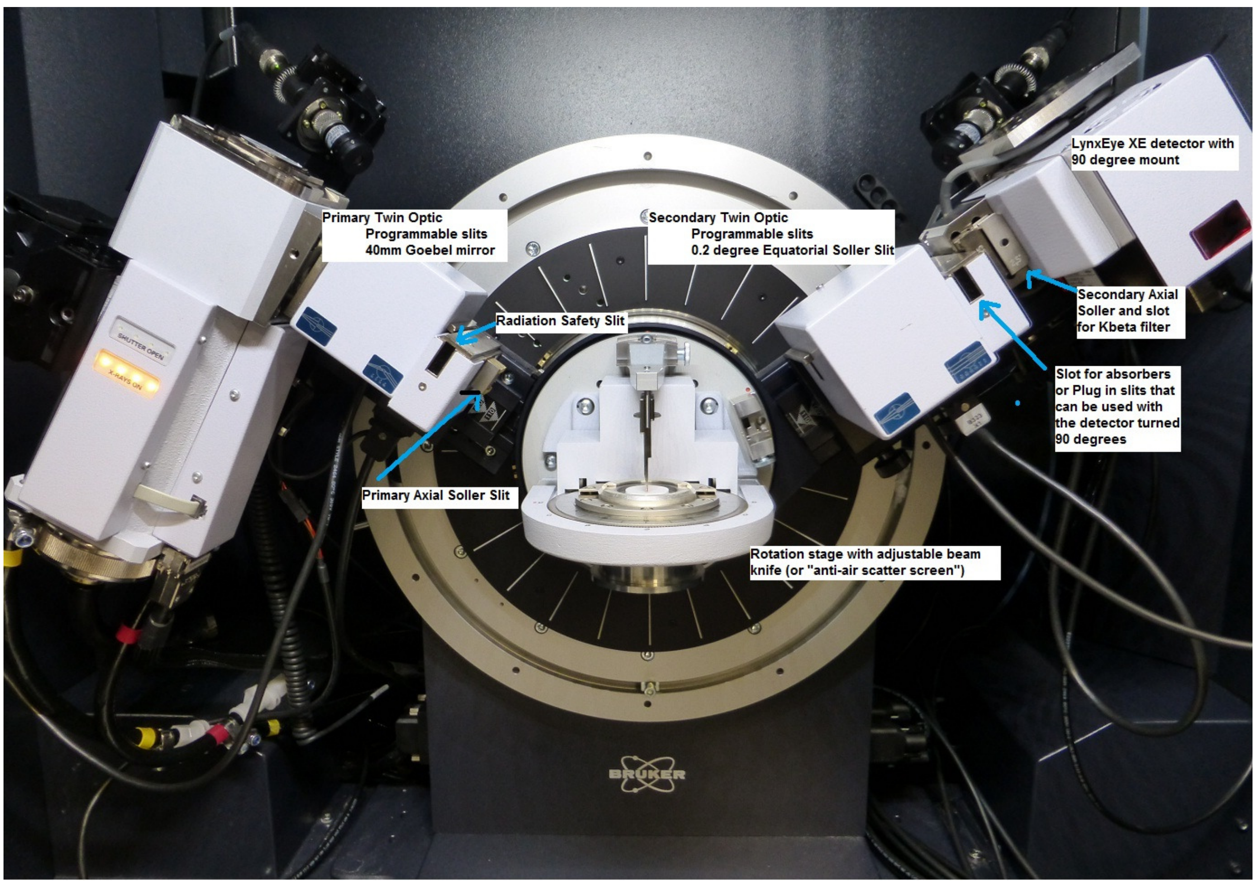
The D8 Advance Configuration with Twin-Twin optics and LynxEye detector

The described configuration is for a D8 Advance DaVinci system in theta/theta geometry with Twin-Twin optics, rotation stage and LynxEye XE detector. The individual components are labelled in the image below.

The rotation stage can be exchanged easily for a capillary stage, sample changers, a compact cradle for texture measurements, a compact xyz stage for micro diffraction or non-ambient stages without affecting the alignment of the optical components.



