

XRD Safety and General User Responsibilities

CAUTION: X-ray Generating Equipment!!

The Bruker D8 ADVANCE XRD uses a high voltage power supply to generate x-ray radiation. Before using the Bruker D8 ADVANCE XRD, all users must be certified by the Radiation Safety Officer (Daniel Harlan, 541-737-7082, daniel.harlan@oregonstate.edu) in x-ray safety. In addition, training on the use of this equipment by one of the instrument [principle investigators or the XRD manager](#) must be administered prior to authorized use of the equipment.

In case of an accident or emergency contact emergency services or OSU Campus Police at:

911 or 541-737-7000

Emergency Instrument Shutdown

NOTICE: In the event of an instrument malfunction, fire, earthquake, or other emergency the Bruker D8 can be shut down rapidly using the Emergency Instrument Shutdown procedure below. Do not use this procedure for routine shut down of the instrument.

1. Hit the **red *Emergency Stop* button** on either rounded front corner of the D8 cabinet. X-Rays are no longer being generated, but the storage capacitors in the generator will continue to hold electrical charge for >15 minutes.
2. Turn off power to other parts of the instrument by depressing the **red "0" button** on the rounded right-hand corner of the D8 cabinet.
3. **Call** the XRD Manager (contact info bellow), leave message ***and*** send email

XRD Managers contact information

Name:

Name: Austin Fox

Phone:

Phone:

Email:

Email: foxa@onid.orst.edu

Bruker D8 θ - 2θ Standard Operating Procedure (SOP)**Basic Parameters**

Geo	Start	Stop	Step	Time	Detector	Det Slits	Detector Optics	Det Filters	Tube Optics	Tube Slits
θ - θ	10°	90°	0.03°	1.7s/step 2deg/min	Lynx eye	8mm	2.5 Soller	Ni	Göbel Mirror	0.6mm, Snout
θ - θ	10°	90°	0.05°	1.7s/step 5deg/min	Lynx eye	8mm	2.5 Soller	Ni	Göbel Mirror	0.6mm, Snout

