

# Bruker D8 Discover X-ray Diffraction System



X-ray diffraction (XRD) is a non-destructive technique for analyzing the structure of materials, primarily at the atomic or molecular level. It works best for materials that are crystalline or partially crystalline (i.e., that have periodic structural order) but is also used to study non-crystalline materials. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced. By convention, the angle between the incoming and outgoing beam directions is called  $2\theta$ . In a sample, consisting of sheets of charge separated by a distance  $d$ , constructive interference (greater scattered intensity) is observed when Bragg's Law is satisfied:

$$n \lambda = 2 d \sin \theta$$

where  $n$  is an integer (1, 2, 3, ...),  $\lambda$  is the wavelength of the x-ray beam, and  $\theta$  is half the scattering angle  $2\theta$  shown above. After passing the NCF safety exam, users can request training on this machine by through I-Lab services. **Maximum reservation time is 4 hours.** Those not trained can request an NCF work service order by contacting the lab director.

**Location: Packaging bay**

**Training: 3 sessions (2 trainings and a checkout session)**

## Technical Specifications:

- Copper x-ray tube with  $\text{CuK}\alpha$  radiation = 1.5418Å
- LYNXEYE 'compound silicon strip' detector for fast X-ray diffraction measurements. Resolution - 0.008°
- High-res detector designed for ultra-high resolution -  $\sim 0.0035^\circ$ - measurement of single crystals and thin films.
- Nickel foil filters are available for removing  $\text{K}\beta$  radiation.
- Copper absorbers (0.2/0.2 and 0.05 mm thick) are available to remove high-energy photons due to their k-absorption-edge.
- Copper slits are available for make the beam parallel.

## Important:

- ***Users must pass the “radiation safety training” from UIC’s Environmental Health and Safety Office before getting access to the XRD instrument.***
- ***Do not open the glass door when the X-ray shutter is open.***
- ***Chillers needs to be on for at least 20 mins after using the XRD.***

## System Startup

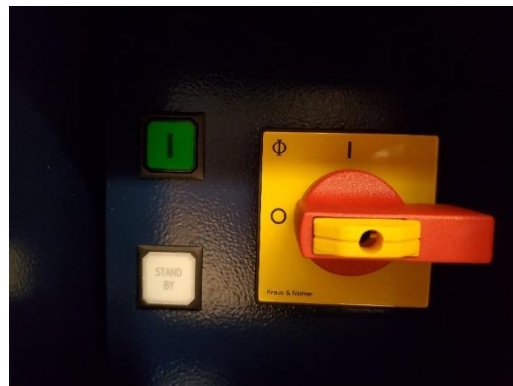
1. Put the “XRD” tag on the right HX300 chiller in the hallway. Flip the main switch to turn it on and wait for the display to stabilize, then press the **Start/Stop** button.



- Turn on the chiller behind XRD computer by flipping switch to the **ON** position.



- Push the green **power button** on left side of the machine.



- After a few moments, push the **top button** on the front of machine and wait for the system to progress through its processes.



Note that if the system has not been used in a few days, it will go through the tube conditioning step which can take up to an hour. Once the top button shows generator stable, the system is ready for use.

**Generator Button Display**

	System booting		Safety alarm
	Generator ready		Generator error
	Tube heater on		Water cooling error
	Generator ramping		Tube conditioning
	Generator stable		

**Enclosure Display**

	(blinking) System booting		Sample changer error
	(steady) System ready		Detector error
	Computer connected		Drive collision
	Measurement running		Drive error
	Door open		

**Enclosure Lights button**

**Enclosure Doors button**

**Emergency Stop buttons**

**Power ON button**

**Mains Disconnect switch**

**Standby button**

## Startup

1. Check that neither of the large red **Emergency Stop** buttons are pressed.
2. Turn the Mains Disconnect switch from to .
3. Press the Power ON button .
4. Wait for to appear on the Generator button display.
5. Press the Generator button to turn on X-rays.

## Shutdown

1. Check that measurements are complete.
2. Press the Generator button to turn off the generator.
3. Press the Standby button .
4. Turn the Mains Disconnect switch from to .

**D8 ADVANCE**  
**Serial Number:** 210066

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**BRUKER**

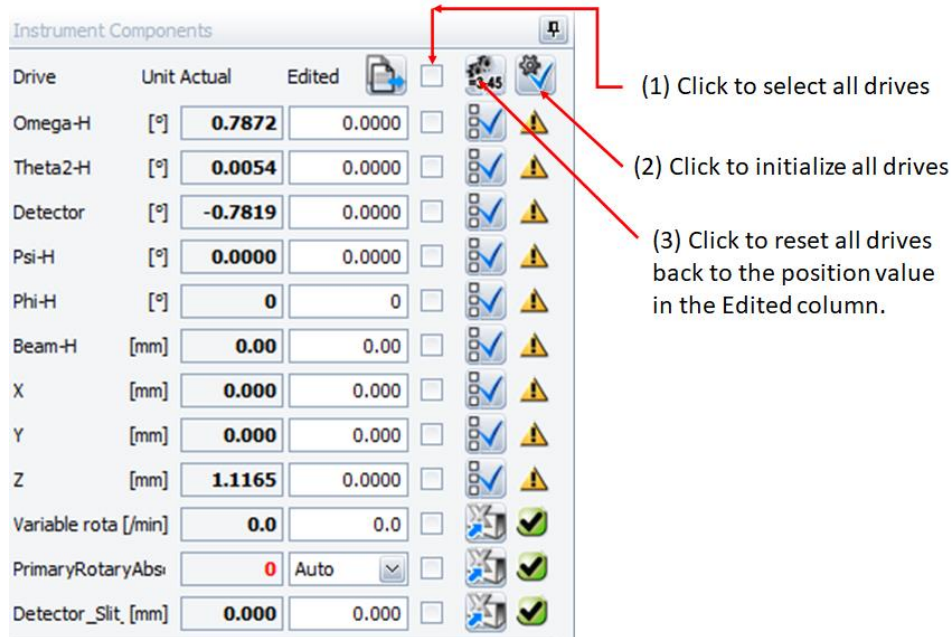
### Operation

5. Place the desired sample holder on the stage. A detailed description on how to change the sample holder is covered in the Appendix section, *Changing Sample Holder*.

6. Open the Diffrac suite software . You will be prompted for a login. Chose the **Lab Manager** account and click **Ok**. There is no password.