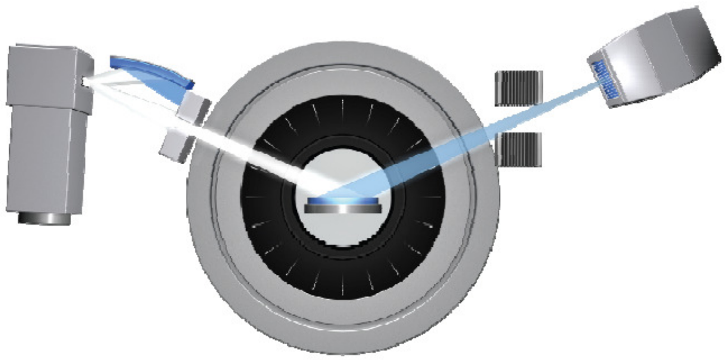
The version of the Diffrac.Measurement software in the screenshots is 3.3.47

The example measurements that are shown were collected with a 2 year old tube in the Application lab, slightly higher intensities may be observed on newer systems.

The described alignment steps are essentially an illustrated and annotated version of the service alignment document for the TWIN-TWIN configuration.

Modes of Operation for the TWIN-TWIN configuration

Bragg-Brentano Geometry

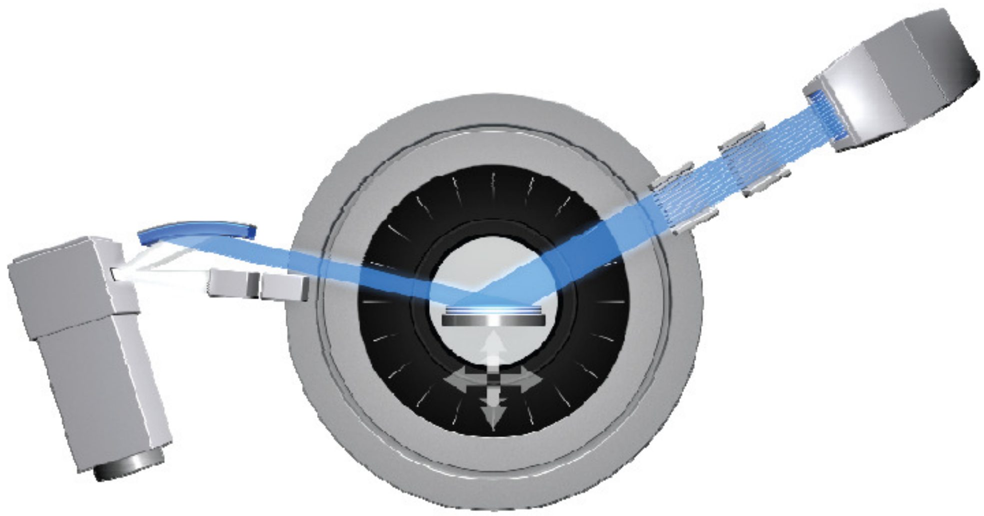


Primary Twin Optic	Set to Motorized Slit: Opening Degree (or slit width) for fixed slits Set to Motorized Slit: Fixed Sample Illumination for variable slits
Radiation Safety slit	Empty or 6mm (small slits will block the incident beam)
Primary Axial Soller	2.5° (4° for more intensity)
Secondary Twin Optic	Opened all the way (2.6°) or completely removed for more intensity
Slit Mount	Empty
Secondary Axial Soller	2.5° (4° for more intensity)
Filter	Ni
LynxEye detector	1D mode (set to 2.95°) for start angles > 3 °

This configuration is the standard setup for powder samples that can be prepared in flat plate geometry. Any deviation of the sample surface from the correct reference height, either through sloppy sample preparation or sample penetration into low density samples will lead to a shift of diffraction peaks.

