

Bruker D8 HRXRD  
**Collecting X-Ray Reflectivity Data**  
**using the PathFinder Detector**

**Abridged SOP for Manually Aligning a Sample and Collecting Data  
using XRD Commander**

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This SOP describes the steps necessary to manually align a sample and collect a single X-Ray Reflectometry (XRR) scan using the Pathfinder detector system. This SOP is designed to act as a general guide that will work for most samples. You might be able to devise a more efficient procedure for your specific sample.

These instructions can also be used for the collection of diffuse scatter data, small-angle X-ray scattering (SAXS) data, and small-angle X-ray diffraction (SAXD) data.

Other SOPs are available that describe the different mounts and modes of operation using the LynxEye detector, details of using XRD Commander, and details of using XRD Wizard for automated data collection.

This manual will instruct you to **Drive, Scan, Zoom, Optimize**, within XRD Commander. The general instructions for using XRD Commander the document “Expanded SOP for the Bruker D8 HRXRD using XRD Commander and XRD Wizard.docx”, which can be found on the desktop of the Bruker D8 HRXRD data collection computer. That SOP gives more explicit step-by-step instructions.

The sections of this SOP are:

- I. Hardware choices to make before doing an XRR measurement
  - II. Aligning the sample
  - III. Manually collecting an XRR pattern
  - IV. Using XRD Wizard to collect an XRR pattern
- Appendix A. A brief overview of XRR basics

## **I. Selecting a Hardware Configuration for XRR Data Collection**

### **1. Incident-Beam Configuration**

The Goebel mirror is always mounted, as is the rotary absorber. By default, the Ge(022)x4 asymmetric monochromator is always mounted. The monochromator can be removed so that you collect data with the Goebel mirror by itself.

*Please remember that you are never allowed to remove or insert the incident-beam monochromator. If you want the monochromator changed, you must contact SEF staff to make the change for you. Requests to change the monochromator should be e-mailed to [speakman@mit.edu](mailto:speakman@mit.edu) at least one business day in advance whenever possible.*

Incident-Beam Configuration	Intensity (cps)	Beam Spectrum	Beam Divergence (arc-seconds)	FWHM of Si(220)
Mirror only	170,000,000	K $\alpha$ 1+K $\alpha$ 2	108	0.07°
Ge(022)x4 asymmetric	18,000,000	K $\alpha$ 1	25	0.008°

***For films that are <10nm thick***, it is necessary to use only the Gobel mirror because the data must be collected to very high angle; this requires a high incident-beam intensity.

***Films that are very thick, >100 nm, and superlattice stacks*** require an incident-beam monochromator in addition to the Gobel mirror. The monochromator further reduces the divergence of the X-ray beam and provides much better angular resolution of the intricate detail in the fringes produced by a thick film or multilayer superlattice stack. However, the monochromator greatly reduces the incident beam intensity, and therefore limits the maximum angle to which XRR data can be collected.

***Films with an intermediate thickness, between 10nm < t < 100 nm***, can be collected with or without the incident beam monochromator.

The beam height limiting slit, which goes in the first monochromator slit position, must not be larger than 0.2mm. A smaller slit is appropriate for very small samples or when the critical angle must be measured with great accuracy for calculations of film density and/or composition. A larger divergence slit provides greater intensity at high angles for the analysis of very thin films (<10nm thick).

The length (L) of the X-ray beam on the sample is:  **$L=h/\sin(\omega)$** , where h is the height of X-ray beam determined by the slit and  $\omega$  is the incident angle.

Height-Limiting Slit	Length of X-ray Beam at incident angle equal to ...		
	0.5°	1°	4°
0.2 mm	23 mm	11.5 mm	2.9 mm
0.1 mm	11.5 mm	5.7 mm	1.4 mm
0.05 mm	5.7 mm	2.9 mm	0.7 mm

## 2. Decide what Detector System and Receiving-Side Optics you are going to use

XRR data are usually collected with the Pathfinder detector, which is installed on the system by default. The Pathfinder is a scintillation point detector. The scattered-beam path passes through a single computer-controlled motorized receiving slit. The detector can be saturated or even damaged by an X-ray beam that is too intense, so an absorber must sometimes be used to reduce the intensity.