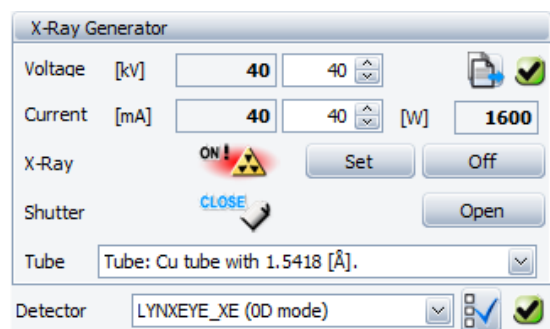
7. Once the software opens, click on the **Commander** tab on the left-hand side of the screen. You can also click the same tab at the top of the window. Follow the steps in the image below.



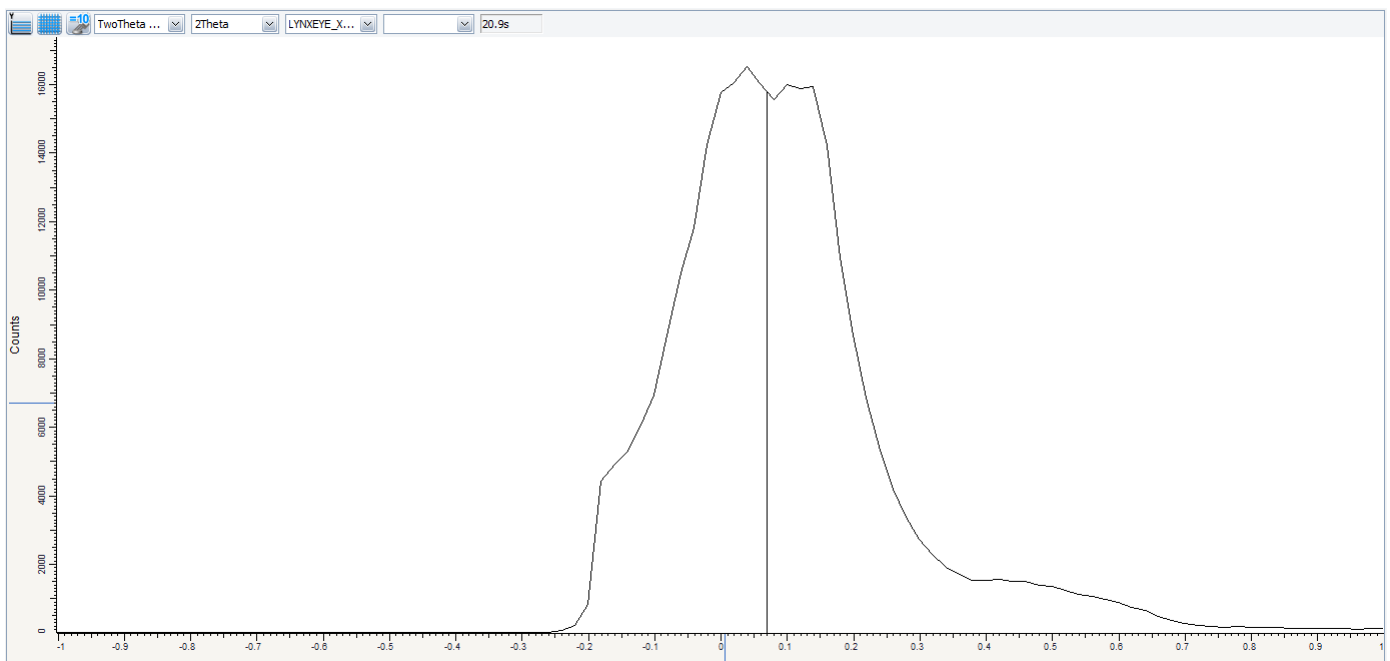
8. Click on **File** → **Application** → **Powder Diffraction** for powder and bulk samples. Click on **High Resolution** for thin film samples.



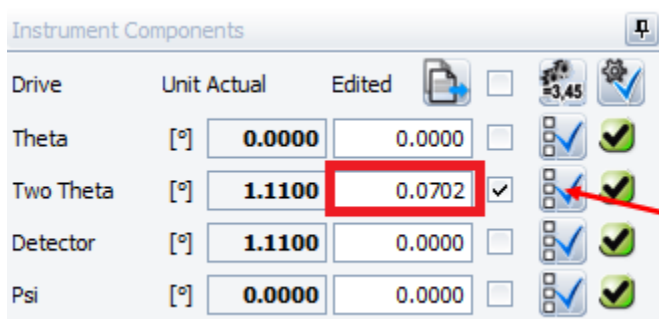
9. In the **X-ray Generator** section, increase the voltage and current to your desired values. 1600 Watts is a good place to start. The maximum values are 50 KV and 60 mA respectively. Click on **Set**.



10. In the **Detector** field, choose the one you are using and click the check mark next to it. Usually, this is the OD detector.
11. In the **Instrument Components** section, set **Detector\_Slit** to your desired values. 1.2 mm is a good place to start. The range is 0 → 9.5 mm. Click on **Set**.
12. Change the **Primary Rotary Absorber** from **Auto** to **1** in the drop-down menu. This is to avoid initialization errors. After the first calibration scan, it can be changed to another value.
13. At the bottom of the window, in the **Scan Setup** section, use the drop-down menu to select **Two-Theta**.  
This is to get the offset of the beam with the sample holder in place. The parameters for this can should be
  - a. Scan range → -1 to 1
  - b. Scan mode → Continuous
14. Repeat step 12 with **Primary Rotary Absorber** of **73.88**. Then repeat step 13.
15. You will get a graph like the one below. Bisect the graph by double clicking the apex. Try to make both sides of the graph symmetric.

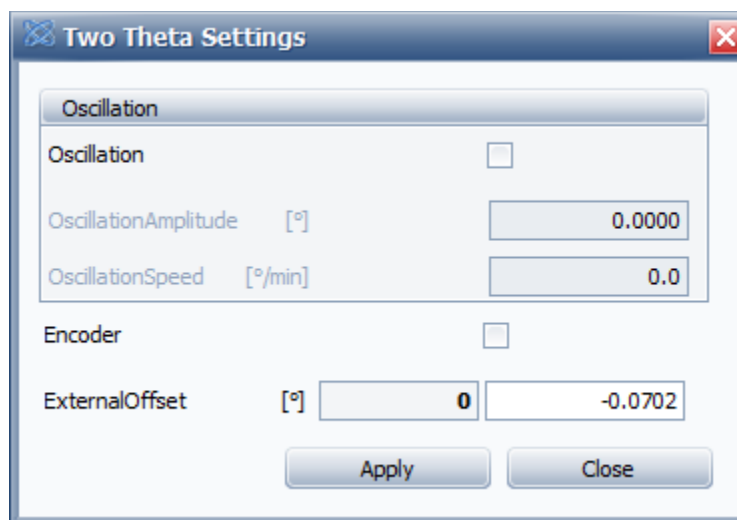


16. A number will appear in the **Two Theta** field. Copy the number and paste the opposite in **ExternalOffset** field of **Two Theta Settings**. Click **Apply** and close the pop-up window.



