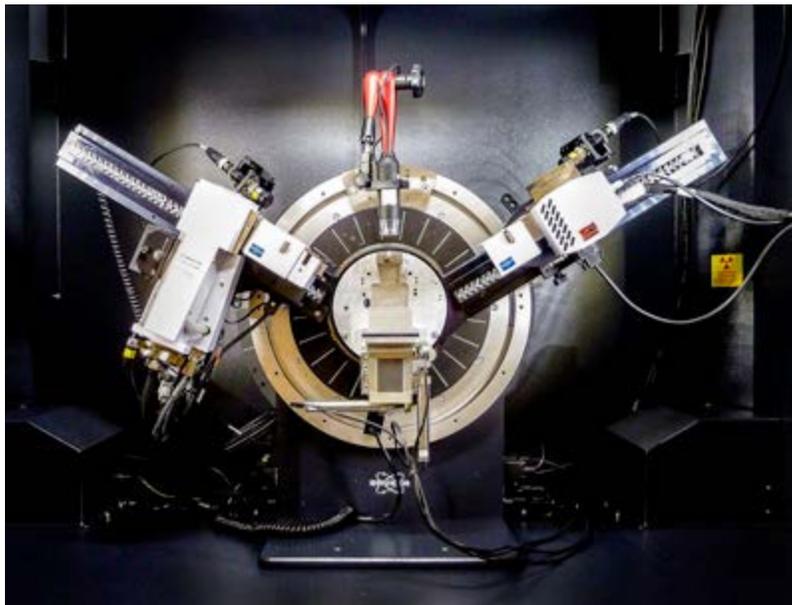


# Powder diffractometer operation instructions

## D8 Advance with a Cu K $\alpha$ sealed tube and Lynxeye

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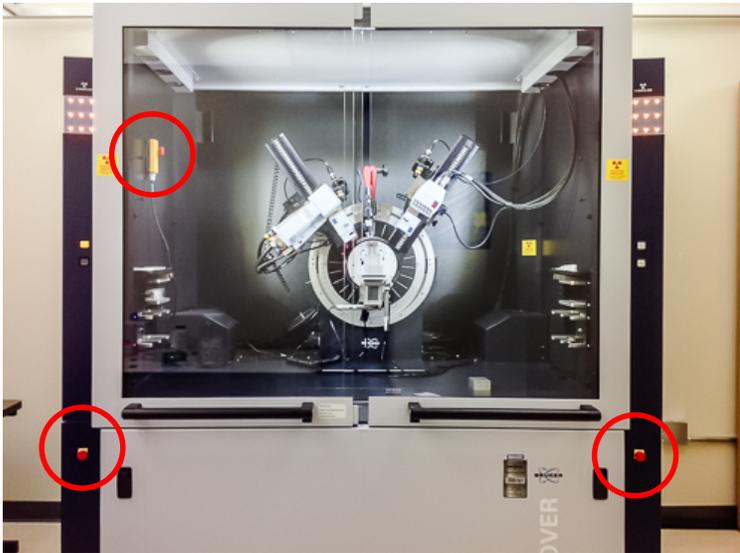
MARCH 30, 2016



## SAFETY.

For radiation safety materials refer to <http://www.ehs.wisc.edu/raddevices.htm>.

There are three emergency shut-off buttons on this instrument:



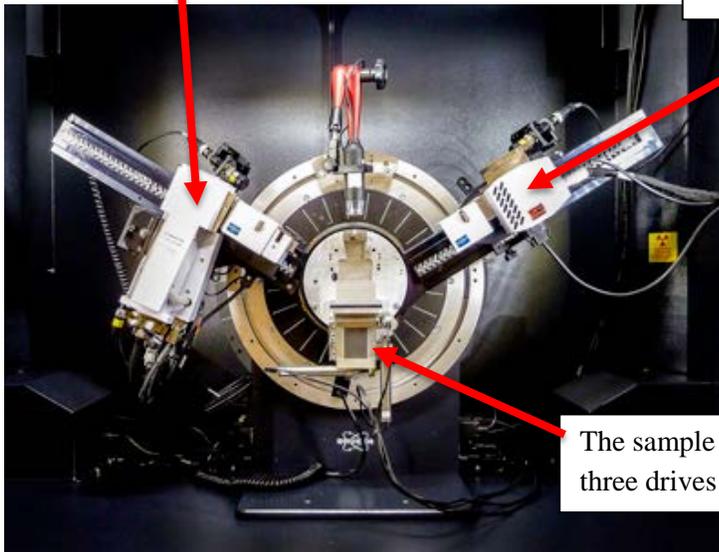
Always ensure the shutter is closed prior to opening the instrument doors.

If anything goes wrong with the instrument – do not try to fix it – contact Iliia Guzei ([iguzei@chem.wisc.edu](mailto:iguzei@chem.wisc.edu), 608-263-4694), or Bob Shanks ([rbshanks@wisc.edu](mailto:rbshanks@wisc.edu), 608-262-0563).

## SETUP

The Cu K $\alpha$  X-ray tube is on the left. The beam is collimated with a 0.6 mm slit

The 192 channel Lynxeye detector is on the right. The 0.2-mm nickel foil filters out K $\beta$  beams at the detector.



The sample stage is motorized with three drives (X,Y,Z).

The X-ray tube is always energized and the yellow light is on. When the shutter is closed (red light on the X-ray tube is off) it is safe to open the enclosure. Do not attempt to open the enclosure when the shutter is open.



The instrument doors are opened by pushing on the button in the handle. The switch under the handle controls how the doors open (slide or swing).

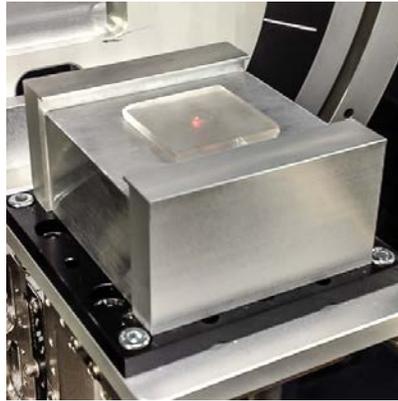
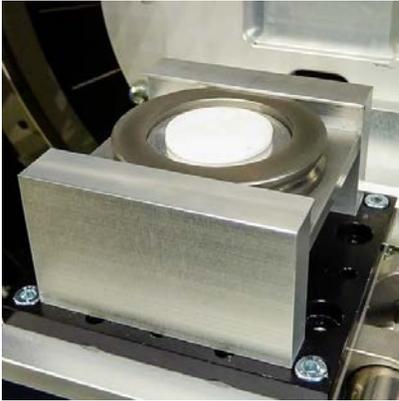


Press the grey button on the camera to turn it on.