Running the D8 Instrument

1. Log into user account
2. Open up "XRD Commander" (opens data collection software—see Figure 1)

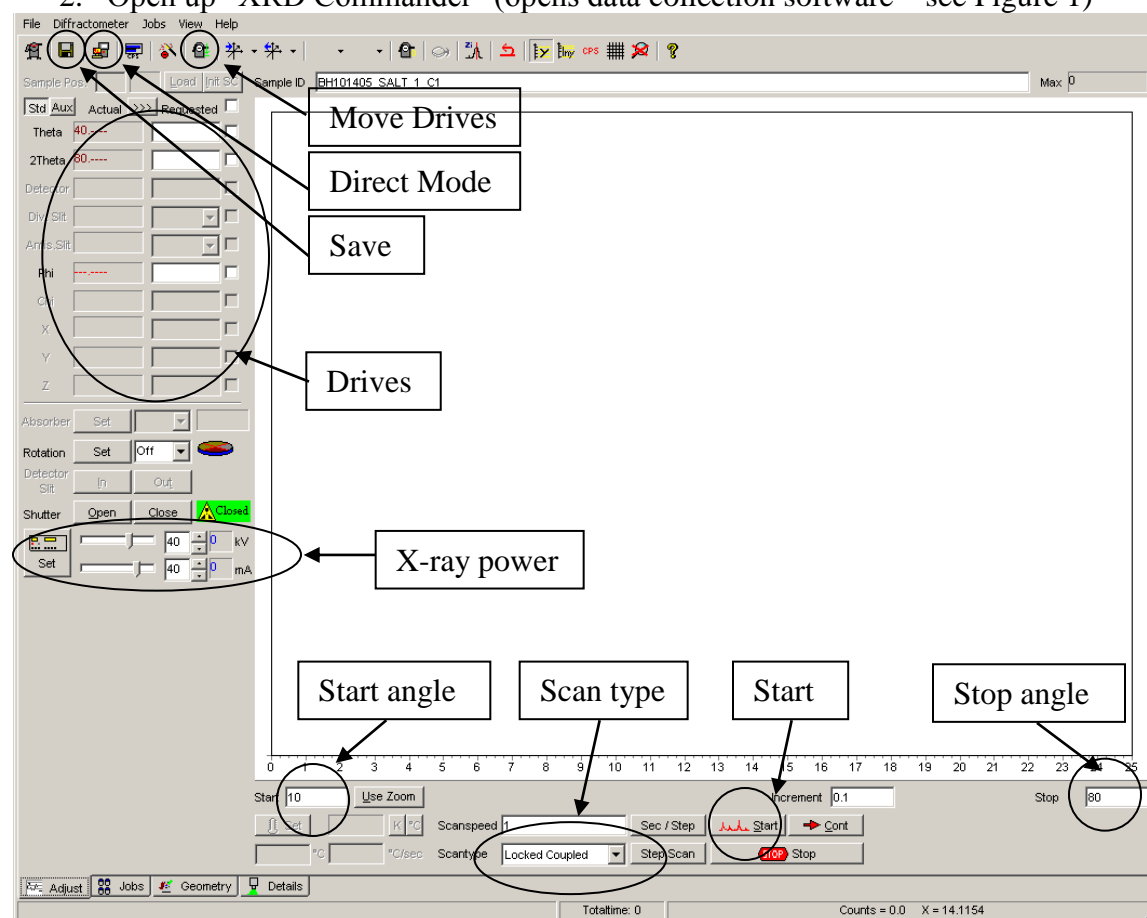


Figure 1. XRD Commander. Circled items referred to in SOP.

3. Ramp up X-ray generator power (at start up power = 20 kV, 5 mA)
 - a. Enter 40 kV, 40 mA, hit "set" (Pull sliders all the way to the right)
4. Choose scan type "locked coupled" (Figure 1, circled)

5. Load your sample
 - a. Push DOOR OPEN button (**Square Green** button on right corner button panel)
 - b. CAREFULLY Open glass doors to the D8 cabinet and LOAD YOUR SAMPLE.
 - c. Close doors gently (should hear a "clunk" as doors lock)
6. Align Sample
 - a. Turn on laser
 - i. Hit "Direct Mode" (Figure 1, circled)
 - ii. Enter OC16,1
 - iii. Hit "Send Command"
 - iv. Hit "Close"
 - b. Start "Video" program
 - c. Adjust Z drive to bring laser to center of video
 - i. Enter new Z in "Drives" (Figure 1, circled)
 - ii. Make sure Z drive checkbox is selected
 - iii. Hit "Move Drives" (Figure 1, circled)

NOTE: There is another button that looks very similar to the "Move Drives" button. "Move Drives" is green and to the left. If you hit the other one you will need to call the XRD manager.

7. Enter Scan Parameters
 - a. Enter the START and STOP angles and the increments in the appropriate fields
8. Start Scan
 - a. Click START [This will run a single scan of your sample]
9. Save your data
 - a. Hit "Save" (Figure 1, circled)
 - b. Save as "Raw" and "Txt"

NOTE: XRD Commander does not auto-save. Please make sure to save your data.

Normal Instrument Shutdown

10. Save your data
 - a. Hit "Save" (Figure 1, circled)
 - b. Save as "Raw" (for EVA viewing) and then repeat save as "Txt"
 - a. Ramp down X-ray generator power
 - b. Enter 20 kV, 5 mA, hit "set" (Pull sliders all the way to the left)
11. Turn off laser
 - a. Hit "Direct Mode" (Figure 1, circled)
 - b. Enter OC16,0
 - c. Hit "Send Command"
 - d. Hit "Close"
12. Close "XRD Commander"
 - a. Windows error message may appear, hit "Do not send"
13. Log off user account
14. Remove your samples
15. Close X-ray doors