Advantages: Good resolution and intensity and good crystallite statistics, if a large sample size can be illuminated

Grazing Incidence Diffraction (GID)



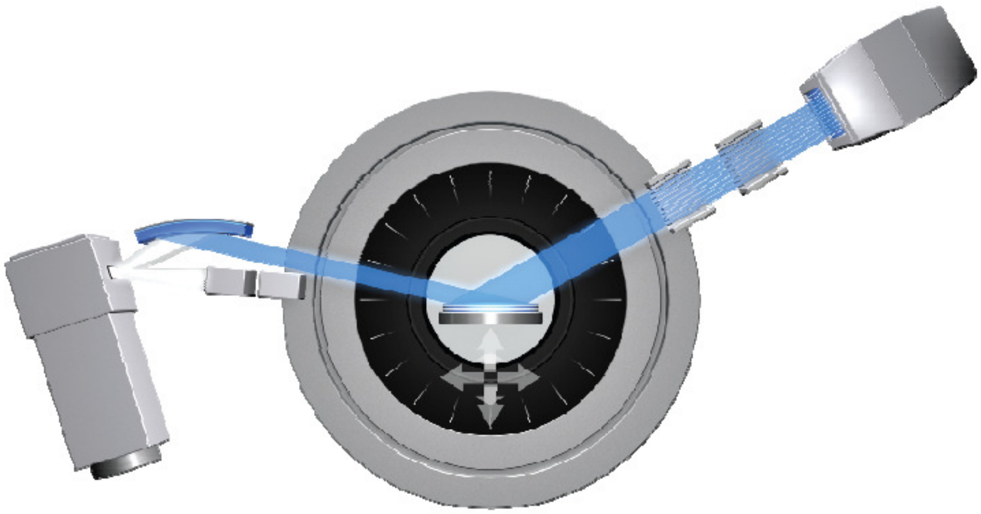
Primary Twin Optic	Set to Goebel Mirror
Radiation Safety slit	1mm for full beam 0.2mm
Primary Axial Soller	2.5°
Secondary Twin Optic	Set to Soller 0.2°
Slit Mount	None
Secondary Axial Soller	2.5° or removed for more intensity
Filter	None
LynxEye detector	0D mode Detector slit set to 14 mm
Scan Type	2Theta scan with theta angle set to a low incident angle

This configuration is used primarily for polycrystalline thin films between a few nanometer and approximately 1 micron thickness for inorganic films. Thicker films of inorganic materials are essentially infinitely thick for Cu radiation and can usually be measured faster and with better angular resolution in Bragg-Brentano geometry

Advantages: By using a low incident angle the whole sample is illuminated and the beam penetration is low, leading to fewer interferences with substrate peaks

Disadvantages: Slower measurement because the LynxEye detector is used in 0d mode. Angular resolution is limited by divergence of equatorial Soller Slits (expected peak width approx. 0.2° FWHM)

Irregular Shaped Samples



Primary Twin Optic	Set to Goebel Mirror
Radiation Safety slit	1mm for full beam, 0.2 or 0.6mm for narrower beam
Primary Axial Soller	2.5°
Secondary Twin Optic	Set to Soller 0.2°
Slit Mount	None
Secondary Axial Soller	2.5° or removed for more intensity
Filter	None
LynxEye detector	0D mode Detector slit set to 14 mm
Scan Type	Coupled TwoTheta/Theta scan

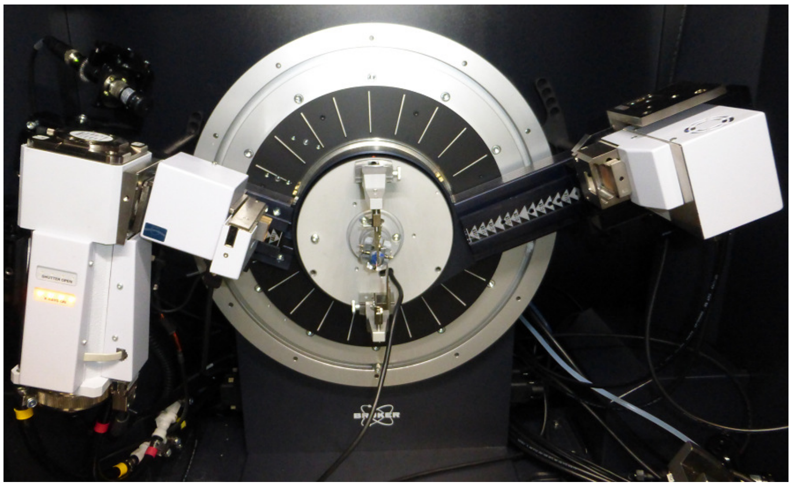
This configuration is used primarily for irregular shaped polycrystalline samples that cannot be ground to powders and that do not have a flat surface to measure in Bragg-Brentano geometry, e.g. rocks, machine parts, museum artifacts, forensic evidence, etc

It is identical to the GID configuration but coupled TwoTheta/Theta scans are used. Several mm height differences can be measured without shifts in peak positions. For very low density samples this configuration can also be advantageous to get sharper diffraction peaks than with Bragg-Brentano without the peak asymmetry caused by sample penetration.