There is one slit inserted into the slit box in front of the detector. This slit will be the same size as the receiving slit (which is computer controlled). This slit is usually 0.2mm, 0.1mm, or 0.05mm.

A larger slit allows more intensity from the specular reflection to reach the detector, but also allows more noise from air scatter and diffuse scatter. A smaller slits reduces the amount of noise and improves the angular resolution for observing fine fringe details. However, the smaller slit may reduce the ability to characterize very thin films (<10nm) using high angle data.

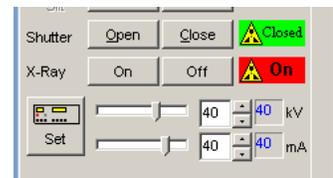
When you insert a slit, make sure it is straight vertically and that it slides all of the way in (about 1cm of the slit will be sticking out of the slot). The blades of the slit should be facing the detector; the numbers on the slit will be oriented upside down with respect to the sample position.

## II. PREPARING TO COLLECT DATA

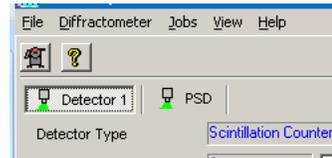
1. Start the programs XRD Commander and XRD Wizard
2. Select the program XRD Commander
3. Select the Adjust page
  - a. There are four tabs along the bottom of the XRD Commander window, labeled Adjust, Jobs, Geometry, and Details



4. Set the X-Ray Generator power to **40 kV** and **40 mA**.
  - ***Give the generator at least 30 minutes at full power to warm up before beginning your measurements!!***
  - a. The generator controls are located on the left-hand side of the XRD Commander window
  - b. The black numbers are the desired value, **the blue numbers are the current value**
  - c. Change the black numbers for kV and mA to the desired setting, 40kV and 40mA
  - d. Click on the  **Set** button
  - e. Wait until the actual values (**in blue**) change to the desired value



5. Set the detector
  - a. Select the Details tab
  - b. In the upper right-hand corner of XRD Commander, make sure that **Detector 1** is selected, not PSD.
  - c. Select the Adjust tab
  - d. Select the Secondary Optic using the drop-down menu
    - i. Select **Pathfinder-Variable Slit**

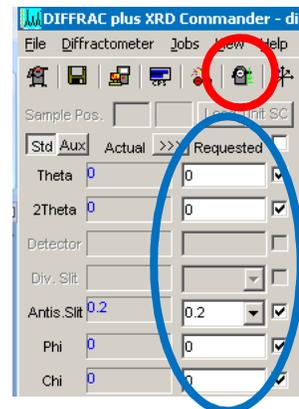


- ii. The drop down menu for the secondary optic is the second blank drop-down box in the Toolbar for XRD Commander
- iii. When you float the mouse over the button, the name of the button appears
- iv. After you select the Secondary Optic from the drop-down menu, the button will be filled with the icon for that optic.

6. Mount the sample- see the Sample Stage SOP for instructions

!! Remember!!

- To **Drive** a motor, type the target value in the Request value column. Once the number is typed, click the **Move Drives** button (circled in red).
- The Receiving Slit is labeled “**Antis. Slit**”. In the instructions below, I will refer to it as the Receiving Slit (or Rec. Slit) when describing what it does and as the Antis. Slit when directing you to make a specific change in the XRD Commander program.
- For all scans, the scan mode should be **Continuous** (not Step)



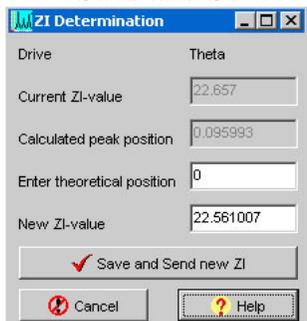
### **III. ALIGN Z BY BISECTING THE BEAM**

- *Why-* we need to optimize Z so that the X-ray beam is properly focused on your sample. We do this by determining the value of Z where the sample cuts the X-ray beam in half.