Drive	Unit	Actual	Edited	Icons
Theta	[°]	0.0000	0.0000	✓, ✓
Two Theta	[°]	1.1100	0.0702	✓, ✓
Detector	[°]	1.1100	0.0000	✓, ✓
Psi	[°]	0.0000	0.0000	✓, ✓

Click for  
Additional Settings



**Two Theta Settings**

Oscillation

Oscillation

OscillationAmplitude [°] 0.0000

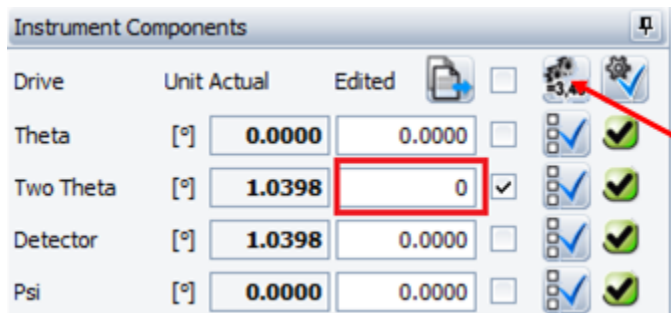
OscillationSpeed [°/min] 0.0

Encoder

ExternalOffset [°] 0 -0.0702

Apply Close

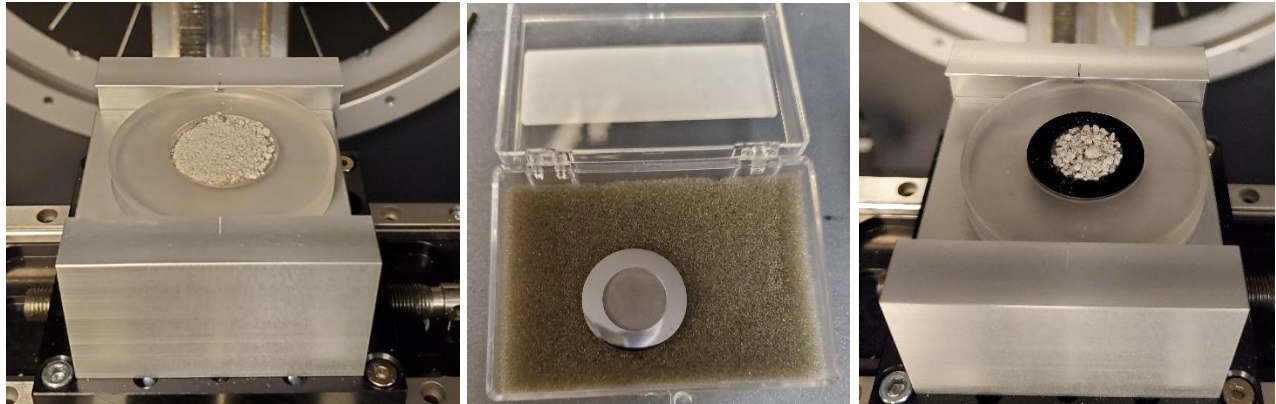
17. Delete the original value in the Two Theta field. Put zero for the new value and then click on icon to reset the drive to zero. The offset still remains in the background to ensure the stage is not in the pathway of the beam.



Drive	Unit	Actual	Edited	Icons
Theta	[°]	0.0000	0.0000	✓, ✓
Two Theta	[°]	1.0398	0	✓, ✓
Detector	[°]	1.0398	0.0000	✓, ✓
Psi	[°]	0.0000	0.0000	✓, ✓

Click to reset drive  
back to the 0

18. Place your sample on the stage. An example with the spring-loaded holder and a powdered sample is shown. If you do not have a lot of powder material, you can use the smaller circular holder as shown.