Advantages: non-destructive technique that can be used for phase identification.

Disadvantages: Slower measurement because the LynxEye detector is used in 0d mode. Angular resolution is limited by divergence of equatorial Soller Slits (expected peak width approx. 0.2° FWHM).

Capillary Measurements

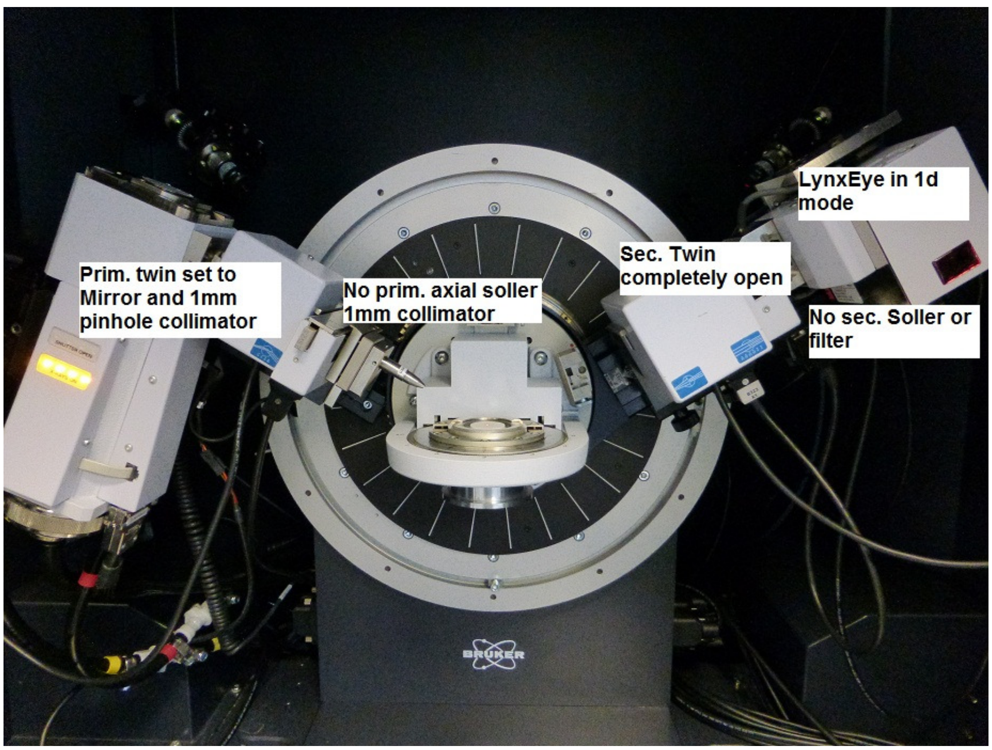


Primary Twin Optic	Set to Goebel Mirror
Radiation Safety slit	1mm for full beam, 0.2 or 0.6mm for narrower beam and better resolution
Primary Axial Soller	2.5°
Secondary Twin Optic	Opened to maximum or completely removed
Slit Mount	None
Secondary Axial Soller	2.5°
Filter	None
LynxEye detector	1D mode set to max opening
Scan Type	Option 1: Coupled TwoTheta/Theta scan, if the top knife edge is mounted above the capillary as in image above Option 2: 2Theta scan with Tube parked at 0, if the top knife edge is mounted offset

