

Operating Procedure for X-Ray Diffraction

Purpose and Scope

This document describes the procedures and policies for using the MSE Bruker D8 Focus XRD. The scope of this document is to establish user procedures. Instrument maintenance and repair are outside the scope of this document.

Responsibilities

This document is maintained by the department Lab manager. The Lab Manager is responsible for general maintenance and for arranging repair when necessary. If you feel that the instrument is in need of repair or is not operating correctly please notify the Lab Manager immediately. The Lab Manager will operate the instruments according to the procedures set down in this document and will provide instruction and training to users within the department. Users are responsible for using the instrument described according to these procedures. These procedures assume that the user has had at least one training session.

Definitions

N/A

Prerequisites

All users must read this document and obtain approval and training from the Lab Manager. The facilities user web page has a link to a video that can provide further training.

Precautions

The XRD uses x-ray radiation. The instrument is designed to prevent radiation exposure.

Procedure

Sample Preparation

XRD can be done on a number of different kinds of samples. The ideal sample will be a crystalline powder that has been pressed into the sample holder, have a smooth surface, and hold in the sample at an angle of 45 degrees. Solid samples, small volumes of sample taped on microscope slide glass or thin films deposited on a substrate can also be used, but will have varying degrees of effectiveness. The more crystalline the sample, the better the results will be.

Operation

Before you start, please observe the following:

Read through this procedure before starting.

Check the status LED's on the lower right hand side of the instrument.

Check the alarm light on the right hand side of the instrument (If it is on, contact Tuesday Kuykendall or Tatyana Galenko immediately).

1. Load your sample:
 - a. Press the green 'door open' button on the right hand side of instrument. If you wait to open the door after you have pressed the button, the doors will relock. Do NOT try to yank the door open. Press the green <open door> button again and proceed.
 - b. Gently pull the handles out toward you and slide the doors open.