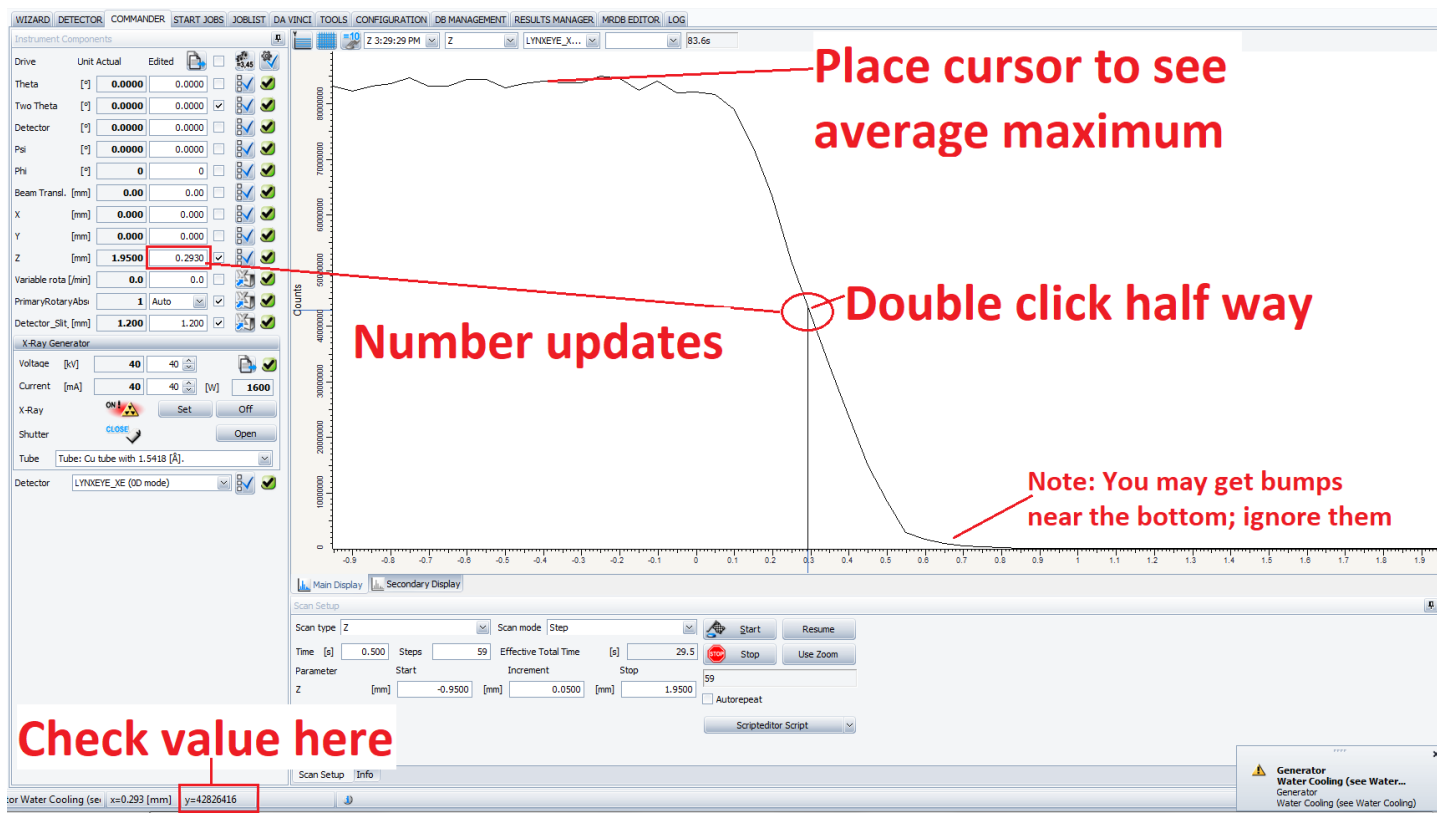


19. Change the scan type to **Z** in the **Scan Setup**. Change the **Primary Rotary Absorber** to **Auto**. Perform the scan with the following parameters and click **Start**.
- Scan range → minimum – 0.9958 to maximum 1.998. If you exceed this range, you will get an error and you will have to reduce the range.
  - Scan mode → Step
  - Increment: anywhere from 0.02 to 0.05. Typically, the scan should be about 30 seconds to 1 minute.

Scan type	Z	Scan mode	Step		Start		Resume			
Time [s]	0.500	Steps	60	Effective Total Time [s]	30		Stop		Use Zoom	
Parameter	Start	Increment	Stop							
Z	[mm] -0.9985	[mm] 0.050	[mm] 1.9515	<input type="checkbox"/> Autorepeat						
										Scripteditor Script

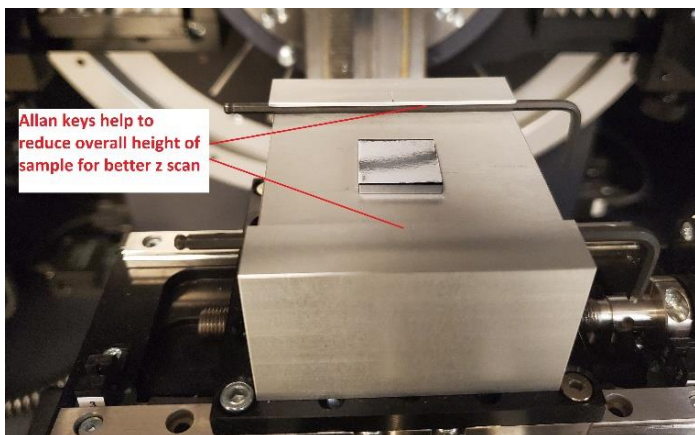


20. You should get a graph similar to the one below. If not, refer to step 22. The point at which a non-zero slope appears is where the beam has met the edge of your sample.



21. Use the mouse to find the average of the flat portion of the graph. A field showing the value updates at the bottom to the Diffrac Suite window. Divide the average by half and find the new value along the x-axis, then trace this value to the slope of the graph. Double click this point on the slope. A **Z** value should show up in the **Z** field of **Instrument Components**.

22. If the z-scan does not reveal your sample edge, you can place Allan keys on the edges of the holder to push down the overall height of the sample and holder. Be sure to use Allan keys that are of the same thickness so as to keep a level height.

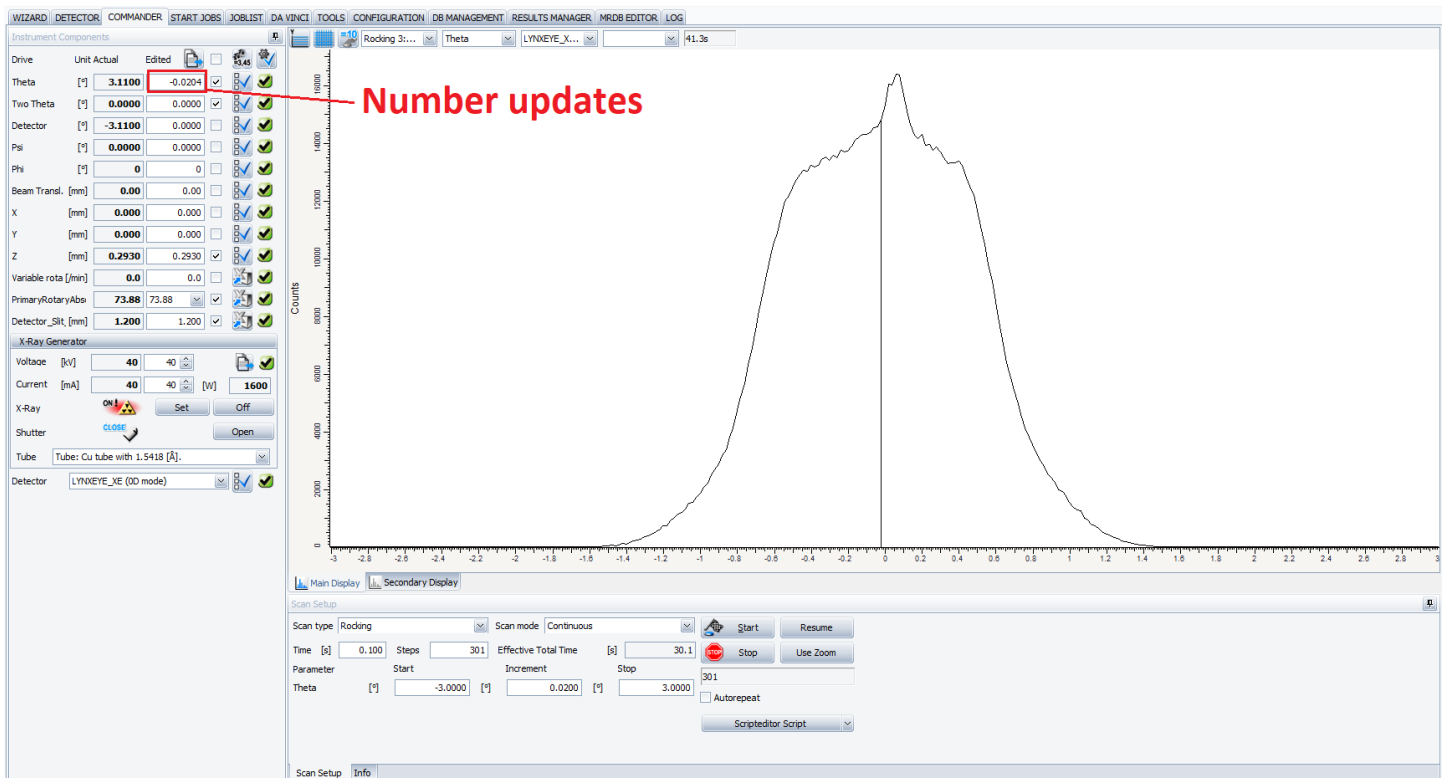


Note: If your **Z** scan that has extra bumps along the slope, it could be b/c of the size of your sample is small. Consider the 1<sup>st</sup> Z for your calculations.