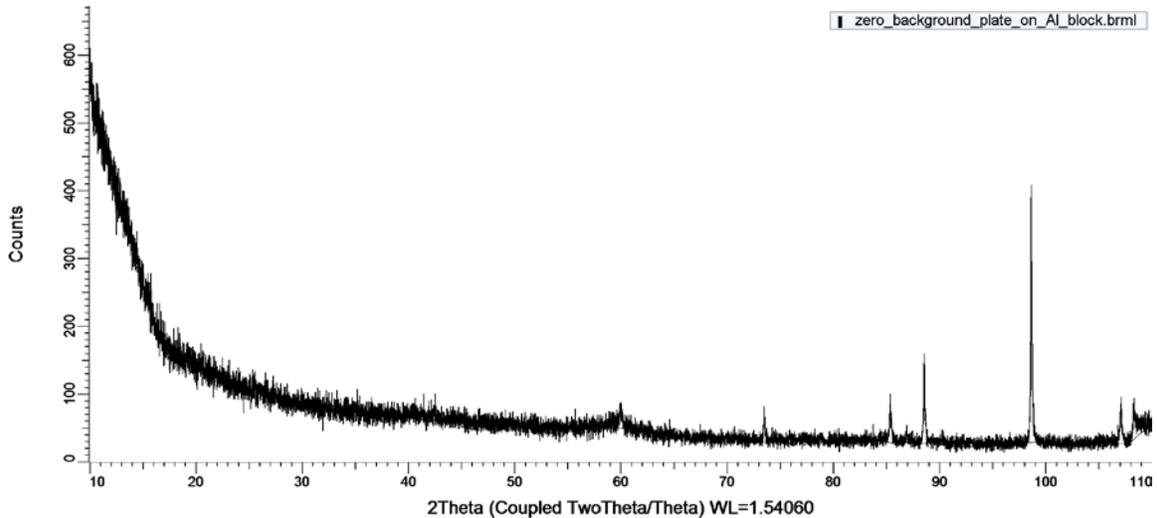


Conventional samples in round sample holders should be placed on the spring plate, the plate goes just beneath the wings, and the spring goes beneath the plate (photo below left). The plate and spring can be removed with two fingers (one on each side of the spring).

For a film, use the zero-background plate placed on a precision aluminum block (photo below right).



FYI: The uncorrected pattern below is for a blank rectangular zero-background plate placed on an Al block. Scan parameters: range: 10-110° in 2 theta, increment = 0.01°, exposure time 0.1 sec/step.



The instrument precision is no better than 0.01°, thus all results should be reported at the most to the second decimal place.

## SAMPLE ALIGNMENT

The goal is to set the correct sample height on the instrument. Height error of +0.1 mm will result in the entire powder pattern being shifted to lower 2theta values by  $-0.039^\circ$  and an error of -0.1 mm will shift the pattern by  $+0.039^\circ$ .

### **Visual alignment**

The goal is to bring the two laser points into coincidence as shown below.



Open "Measurement" program.

Log in with your chemistry user name and password assigned during training.

Select the Commander module.





The axes can be driven only when the instrument doors are closed.

Note that in the example above it is not necessary to move X and Y because the current values are the correct target values.

2. Now change the value of the Z drive, put a check mark next to it and drive the axis. Watch the camera video to track the laser positions. Repeat until the two laser spots coalesce.

### Z-scan.

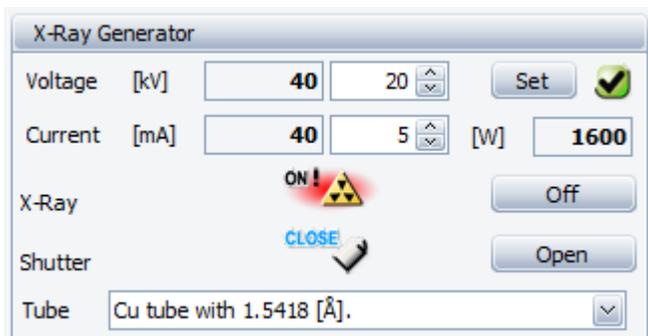
For precise sample alignment a Z-scan is highly recommended.

1. Set Theta = 0° and Detector = 0°. The example below shows the set up for the standard. Its height happened to be 1.500 mm.

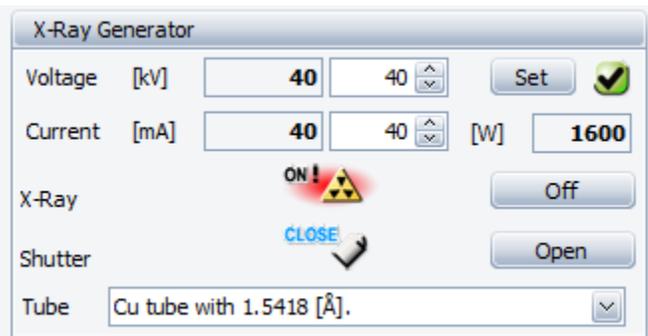
Drive	Current	Requested	<input type="checkbox"/>		
Theta	[°]	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>	<input checked="" type="checkbox"/>	
Two Theta	[°]	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>	<input type="checkbox"/>	
Detector	[°]	<input type="text" value="0.0000"/>	<input type="text" value="0.0000"/>	<input checked="" type="checkbox"/>	
X	[mm]	<input type="text" value="10.000"/>	<input type="text" value="2.000"/>	<input type="checkbox"/>	
Y	[mm]	<input type="text" value="11.240"/>	<input type="text" value="2.000"/>	<input type="checkbox"/>	
Z	[mm]	<input type="text" value="1.500"/>	<input type="text" value="1.500"/>	<input checked="" type="checkbox"/>	