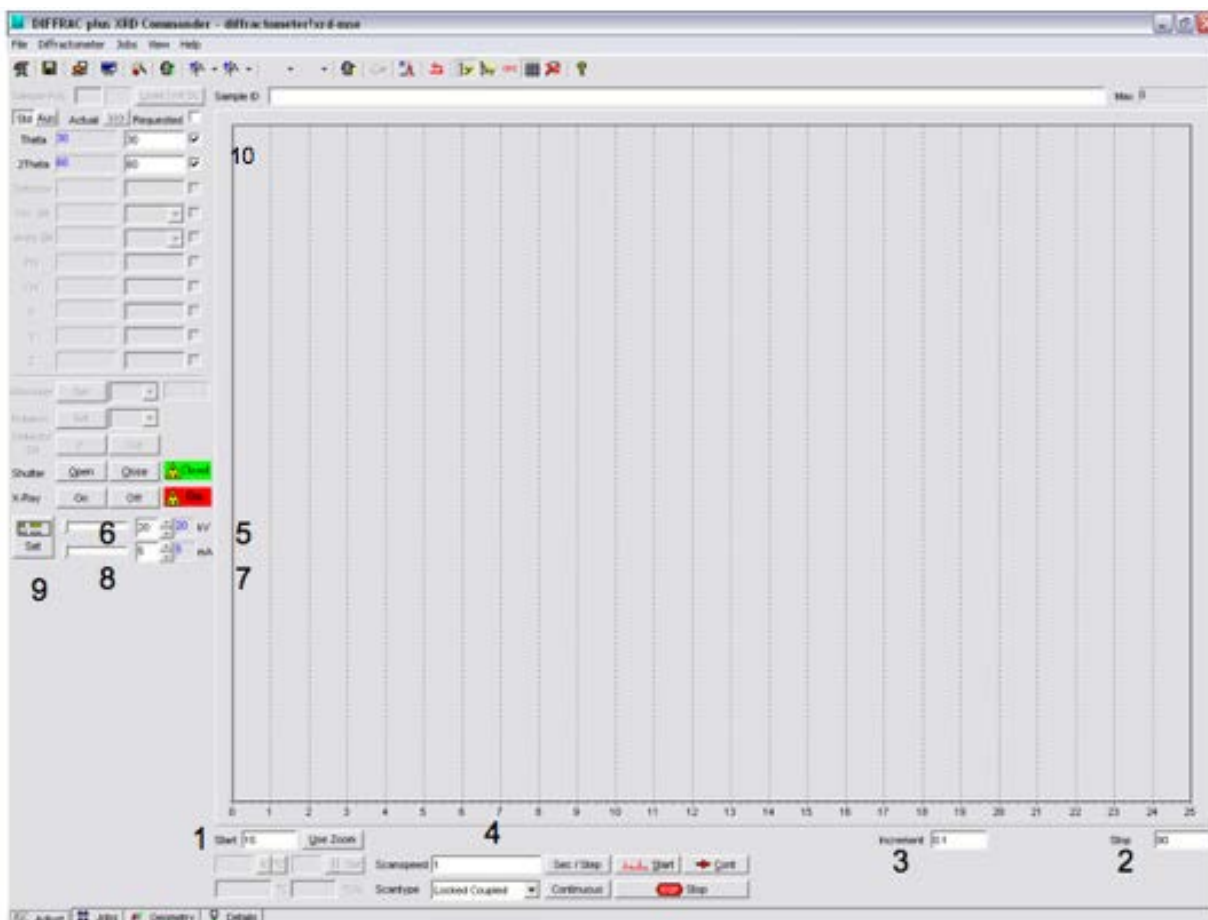
- c. Install the sample by holding it in place with one hand and with the other, press the stage up until it locks. NOTE: If the position of the detector and sample stage are at uncomfortable angles, you may move the drives in XRD commander. (See diagram 1 #10).
 - d. Verify the slits in the 'anti-scattering' and 'detector' positions are what you want. (It would be useful to write down the slit sizes you are using). To remove slits, carefully slide them out of their enclosure. To replace, slide the slit into place with the beveled side down.
 - e. Grip the silver portion of the door handles and carefully slide doors together and gently **but firmly** press handles into interlock. NOTE: if the door is slammed shut or if the handles don't go into the interlocks properly the safety circuit will trigger and shut down the generator. **Please use care when opening and closing the doors.** If the safety circuit is triggered and the generator shuts down the alarm light will be solid. Please contact the Lab Manager immediately if this happens. There are instructions with pictures on the web page.
2. On the computer, expand 'XRD commander' and raise power if necessary as follows:
 - a. Step kV up in increments of 5 - 10kV at 30 sec. between steps until 40kV is reached. Press the <set> button after each change.
 - b. Step mA from 5 – 10, then increments of 5 - 10mA until 40mA is reached. **Do not start a scan before completing this step. ***
 - c. Be sure that the status values and requested values (blue and black types) kV and mA match before starting to ramp up power.*
 - d. On the left-hand side of the window in XRD commander there <shutter> and <x-ray> buttons to open and close the shutter or turn on and off the x-rays. **PLEASE DO NOT PRESS.** The indicator to the right of the buttons give the status of the shutter and x-ray. When the shutter light is green, the shutter is closed and the enclosure doors can be opened.
 3. For a quick scan (not recommended)
 - a. In XRD Commander <adjust> tab at the bottom of the page, input scan parameters, start and end 2 θ * The limits are **3 – 135 - 2 θ**
 - b. Step size (for quick scan 0.5 – 0.1 - 2 θ)
 - c. Step time (typical is 1 sec)
 - d. Before starting scan make sure power values match values in parameter file.
 - e. Turn around and check the status LED's to see if the alarm is flashing or solid...If not:
 - f. Start scan.
 - g. When scan is complete, go to <File>, <Save as> and name file and save. Data is NOT automatically saved in this mode.
 4. For an auto-saved scan:
 - h. In XRD Commander select the jobs tab at the bottom of the page. *
 - i. Select 'create job' icon on the tool bar. (The icon picture is the same as the job tab)
 - j. Fill in the sample ID, parameter file, and raw file fields. * The parameter file should be selected.
 - k. Before starting scan make sure power values match values in parameter file.
 - l. Turn around and check the status LED's to see if the alarm is flashing or solid...If not:
 - m. Start scan.

- n. Observe scan in the adjust tab,
 - o. Data is automatically saved.
 - p. When your scan is completed, remove your sample. Do not yank on, pull on or apply any force or torque to the sample stage pressure unit.
 - q. When you are finished, you can find your data in the 'short-cut to scans' file on the desktop. DO NOT insert a thumb drive (usb) into any of the usb ports on the instrument computer. All data is saved on a shared drive on the JADE computer in the room next door.
 - r. Remember to remove your sample and close the enclosure doors when your scan has completed.
5. To create a permanent parameters file for an auto-saved scan:
1. Open XRD wizard* from the icon at the bottom of the desktop.
 2. Select the quick edit tab.
 3. Fill in the portions with a white background as necessary*.
 4. Select 'OK'.
 5. Select 'file' 'save as' and name your file. It should automatically save in the parameters file.
 6. DO NOT OVERWRITE PRE-MADE PARAMETER FILES. If you want to use wizard to run a scan you must create your own.

