Bruker-AXS D8 DISCOVER Diffractometer

Before you analyze your sample, you must mount it in the appropriate sample holder. Several sample holders are available in the X-ray lab. Choose the one that meets your needs and then place the sample holder in the appropriate sample stage on the goniometer.

Before opening the D8 door, check for any abnormalities in and around the instrument. Give particular attention to the shutter lights on the X-ray tube. The **green light** indicates that the tube is energized (light should be lit). The two **red lights** indicate the **shutter position** (if they are lit the shutter is open and you must not open the doors on the D8; if the red lights are off, then it is safe to open the doors and mount the sample stage on the goniometer).

System Control Buttons

The control panels of the D8 are located at the front side of the system.

Stop Button

If hit, it switches off the control electronics and high-voltage generator immediately. The X-ray source is turned off, and all moving drives will stop instantly.

Turn On/Off Illumination

This button turns the fluorescent lights located in the D8 enclosure on or off.

Turn On/Off Fan

This button turns the fan on top of the D8 enclosure on or off.

Open Door Button

Under normal operating conditions the door handles are locked by a mechanical shutter. To open the front door(s) this button must be pressed. Then the front door(s) can be opened. If the Open Door button is activated while the tube window is open, the X-ray shutter will close automatically. After closing the front doors, the X-ray shutter can be opened again.

XRD Commander is the main measurement program in the DIFFRAC suite. It can be used to perform immediate measurements to get a quick overview of a sample or to align the diffractometer, as well as to perform batch measurements.



The **XRD** Commander window is separated into up to five areas:

Drive Control

This area shows all configured goniometer drives. For each drive, its actual position is shown in blue. If any of the drive positions are displayed in dark red, you will not be able to use the D8 — Ask for help. The checkmarks at each drive control the operation of the drives. Only drives that are checked will be affected by these commands. To control all drives, use the checkmark at the top of the Requested area.



Diffractometer Components Control

This area is used to control various components of the D8 for standard runs. These controls should remain as they are set. If you need to **change any** of these controls please **ask for help.**

Scan Control

In this area, the parameters for immediate measurements can be set.

Scantype

Choose the scantype from the dropdown list — Z (Z scan) or Locked Coupled.

A Z scan is used to position a sample at the correct height on the Z drive. A Z scan should be performed for each sample before the XRD measurement — if you do not know how to make a Z scan, please **ask for help**.

A Locked Coupled scan is used for standard XRD scans of any 2Φ values.

Start, Stop, and Increment

Enter the start 2Φ (a value $\ge 2 2\Phi$). If you start a scan of $< 2 2\Phi$, please **ask for help.** Enter the stop 2Φ (a value $\le 140 2\Phi$). If you need to go beyond a stop value of 140 2Φ , **ask for help.** Enter the increment value or stepsize for the scan.

Scanspeed

Enter the scan speed. Use the push button to the right of the input field to switch between <u>seconds per step</u> and <u>degrees per minute</u>.

Continuous/Stepscan

Press the button to change between continuous or stepscan mode. The actual setting will be displayed on the button.

Start, Stop, Cont

These buttons are used to begin or end an immediate measurement. The Cont button will continue an interrupted scan.



Chart Area

The chart area shows the current scan data.

<u>Z Scan Setup</u>

In order to find the Z drive zero position for your sample,

- In the Drive Control area set all drives to zero and place checks in all drive positions.
- Set the *scantype* to Z.
- Place a copper filter in the front detector slit housing.
- Run the scan.
- Using the mouse, find the midpoint of the Y axis intensity (400,000/2=200000), double left click, and the correct Z value will be placed in the Z drive control box.
- Remove the copper filter from the detector slit housing.







XRD measurement

- Set the *start*, *stop* 2Φ angles and the increment value.
- Set the *Scanspeed*.
- The continuous button should be displayed.
- Press Start to begin the measurement.



After the scan is complete

Using $Menubar \rightarrow File Menu \rightarrow Save As$, the measured data can be saved as a RAW data file to your folder on the hard drive. XRD Commander displays the Save As dialog box, and you must enter the RAW filename. The filename can be anything you wish; however, the Sample ID that was used in the Chart Area box does not carry over to the Save As box. The RAW data file can later be examined (e. g., with EVA).