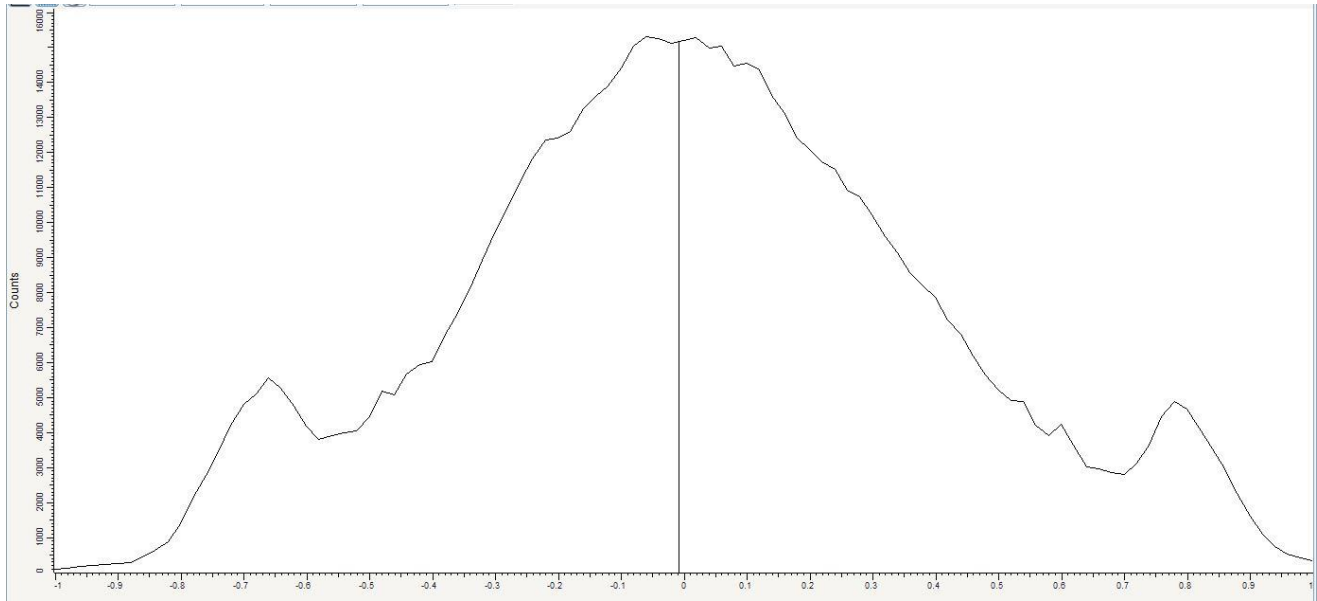
23. Change the scan type to **Rocking** in the **Scan Setup**. Change the **Primary Rotary Absorber** to **73.88** or **5998**. Perform the scan with the following parameters and click **Start**.

- a. Scan range → minimum -3.0 to maximum 3.0. If you exceed this range, you will get an error and you will have to reduce the range.
- b. Increment: anywhere from 0.02 to 0.05. Typically, the scan should be about 30 seconds to 1 minute

24. You should get a graph somewhat similar to the one below. Bisect the graph by double clicking the apex. Try to make the both sides of the graph symmetric. A number will appear in the **Theta** field **Instrument Components**. This finds the angular offset of your sample on the stage.



Note: You could also get something that looks like the one below. Proceed with the graph.



Note: For some bulk samples, you might see a graph that looks almost like a straight line. Proceed with the same steps.

25. Now repeat the **Z** scan with **Primary Rotary Absorber** to **Auto**. This is to see if the z graph has changed in intensity or shape. Higher intensity and/or steeper slope mean a better z scan. Repeat step 21 if the new scan is better.

Note: You can compare the new scan to the old one by click on the locations shown below.

Click to select type of previous measurement



Click to compare current scan with previous measurement

26. If the new Z scan is not good, repeat the **Rocking** and **Z** scans until satisfactory graphs are achieved. You might need to play with the placement of your sample or change the sample holder.

27. Change the scan type to **Coupled TwoTheta/Theta** or to any other scan type you desire in the **Scan Setup**. Change the **Primary Rotary Absorber** to **Auto**. Perform the scan with the following parameters and click **Start**.

Scan Setup

Scan type: Coupled TwoTheta/Theta Scan mode: Continuous

Time [s]: 1.000 Steps: 1001 Effective Total Time [s]: 1001

Parameter	Start	Increment	Stop
2Theta [°]	5.0000	0.0500	55.0000
Theta [°]	2.5000	0.0250	27.5000

Buttons: Start, Resume, Stop, Use Zoom

Autorepeat:


Scripteditor Script

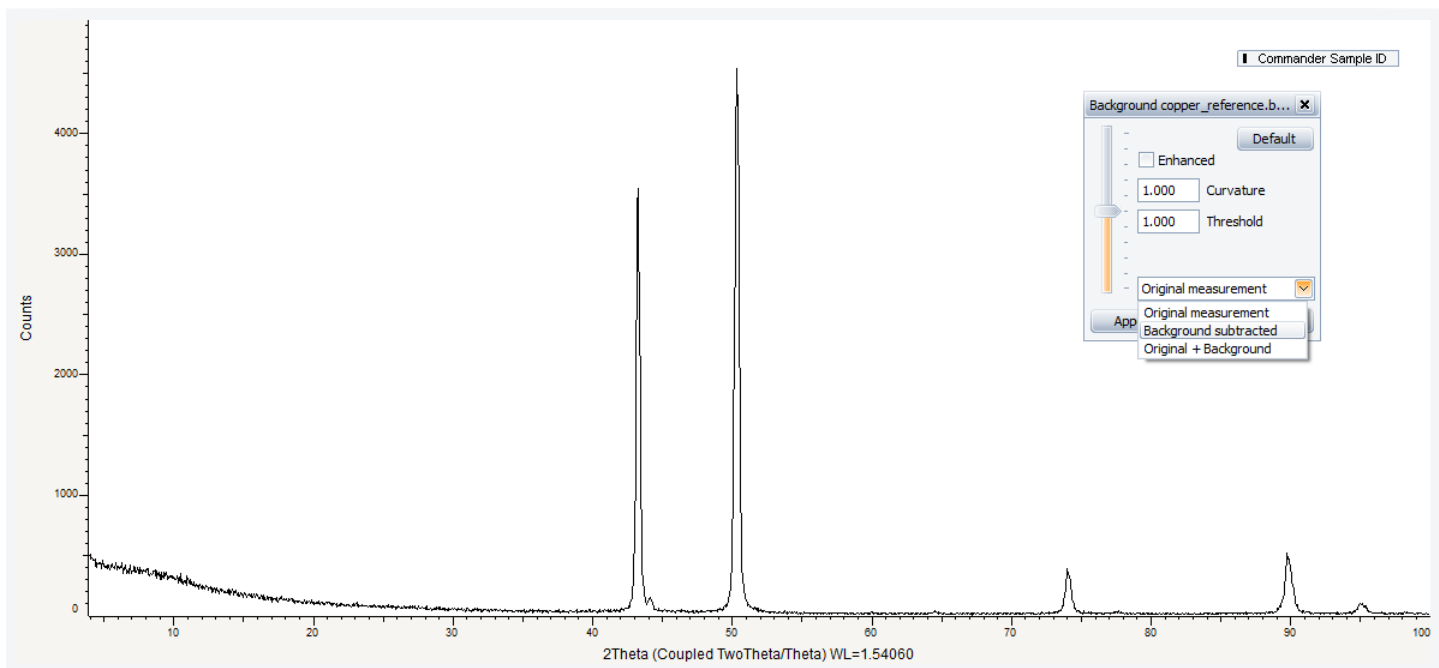
28. To save your data, go to **File** → **Save As**. Save the file with the *.brml* extension.

