are being used, an additional requirement of a lab coat is required. Lab coats are to be provided by the user.

Whenever a user is in the AMSEC labs, the minimum requirement for eye protection is wrap around impact glasses. Anytime liquid chemicals are present in the same room as the user without a direct barrier, all users in the lab must wear chemical splash goggles. Splash goggles must be approved by State of Washington Administrative Code (WAC 296-24-078).

If chemicals being used are considered toxic, caustic, corrosive, flammable solvents, carcinogenic, mutagenic, or teratogenic, a minimum of disposable nitrile gloves is required. Avoid chemical transfer by taking off gloves when using anything other than the chemical(s).

### 2. X-ray exposure

The X-ray Diffractometer utilizes X-rays to carry out analyses. At any given time, X-rays are being produced by the XRD. As long as none of the interlocks are defeated, the chances of exposure to X-rays is nearly zero. The system has many different safety devices in place to shut down the X-ray generator if any malfunction occurs. This means that at no time should anyone tamper with or attempt to dismantle or repair any part of the instrument. The only exceptions are changing out the prefix modules for different types of analyses. These procedures are outlined in the standard operating procedures for the XRD.

All users of the XRD are required to participate in the X-ray radiation safety training course on Canvas. No user may have access to the instrument without passing the quiz at the end of the course with an 80% or higher. Contact the lab manager to access the quiz if you have not done so. If you already have access to the instrument but have never taken and/or passed the course, you must do so immediately.