#### 1. **Set** the Absorber

- a. Using the **Absorber** drop-down menu, select a value
- b. Click the **Set** button
  - If using the Gobel mirror, **Set** the Absorber to 78.2
  - If using the Ge(022)x4 monochromator, **Set** the Absorber to 78.2
  - If using the Ge(044)x4 monochromator, **Set** the Absorber to 1

2. Determine the position of the direct X-ray Beam by using a Detector Scan
  - a. **Drive** the instrument to the following positions:
    - i. Theta=0
    - ii. 2Theta=0
    - iii. Antis. Slit= 0.2
    - iv. Phi=0
    - v. Chi=0
    - vi. X=0
    - vii. Y=0
    - viii. Z=-1.5
  - b. **Start** a Detector Scan
    - i. Scantype= Detector Scan
    - ii. Start= -0.2
    - iii. Increment= 0.002
    - iv. Stop= 0.2
    - v. Scanspeed= 0.2 sec/step
  - c. Redefined the peak maximum as  $0^\circ$  2Theta
    - i. click on the  **Zi** button in the toolbar to open the *Zi Determination* window
    - ii. Set “**Enter theoretical position**” to **0**
    - iii. Click **Save and Send new Zi**



- If the X-ray beam has an odd shape, such that the **Zi** peak search does not properly identify the peak centroid, then see Appendix B (pg 20) for the refined procedure.
- d. Repeat the Detector Scan and make sure that the peak is centered around  $0^\circ$  2Theta
3. Determine the Z position where the sample cuts the X-ray beam intensity in half
    - a. **Drive** 2Theta to 0
    - b. **Start** a Z scan
      - i. Scantype= Z
      - ii. Start= -1.0
      - iii. Increment= 0.01
      - iv. Stop= 1.0
      - v. Scanspeed= 0.1 sec/step
    - c. **Optimize** at the point on the chart where the X-ray intensity is  $\frac{1}{2}$  the maximum intensity
      - i. If the intensity of the X-ray beam in the Z-scan does not go all the way to zero, then see Appendix C (pg 21) for details on how to deal this.

4. Make sure that the sample surface is parallel to the X-ray beam
  - a. **Start** a Rocking Curve Scan
    - i. Scantype= Rocking Curve
    - ii. Start= -1
    - iii. Increment= 0.01
    - iv. Stop= 1
    - v. Scanspeed= 0.1 sec/step
  - b. **Optimize** on the center of the maximum
  
5. Iteratively improve the alignment of Z and Theta
  - a. Repeat the Z and Rocking Curve scans until the optimal position for both does not change by more than  $\pm 1\%$  between successive scans
  - b. The Z Scans that you use should have parameters:
    - i. Scantype= Z
    - ii. Start= optimized Z position - 0.3
    - iii. Increment= 0.005
    - iv. Stop= optimized Z position + 0.3
    - v. Scanspeed= 0.1 sec/step
  - c. The Rocking Curve scans that you use should have parameters:
    - i. Scantype= Rocking Curve
    - ii. Start= optimized Theta position - 0.5
    - iii. Increment= 0.005
    - iv. Stop= optimized Theta position + 0.5
    - v. Scanspeed= 0.1 sec/step
  
6. When you have determined the optimal aligned Z value
  - a. **Drive** Z to the optimized value
  - b. Uncheck the box next to Z so that Z will not be changed again
  - c. This optimal Z value will not change for any of the scans of your sample
  
7. You might want to record the optimized value of Theta as *Tilt(sample)*
  - a. *Tilt(sample)* indicates the Theta value when the physical surface of the sample is parallel to the X-ray beam
  - b. If the tilt of the substrate peak (determined later) is significantly different than *tilt(sample)*, this would indicate a miscut in the substrate
  - c. Assuming that your sample is not miscut, we can use the *tilt(sample)* to save time when aligning on the Bragg peak of the substrate.