### Analysis of Data

29. Open up the Diffrac Suite Eva software 



30. Import your file by going to **File** → **Import From File**. You can import multiple files.
31. Remove the background from the data by clicking **Background** in the **Tools** section on the left-hand side of the window. The icon looks as such  **Background**.
32. A window with options will pop-up. From the menu, select the **Background Subtracted** option. This will flatten the data, removing background signals.



33. You can match the data to a scan in the open source Crystallography Open Database. You can match either by scan, name, or database identification number. Click on any one of these in the **Tools** section on the left hand side of the window. The following window will pop up.

Search / Match (scan) copper\_reference.brml #1

Rebuild Chemical    Chemical Filter #1 Database    Database Filter #1

Database COD2016: 352140

Chemical Filter Database Filter Candidate List Selected Candidates

H	<i>D</i>																	He					
Li	Be	29 <b>Cu</b>	Copper														B	C	N	O	F	Ne	
Na	Mg																	Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr						
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe						
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn						
Fr	Ra	Ac																					
Lanthanoids/ Actinoids			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu							
			Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr							

2Th=77.032 Cnt-  
d=1.23696

Discarded At Least One Mandatory Not Checked Reset

34. Select **Chemical Filters** and then choose the elements that compose your sample. Note the colors at the bottom indicating **Discarded**, **At Least One**, **Mandatory**. *Mandatory* means included in filter, while *At Least One* means an at least an isotope is included.
35. Click on **Candidate List**, then click **Search** at the bottom of the window. The software will search the database and produce a list of candidates with confidence values of matching.

Search / Match (scan) copper\_reference.brml #1

Rebuild Chemical  Chemical Filter #1 Database  Database Filter #1

Database COD2016: 352140 - After Filters: 14449

Chemical Filter Database Filter Candidate List Selected Candidates

Index #	%	Source	ID	Quality	Status	I/Icor	Mi... 0	Name	Formula	Crystal System	a	b	c	alpha	beta	gamma	Spacegroup	Z	Volume
<input checked="" type="checkbox"/>	1	77	COD2016	COD 1...	Quality Un...	Status U...	11.51		Cu <sub>0.8</sub> Fe <sub>0.2</sub>	Cubic	...						F m -3 m	4	47.38
<input type="checkbox"/>	2	77	COD2016	COD 1...	Quality Un...	Status U...	11.98		Cu <sub>0.951</sub> Zn <sub>0.049</sub>	Cubic	...						F m -3 m	4	47.39
<input type="checkbox"/>	6	75	COD2016	COD 1...	Quality Un...	Status U...	12.02		Ag <sub>0.04</sub> Cu <sub>3.96</sub>	Cubic	...						F m -3 m	1	47.44
<input type="checkbox"/>	8	76	COD2016	COD 7...	Quality Un...	Status U...	12.73		Cu	Cubic	...						F m -3 m	4	47.32
<input type="checkbox"/>	9	75	COD2016	COD 4...	Quality Un...	Status U...	12.86		Cu	Cubic	...						F m -3 m	4	47.26
<input type="checkbox"/>	3	76	COD2016	COD 9...	Quality Un...	Status U...	11.95	<input checked="" type="checkbox"/>	Co... Cu	Cubic	...						F m -3 m		47.24
<input type="checkbox"/>	4	76	COD2016	COD 9...	Quality Un...	Status U...	11.95	<input checked="" type="checkbox"/>	Co... Cu	Cubic	...						F m -3 m		47.24
<input type="checkbox"/>	5	76	COD2016	COD 5...	Quality Un...	Status U...	11.95	<input checked="" type="checkbox"/>	Co... Cu	Cubic	...						F m -3 m	4	47.24
<input type="checkbox"/>	7	73	COD2016	COD 9...	Quality Un...	Status U...	11.95	<input checked="" type="checkbox"/>	Co... Cu	Cubic	...						F m -3 m		47.16

Group Duplicates

Matched 2916 / 14449 Candidates in 2.9 s.