2. Set the generator to 40 kV and 40 mA.

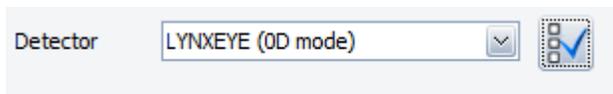
In the example below the current power is 40 kV and 40 mA (the desired values), but the “Requested” values are 20 kV and 5 mA, and if these requested values remain unchanged the instrument will power itself down when a new scan is started.



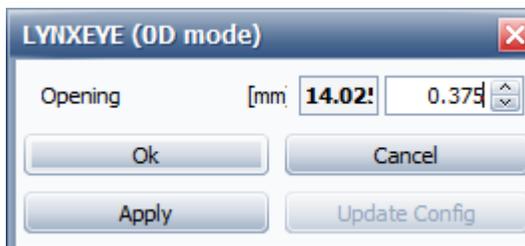
Thus, change the Requested values to 40 kV and 40 mA and press the Set button.



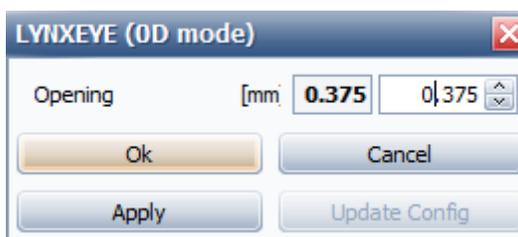
3. Change the Detector settings to Lynxeye 0D by selecting in from the dropdown menu.



Click the check box on the right to adjust the detector's properties.

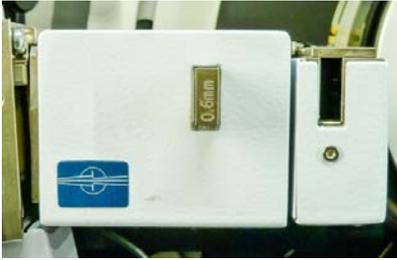


Change the Opening value to 0.375, then press Apply. It is important to first press Apply rather than OK.



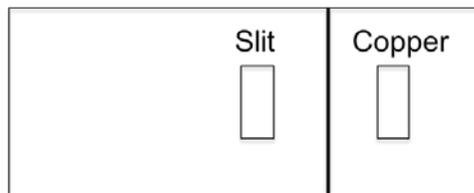
Now press OK.

4. On the instrument, inside the enclosure, replace the 0.6 mm slit



with a 0.2 mm slit. Only use the slits stored on the left side of the instrument! Insert the 0.1 mm Cu foil into the right opening.

Note – the beveled edge of the slits should be on the bottom of the inserted end.



5. Adjust the Scan parameters in the Experiment Design module.

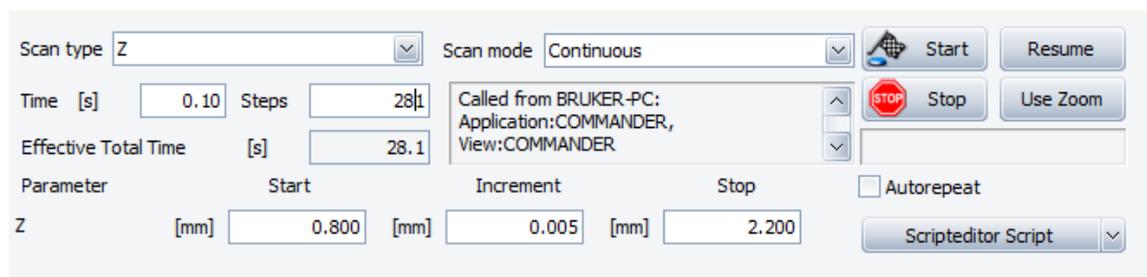
Select Scan type Z.