#### **IV. ALIGN THE TOTAL EXTERNAL REFLECTED X-RAY BEAM**

- We want to align on the sample to produce the strongest reflected signal.
  - In these alignment procedures, the most important thing is to align the sample so that it produces the most intense and sharpest rocking curve from the substrate peak.
    - If you are not sure what the optimal value is for a position, such as the Chi tilt, then collect rocking curves at different values for that position. The optimal position is the one that gives you the most intense and sharpest rocking curve.
1. **Set** the Absorber to **Auto**
  2. **Drive** 2Theta to 0.4.
  3. **Drive** the Antis. Slit to the size that you will be using to collect data.
    - a. This is usually the same size as the height limiting slit you put in the monochromator.
    - b. This could be 0.2 to 0.1mm. The slit cannot be smaller than 0.1mm.
  4. Optimize the substrate tilt in the diffraction plane (Omega) using a Rocking Curve
    - a. **Start** a coarse rocking curve
      - i. Scantype= Rocking Curve
      - ii. Start= 0 or *Tilt(Sample)*, whichever is smaller
      - iii. Increment= 0.01
      - iv. Stop= 0.4 + *Tilt(Sample)*
      - v. Scanspeed= 0.1 sec/step
    - b. Collect a more precise rocking curve
      - i. **Zoom** around the peak and click the **Use Zoom** button
      - ii. Change the increment to 0.002deg
      - iii. **Start** the rocking curve
    - c. **Optimize** on the Rocking Curve Peak
      - i. Optimize on the center of mass of the parabola that defines the top half of the peak
      - ii. This will not necessarily be the maximum of intensity

#### **V. ALIGN ON A REFLECTED INTERFERENCE FRINGE**

1. Collect a Reflectometry Curve to find an interference fringe
  - a. **Start** a 2Theta-Omega Scan
    - i. Scantype= 2Theta-Omega
    - ii. Start= 0.2
    - iii. Increment= 0.005
    - iv. Stop= 4
    - v. Scanspeed= 0.1 sec/step
  - b. Change the intensity scale to log by clicking on the “ln y” button



- c. Optimize on the first or second clearly defined fringe peak
  - i. if fringes are closely spaced together, optimize on the second fringe peak
  - ii. if fringes have long periods, optimize on the first fringe peak
  - iii. if you don't see any fringes, determine the critical angle (the angle at which intensity begins to decrease from its maximum) and optimize at a point at least  $0.2^\circ$   $2\theta$  higher than that angle
  
2. Optimize Theta using a Rocking Curve
  - a. **Start** a Rocking Curve
    - i. Scantype= Rocking Curve
    - ii. Start= *current Theta position - 0.2*
    - iii. Increment= 0.002
    - iv. Stop= *current Theta position + 0.2*
    - v. Scanspeed= 0.1 sec/step
  - b. Change the intensity scale to linear by clicking on the “y” button
 
  - c. **Optimize** on the maximum of the rocking curve
  
3. Optimize the substrate tilt in the axial direction (Chi)
  - a. **Start** a Chi scan
    - i. Scantype= Chi
    - ii. Start= -2
    - iii. Increment= 0.02
    - iv. Stop= 2
    - v. Scanspeed= 0.1 sec/step
  - b. **Optimize** on the centroid of the peak
    - i. If there are two peaks, then optimize in between those peaks
      1. This may mean that the sample is too rough to collect a good XRR pattern
    - ii. If there are three peaks, optimize on the peak in the middle
    - iii. If the peak is too broad to clearly resolve the maximum, then repeat the Chi scan using a range from -4 to 4 deg with a 0.05deg increment
  
4. Optimize the Rocking Curve again
  - a. **Set** the scan type to Rocking Curve
  - b. **Start** a Rocking Curving using the previous scan parameters
  - c. **Optimize** on the Rocking Curve
  - d. The rocking curve may be much sharper than the previous scan
    - i. **Zoom** around the peak and click **Use Zoom** to redefine the start and stop positions
    - ii. Change the increment to 0.001 deg
    - iii. **Start** the Rocking Curve scan
    - iv. **Optimize** on the centroid of the rocking curve
  