Search / Match

Whole Range  Subrange

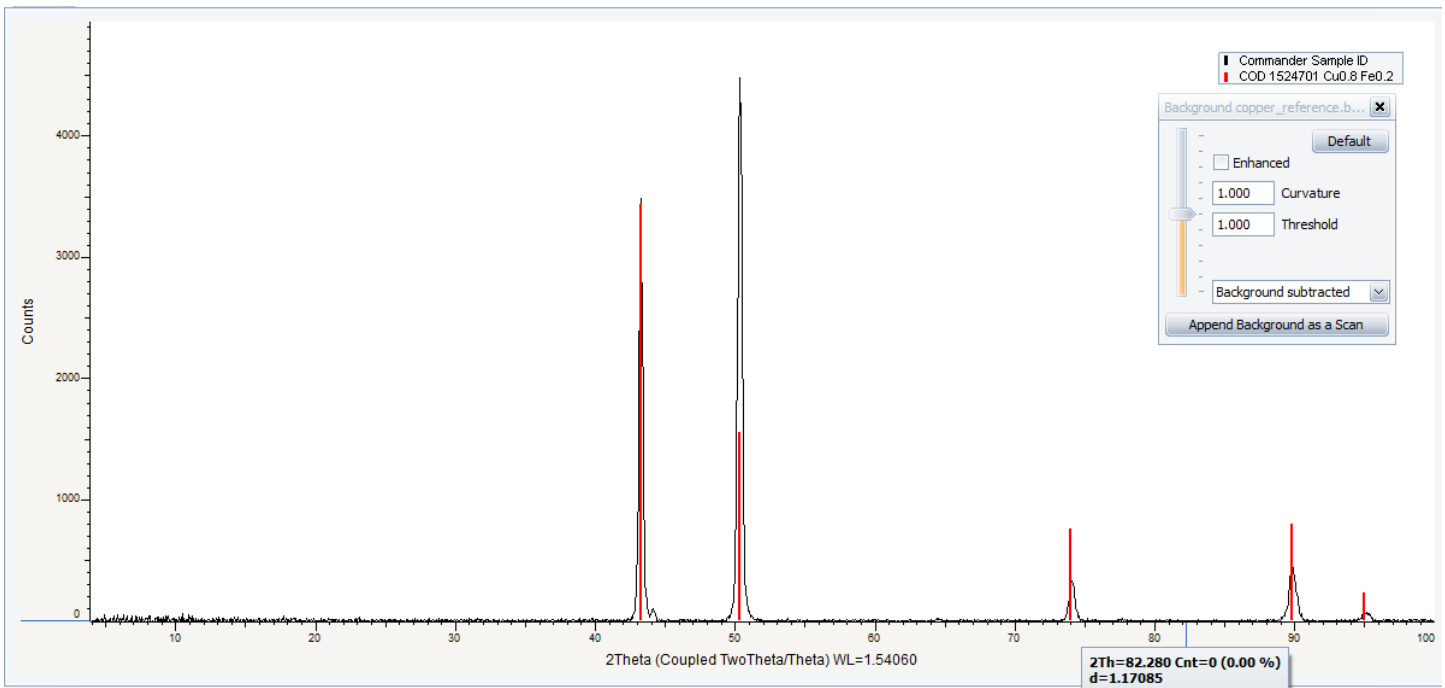
Criterion: 2: Neutral

Auto

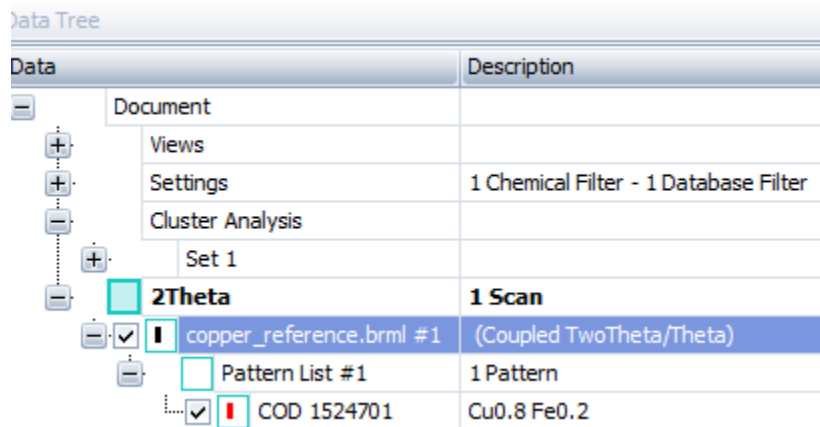
Search

Search / Match Search by Name Filter Lists

36. Select the one that best matches. You can then see the database file overlaid on your data.



37. You can move back and forth between scan data by checking the boxes in the **Data Tree** section. The selected choices will update in the main window.



Data Tree		
Data	Description	
-	Document	
+ -	Views	
+ -	Settings	1 Chemical Filter - 1 Database Filter
-	Cluster Analysis	
+ -	Set 1	
-	<b>2Theta</b>	<b>1 Scan</b>
- <input checked="" type="checkbox"/>	copper_reference.brml #1	(Coupled TwoTheta/Theta)
- <input type="checkbox"/>	Pattern List #1	1 Pattern
- <input checked="" type="checkbox"/>	COD 1524701	Cu <sub>0.8</sub> Fe <sub>0.2</sub>

38. To export data, click on **Export Bg Subtracted Scan** in the **Tools** section on the left-hand side of the window. Save the background subtracted file with whatever file format you wish. The .xy extension is usually used.

#### Shutting down

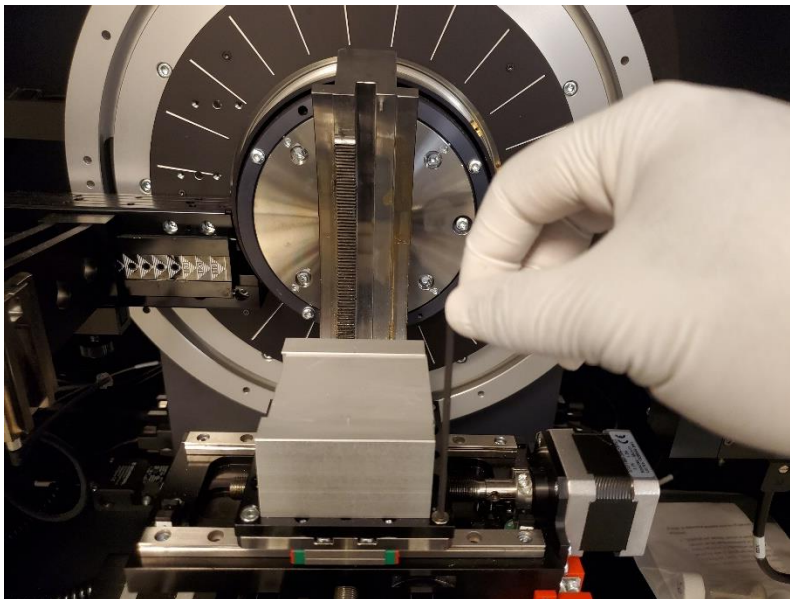
39. Reset generator voltage and current to 20 kV and 5 mA respectively.
40. Reposition all drives back to zero. Close the software.
41. Turn off XRD generator by pressing the top button on the front of the machine as in Step 4.
42. Once the generator is off, press the Standby button on the side of machine.
43. Wait for 30 minutes before turning off the chiller behind the machine.
44. After turning off the smaller chiller, turn off the main chiller in hallway. Press the **Start/Stop** button and wait for the chiller to stop, then flip the main switch to turn it off.

## Changing Sample Holders

There are two types of samples holders that can be used. One is a stationary spring-loaded holder that can be use for holder bulk and powdered samples. The other is a vacuum holder for small and/or thin/fragile samples. The vacuum is not very strong so that the sample is not harmed. There are extra plates that can be placed underneath the holders for special samples.