Set Time to 0.10 sec.

Choose the Start value to be your sample height (call it H) to be  $H-0.5$ .

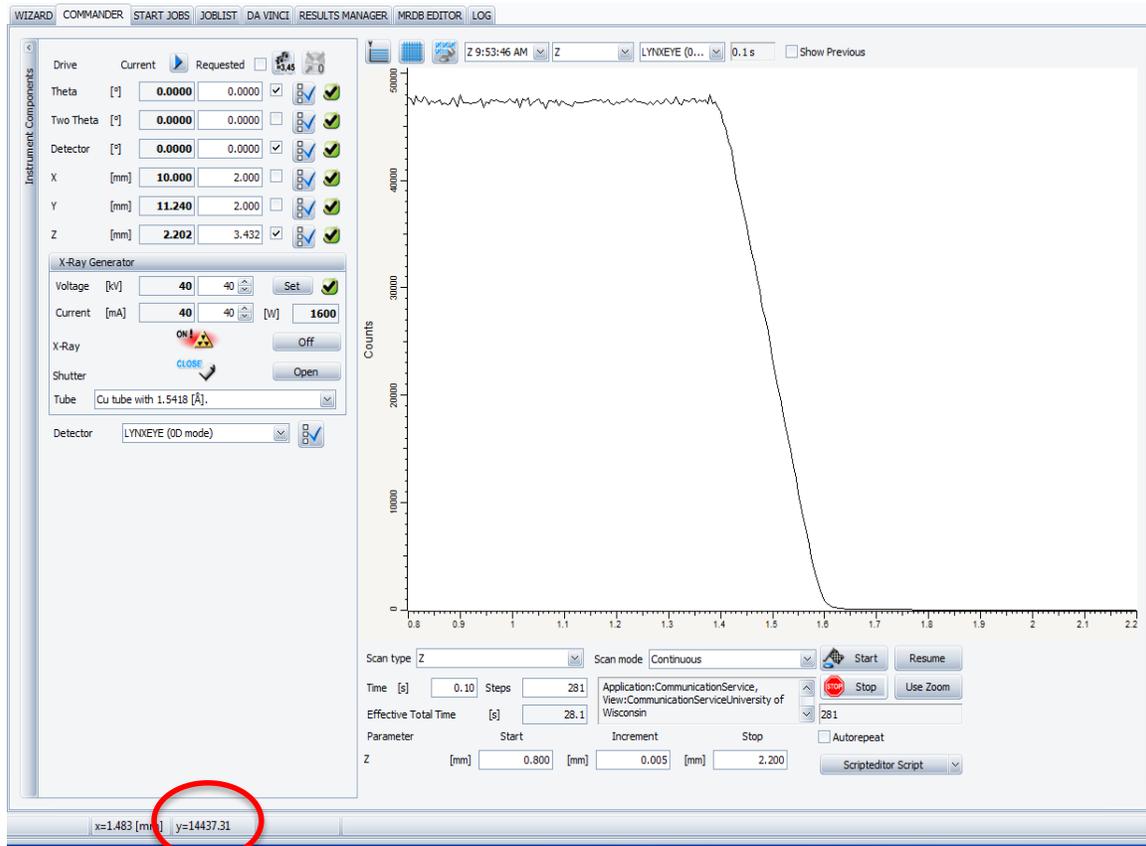
Choose the increment to be 0.005 mm.

Set the Stop to be  $H+0.5$ .