5. Repeat steps 4 and 5 (optimize Chi and optimize rocking curve) until both are optimized
  - a. The optimum rocking curve and Chi positions should not change by more than  $\pm 5\%$  between successive scans
  - b. Chi should be optimized to produce the most intense rocking curve

## VI. COLLECT THE XRR PATTERN

1. Collect your Coupled Scan
  - a. Scantype= 2Theta-Omega
  - b. Start= 0.1
  - c. Increment= 0.001
    - i. Thinner films, <10nm thick, have a long period to their fringes and can be collected with a larger increment. An increment of  $0.002^\circ$  is typical, though increments as large as  $0.004^\circ$  may be used.
    - ii. Thicker films may require a smaller increment if collected using the monochromator.
  - d. Stop is usually a value between  $4^\circ$  and  $10^\circ$ 
    - i. Thinner films require a longer scan range. Films <10nm thick should have data collected out to at least  $8^\circ$  2theta, up to a maximum of  $10^\circ$  2theta
      1. Intensity greatly decreases at higher angles, possibly necessitating the use of a multirange scan (explained in section IV).
    - ii. Thicker films can be well characterized with data collected only to  $6^\circ$  2theta
      1. For quick scans of simple films, sometimes data will only be collected to  $4^\circ$  2theta
      2. It is not improper to collect data out to a maximum of  $10^\circ$  2theta for thick films.
  - e. Scanspeed is usually 0.2 sec/step or longer
    - i. If you are not sure what the scanspeed should be, you can use the **Repeat** option to repeatedly collect the scan, adding the results until you have a good enough signal for analysis.
    - ii. You might want to use a fast, coarse coupled scan to make sure you have good start and stop values and to evaluate what scanspeed you should use.
2. Save your Scan
  - ***Data are not saved automatically when you collect them with XRD Commander***
  - a. Before saving the scan, you can type a description of the sample and/or experiment in the **Sample ID** line in the toolbar. This will be saved as the sample ID in the data header.
  - b. Save the scan by going to *File > Save* or clicking on the **Save** icon in the toolbar
  - c. Save your data in the “Diffrac Plus Ext. RAW file (\*.raw)” format

## IV. USING XRD WIZARD TO COLLECT AN XRR PATTERN

XRD Wizard can be used to write a program to collect and automatically save data. It can also be used to facilitate running multiple measurements one after the other.

This section will describe using a HRXRD type program to collect XRR data. XRR data can also be collected using an XRD type program. This allows you to collect the XRR data using different scan speeds for different 2theta ranges in the same data file. Consequently, a faster scan speed can be used to collect data at low angles where the reflected beam is very intense and using slower scan speeds at high angles where the signal is very weak.

### **Collecting XRR Data using an HRXRD Program**

1. Prepare the instrument and align the sample following steps I and II above.
2. Start XRD Wizard
3. If a new, blank HRXRD program is not created automatically, then
  - a. go to *File > New* and create a new program
  - b. A dialogue box will ask you select the type of program that you want to create
    - i. Select HRXRD

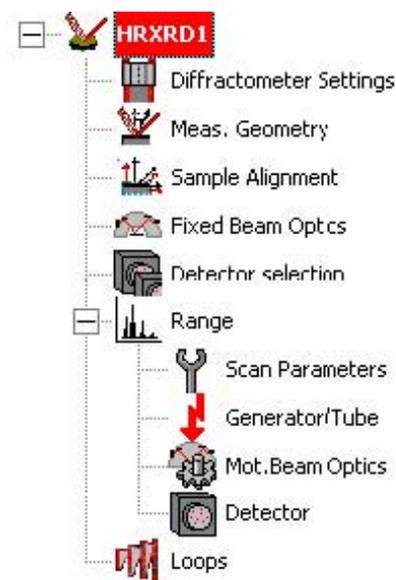
XRD Wizard will walk you through the creation of a data collection program as outlined in the **Structure Edit hierarchal tree** (shown right). In each form, you will complete the data and click **OK**. When you click **OK**, XRD Wizard will take you to the next form shown in the hierarchal tree.