### Changing Spring-loaded holder

45. Place the holder onto the stage and use an Allan key to tighten the screws.



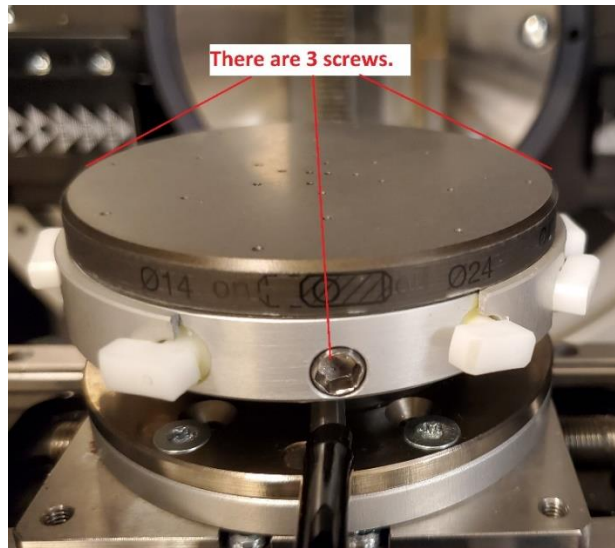
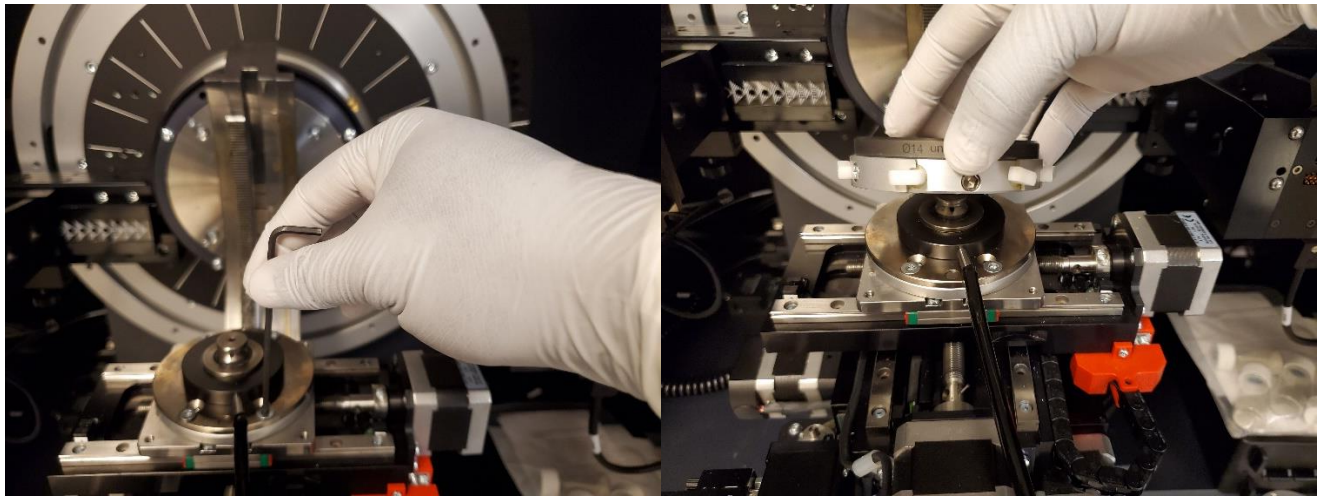
46. You can place you sample directly on top of the holder.

47. To replace the holder, use an Allan key to remove the screws. **Be careful not to lose the screws.**

### Changing Vacuum holder

48. Place the base of the holder onto the stage and use an Allan key to tighten the screws. Place the vacuum holder and push down. Use an Allan key to tighten the vacuum holder.

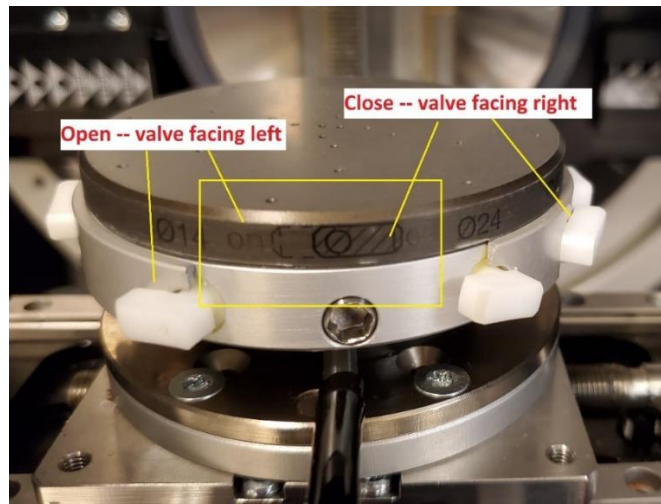




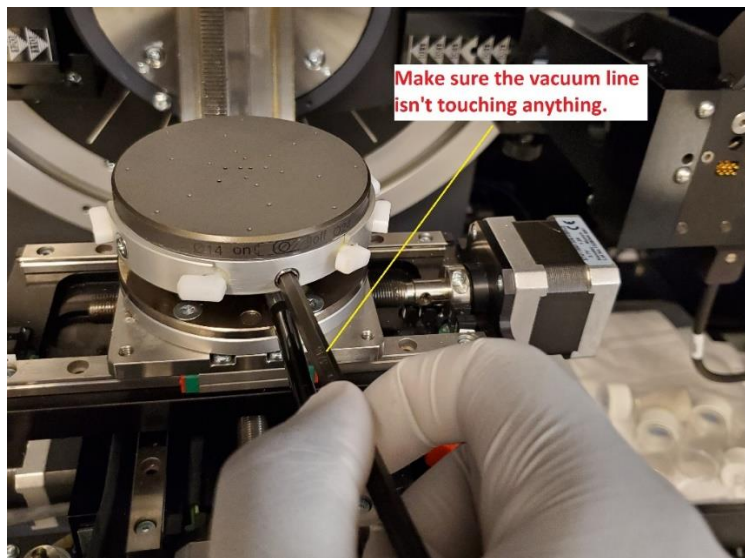
49. Turn on the vacuum pump by flipping the switch on the outlet extension behind the x-ray tube.



50. Open the vacuum hole for your sample by flipping the plastic valves on the side for vacuum holder. The numbers on the holder correspond to diameters of the holes on the holders. Choose the ones to open based on the size of your sample.



51. Make sure the vacuum line is not touching anything. This is to make sure it is not restricted during a scan.



52. To replace the holder, close the open vacuum lines and turn off the vacuum.

53. Loosen the holder and remove the it.

54. Use an Allan key to remove the screws. **Be careful not to lose the screws.**