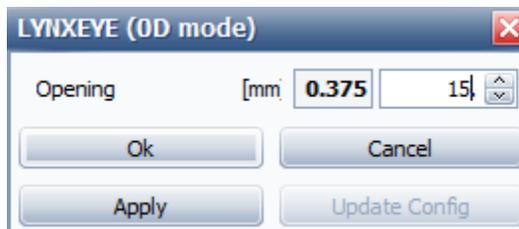
Click on Start.

Your graph should look similar to

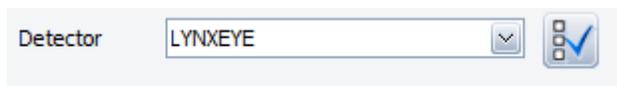


Determine the maximum intensity by placing the cursor at the top curve plateau and checking the value in the status bar (circled in red). Find the midpoint on the curve (use the circled y value in the figure above for precision) and double-click there – the correct Z value will be automatically inserted in the Requested column for the Z.

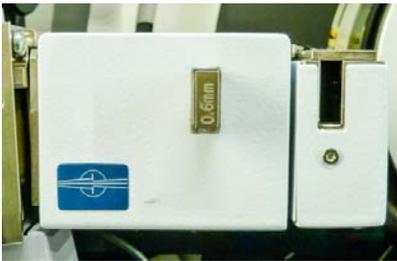
- Change the detector settings in the reverse order. First set the opening 14.325 (or type 15 and the software will set the closest physically possible value automatically).



Then change the detector type to Lynxeye.



7. Inside the enclosure remove the Cu foil from the slit and replace the 0.2 mm slit with a 0.6 mm slit. Remember: beveled edge down.



### SCAN PARAMETER OPTIMIZATION

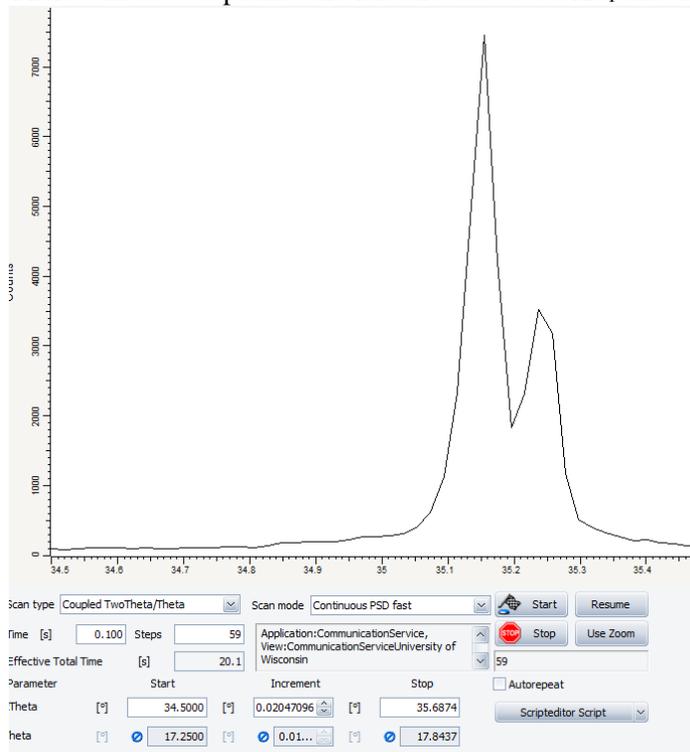
Prior to running a full scan you should choose the correct exposure time and increment.

The procedure is demonstrated for the standard corundum sample. A peak at  $35.149^\circ$  is chosen for the optimization.

Set  
Scan type = Coupled TwoTheta/Theta.  
Exposure = 0.1 sec.  
2Theta start =  $34.5^\circ$ .  
Increment =  $0.02^\circ$ .  
Stop = 35.7.