*XRD Wizard works best when you click on **OK** to move from one form to the next, rather than selecting the next form from the Structure Edit tree.*

4. Fill out the optional information in the first form, then click **OK**
5. The **Diffraction Settings** form has no information for you to complete. Click **OK**
6. In the **Measurement Geometry** form, you may enter information for your substrate and one layer. This is not necessary for XRR data collection.

The fields you can fill are: name, surface(mno)- which is the out-of-plane orientation of the crystal, and azimuth(pqr)- which defines the relationship between in-plane directions of your Substrate and one Layer. You may also click on **Cryst. System...** and identify the Bravais Lattice and lattice parameters for you substrate and one Layer.

- *None of this information is necessary for XRR data.*
- Make sure that the Surface (mno) for the Substrate is **NOT** (000).
- Click **OK**



7. In the **Sample Alignment Prior to Measurement** Form

a. In the field “**Alignment at (hkl)**”, set the four boxes to **0 0 0 s** (as shown below)

b. Expand the program **XRD Commander**

i. Calculate  $Theta(offset) = Theta - (2Theta)/2$

ii. You will need to drive Theta and 2Theta to new positions

1. **Drive** 2Theta to 0

2. **Drive** Theta to  $Theta(offset)$ ,

iii. Click on the “**Transmit Positions**” button



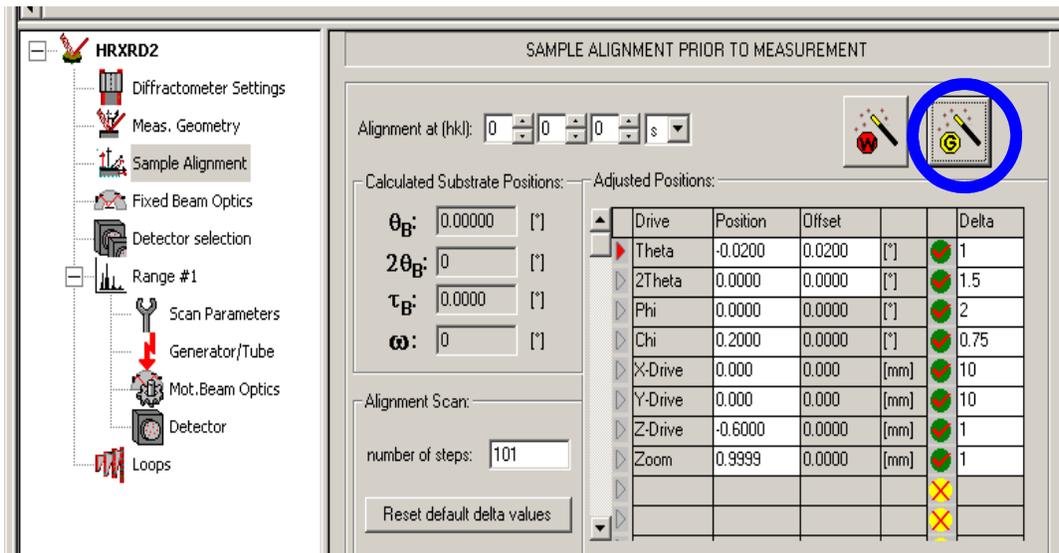
c. Expand the program **XRD Wizard**

i. Click on the “**Get Positions**” button (circled in blue below)

ii. The Offsets column for Theta should be equal to  $Theta(offset)$

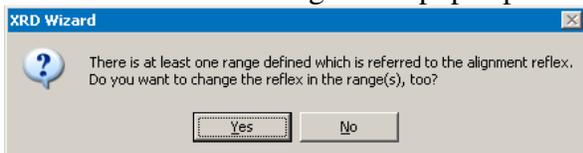
iii. The position values for all other drives (chi, Z) should be the values from XRD Commander

iv. The second to last column should show green circles with a check mark.