Other considerations:

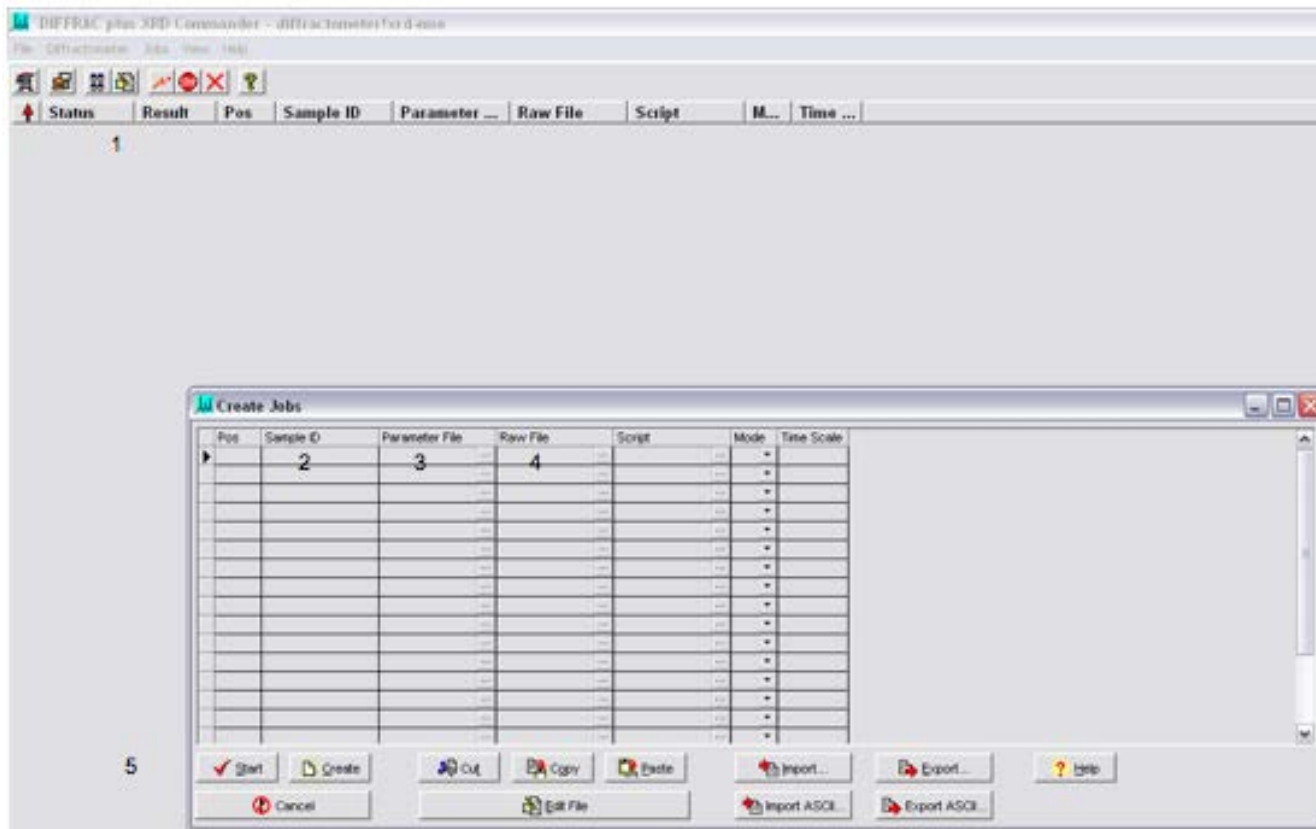
1. When you have finished please remember to remove your sample and close the enclosure doors.
2. Do not store your materials, samples, or sample holders in the XRD room.
3. You are welcome to use the department sample holders provided you do not remove them from the lab and that you clean them when you have finished.
4. Do not close XRD commander. This would prevent the machine from returning to stand-by mode and can reduce the life of the XRD tube.
5. If you sign-up for time on the machine and cannot make it for any reason, please contact the Lab Manager so your name can be removed from the scheduler.
6. Your billed time is the same as your resource scheduled time. This is the time you have the lab.
7. If you need to run a scan that will take more than 2 hours, please notify the Lab Manager and we will arrange for an overnight scan.
8. There are a few users who have graciously agreed to be available to answer questions. They can only answer questions regarding operation of the instrument and hardware. They cannot answer questions pertaining to repair or maintenance. Their contact information is posted on the south outer side of the enclosure.

Below are pictures and labels for the previous operation descriptions where indicated with a *:



1. 2θ start position
2. 2θ end position
3. Increment (step size)
4. Scan speed
5. kV, live display
6. kV, requested
7. mA, live display
8. mA, requested
9. Power 'Set' to change to requested value.
10. To move drives, check off both positions, input values in requested field (30/60 is level with the floor) and select the drive icon -





1. Job Icon
2. Sample ID
3. Parameter File, press button to select
4. Name raw data file
5. Start
6. Adjust tab

1. XRD Wizard shortcut
2. User name
3. Informational only
4. Select scan type
5. Select scan parameters
6. Adjust power
7. Select <OK> and save in the parameters file.

**When you have completed your measurement please make sure to close out the CORAL session!
 Failure to do so may incur extra costs.**

Implementation and Training:

Training consists of a one hour instruction followed in one week by a short test. At the completion of the test, the user will be given the certificate to sign and access to the room.

References:

Attachments: MSE User Agreement

Documentation:

All XRD documentation is located in the XRD room in the cupboard above the computer.