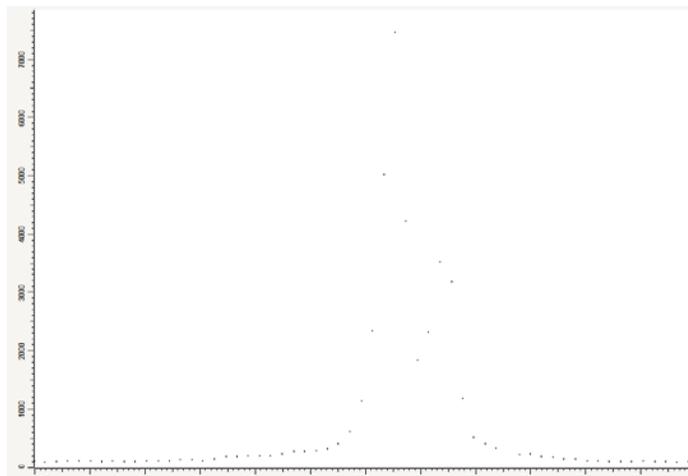
Parameter	Start	Increment	Stop
2Theta [°]	34.5000	0.02047...	35.6874
Theta [°]	17.2500	0.01023...	17.8437

There will be two peaks –the left one due to Cu  $K\alpha_1$  and the right due to Cu  $K\alpha_2$ .

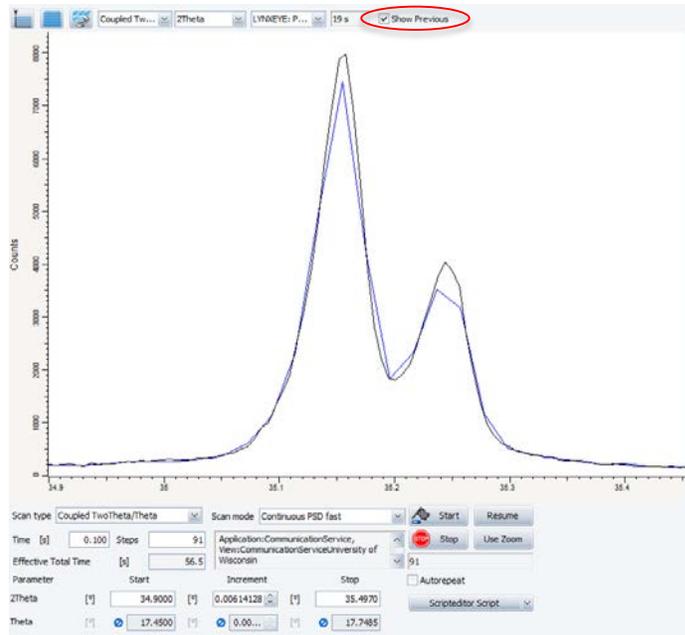


Adjust the Time until you are satisfied with the peak height. For this sample 0.1 sec is fine.

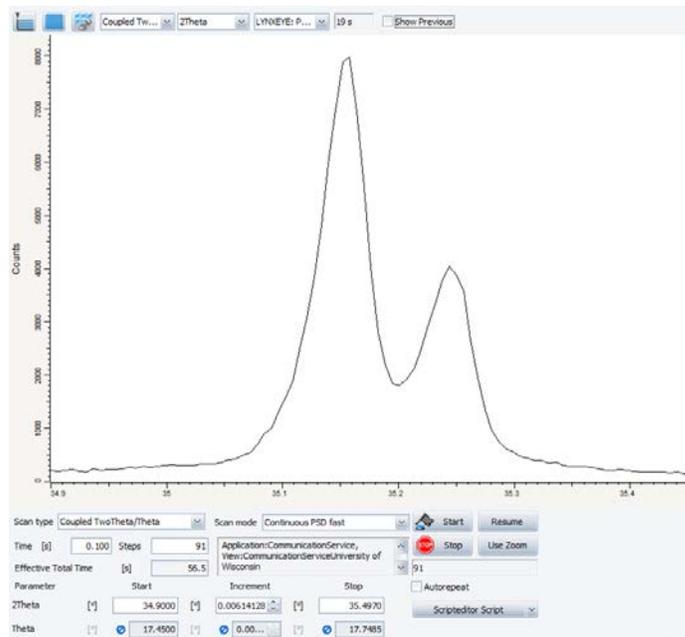
Then choose the Increment. Ideally, there should be 6 to 12 data points above the peak half maximum. The figure below shows the data points (right-click, dots). There are only three points above the half-max in this case. Thus, the step size should be reduced.