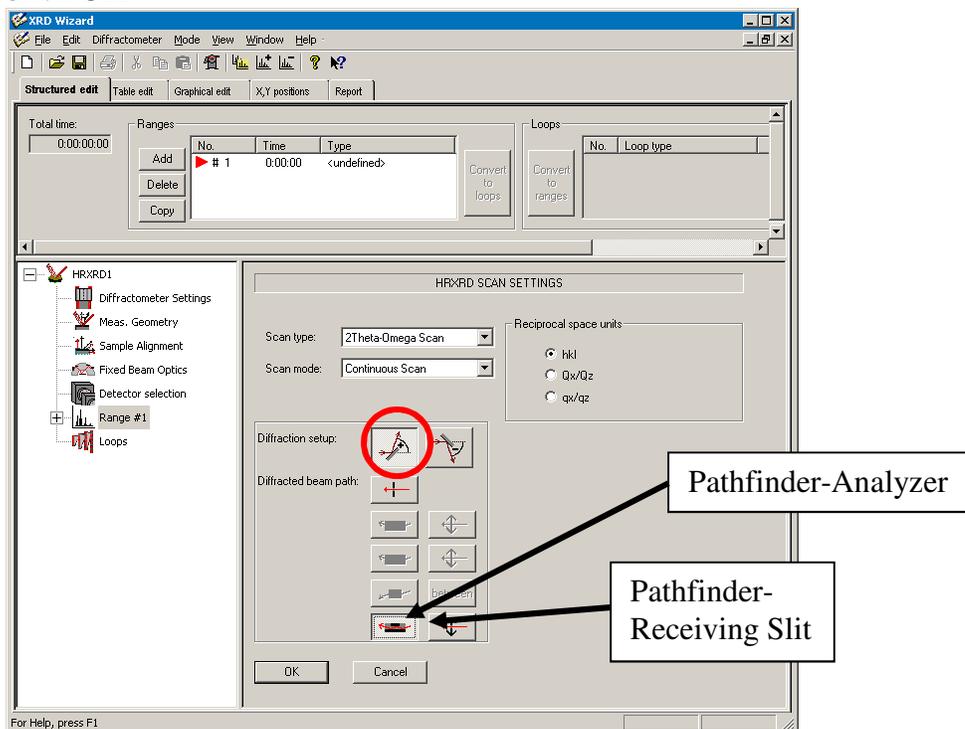
v. Click **OK**

1. if a dialogue box pops up telling you something about a reflex value, click **YES**



8. The **Fixed Beam Optics** form has nothing for you to change. Click **OK**

9. In the Detector Selection form, configure the detector settings that you want to use
  - a. From the drop-down menu, select “**Det #1: Scintillation Counter**”
  - b. Click **OK**
  
10. In the Range #1 form, select the **scan type** from the drop-down menu
  - a. This is usually a 2Theta-Omega scan, but you may choose a different kind of scan
    - i. A Rocking Curve scan would be used to study Diffuse Scatter
    - ii. A Detector Scan would be used for SAXD
  - b. **Scan mode** should be continuous
  - c. The Diffraction setup should always be set to + (circled in red below)
    - i. This designation does not correspond to grazing exit or grazing incident.
  - d. The Diffracted beam path should be set to the receiving-side optic that you want to use.
    - i. You will usually use Pathfinder- Receiving Slit (as labeled below)
    - ii. In some cases, you might use Pathfinder-Analyzer
  - e. Click **OK**



11. In the **Scan Parameters** form, configure the scan parameters that you want
  - a. *Note that start and stop are defined in relative values, not absolute values!!!*
  - b. The Start and Stop parameters are now specified using Relative positions
    - i. The Reference Position is shown in the lower-right corner, in the table labeled “Reference positions”
    - ii. The Rel. start is the range before the Reference Position that the scan will begin. For XRR, this should be 0.1.
    - iii. The Rel. stop is the range after the Reference Position that the scan will stop. For XRR, this should be a value between 4 and 10 degrees. The thinner the film, the longer the scan range needs to be.