If you click the right mouse button inside the Chart area, a popup menu will appear offering the following options:

- Chart Line Color
- Chart Background Color
- Zoom Reset
- Grid
- Linear
- Logarithmic
- X Labels
- Y Labels
- **Apply Ident** (applies the sample identification on the top axis)
- Print Chart
- Remove Peakstick
- Dots (shows the scan data as little dots instead of connected lines)
- Advanced (shows advanced customized options for the chart area such as Save the chart data as a Text, XML, HTML, or Excel file).

Export \rightarrow Data \rightarrow Excel \rightarrow Save As to your folder on the hard drive.





🛄 DIFFRAC plus XRD Commander - difl	fractometerluky	_ B ×			
File Diffractometer Jobs View Help					
¶ ■ ፼ ₩ · 	· \$+ • - • • 1				
Sample Pos. Load Init SC	Sample ID K9-1Q	Max 3920			
Std Aux Actual >>> Requested	K9-1Q				
	4300				
21heta p0 12.7	4200-				
Detector 15 6.35	4000				
Div. Siit 0.49 0.49 ▼ I✓	3800				
Antis.Sit 0.49	3700				
Phi U 0 V	3500				
chi p p M	3400				
	3200				
	3100 3000 Milling Chart	? X			
	2900 Chart Series Data Export Print				
Absorber Set	2800 2700 Series General Axis Lates Legend Panel Paging Walls	3D			
Rotation Set Off 🖵 🥌	2600				
Detector In Out	2400 Measurement	•			
Shutter Open Close	2300- 2300- Add				
X-Ray On Off A On	2100 Pulse Height Analysis Line				
	2000-				
Set t 40	1800				
	1700- 1600-				
	1500 Change				
	1400				
	1200 Help Close				
	900				
	700				
	600 500				
	400				
	300 200				
	100	Company - Compan			
	Start 2 Use Zoom	Increment 0.02 Stop 30			
	²⁰ K [™] C Set Scanspeed 2 Sec / Step Jul. Start → Cont				
30 ¹ °C 5 ¹ °Cleec Scentype Locked Coupled ▼ Step Scen <u>002</u> Stop					
🗠 Adjust 🗱 Jobs 🖉 Geometry 🖳 Detais					
A Chart A III yoo Care	Totaltime: 2	CPS = 3571.4 X = 9.6235			
💶 Start 🕼 🛄 XRD Commander		😌 💓 💥 🚫 🌮 1:53 PM			



🛄 OFFRAC plus XRD Commander - diffractometerluky					
	'त''। ' ०		2000		
Semple Pos	Sample ID (K9-1	4	Wax base		
Tube 15 6 35		K9-1Q			
2Thete 30 12.7	4300				
Detector 15	4100				
	4000				
0.43 ▼ ▼	3800				
	3700				
	3500				
	3400 3300				
	3200				
	3100	Hediting Chart			
	2900	Chart Series Data Export Print			
Absorber Set 💌	2700	Picture Native Data			
Rotation Set Off 💌 🥯	2600	Series: (all) Point Index			
Slit In Out	2400	Eermat IV Point Labels			
Shutter Open Close AClosed	2300	C Text IV Header			
X-Ray On Off <u>A</u> On	2100	C HTML Table Delimiter			
	2000	C Excel			
Set	1800				
	1700				
	1500				
	1400	Lopy Save Sgnd			
	1200	Help Close			
	1000				
	900				
	700				
	600- 500-				
	400				
	200				
	100	All set and a set of the second second and a second and a second and a second and a second a second a second a	- Automa - Automa -		
	Start D	lise Zoom	Increment 0.02 Size 20		
	30 K	B Set Scanspeed 2 Sec / Step Aud. Start - Cont	and one in 10.02 stop 150		
30 C 5 "c/sec Scartype Locked Coupled V Step Scan (122) Stop					
N: Adult 22 Jobs 2 Geometry 9 Details					
		Totatime: 2	CPS = 2902.5 X = 8.9433		
🍠 Start 🛄 XRD Commander			🕲 💥 🖓 🌜 1:55 PM		

After you have completed your measurement, minimize XRD Commander. Do not close the program.