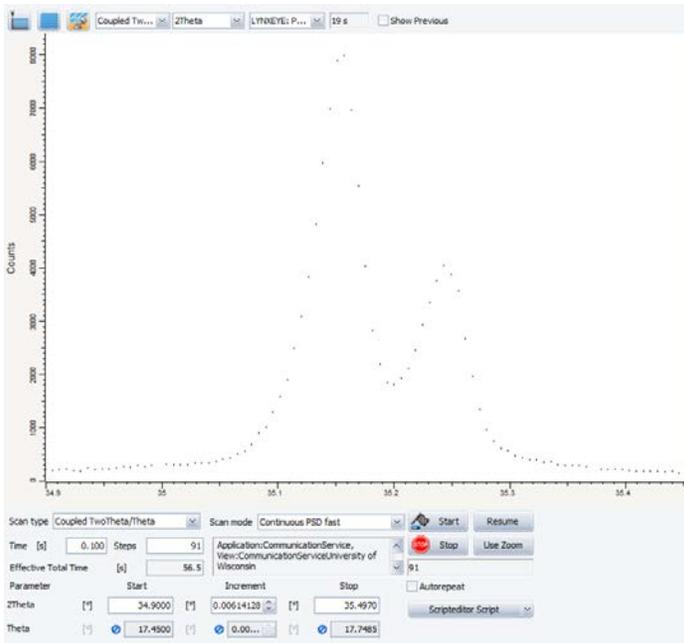
Here, the step size was reduced to  $0.006^\circ$ , and the sample rescanned in a narrower range  $34.9-35.4^\circ$ . The graph below shows the new scan in black and old one in blue (use the Show Previous box (above the pattern window) to show or hide the previous pattern). The peak profiles are much better for the black scan.



Hide the previous scan.



Check the number of data points.

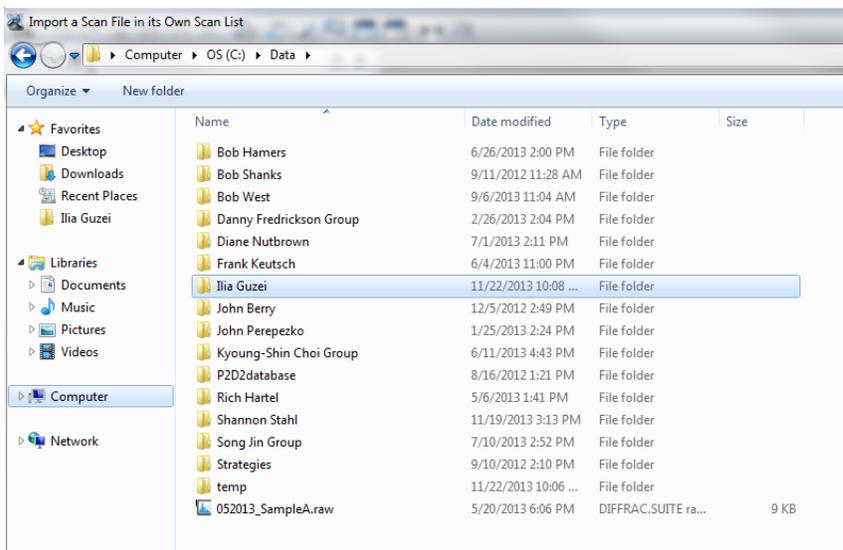


Repeat until you get a satisfactory step size (increment) for your scan.

## FULL SCAN

You are now ready to run a full scan of your sample with the optimized Time and Increment parameters.

When finished, make sure to Save Result File. All data must reside inside C:\Data\Your Adviser\Your name folder.



## Acknowledgement

Many thanks to Anthony Cesnik for sharing his training notes with the other users.