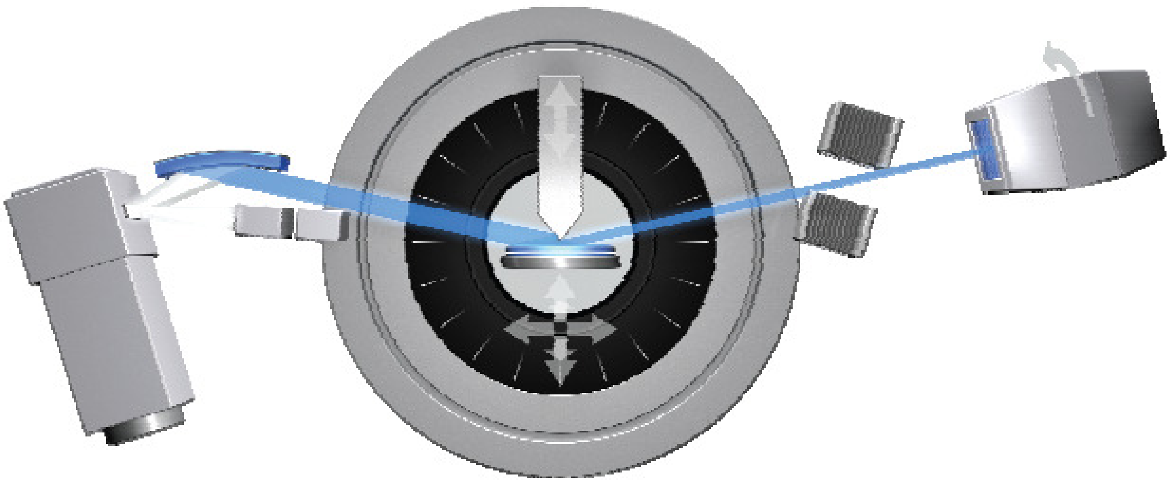
Two knife edges come with the capillary stage, which are both mounted offset from the goniometer center above and below the capillary. This makes it easier to mount and demount capillaries, but it will cut the incident beam incident beam intensity, if coupled twotheta/theta scans are used. For scan ranges up to approx. 70 degrees, this issue can be avoided by positioning the tube at 0 degrees and using 2thetascans. For ranges up to higher angles the spacer of the upper knife edge can be removed so that the beam knife is directly above the capillary (see image above). In this configuration, normal locked coupled scan can be used up to high angles without cutting the incident beam intensity.

Micro Diffraction or Texture measurements

Primary Twin Optic	Set to Goebel Mirror
Radiation Safety slit	Pinhole collimator 1mm or 0.5 mm, larger for
Primary Axial Soller	Removed
Collimator mount with pinhole collimators	Available Options: 2mm, 1mm, 0,5mm, 0.3mm, 0.1mm
Secondary Twin Optic	Opened to maximum or completely removed
Slit Mount	None
Secondary Axial Soller	None
Filter	None
LynxEye detector	1D mode set to max opening
Scan Type	Coupled TwoTheta/Theta scan



Reflectometry (XRR)

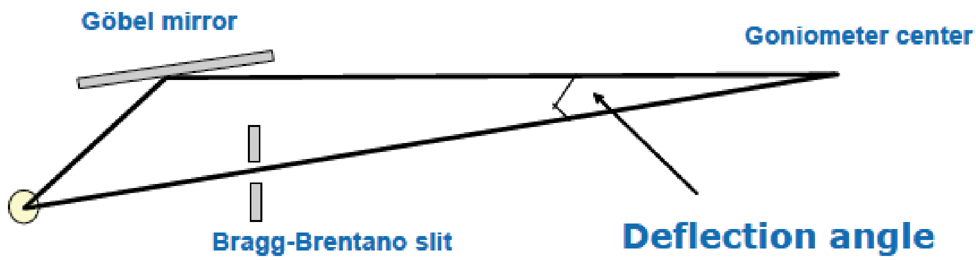


Primary Twin Optic	Set to Goebel Mirror
Radiation Safety slit	1 mm, 0.6mm or 0.2mm
Primary Axial Soller	2.5°
Secondary Twin Optic	Set to Motorized slit: Slit width 0.2mm
Slit Mount	0.1 or 0.2mm
Secondary Axial Soller	2.5°
Filter	None, possibly one absorber foil to avoid detector saturation at low angles
LynxEye detector	0D mode mounted 90° turned
Scan Type	Offset coupled TwoTheta/Theta after the sample offset was determined using a rocking curve

For this mode the detector should be turned 90° for a higher linear range. Nevertheless an absorber is usually necessary for the low angle region. Depending on the sample these measurements are often collected in multiple ranges from 0.1° to perhaps 10° 2theta.

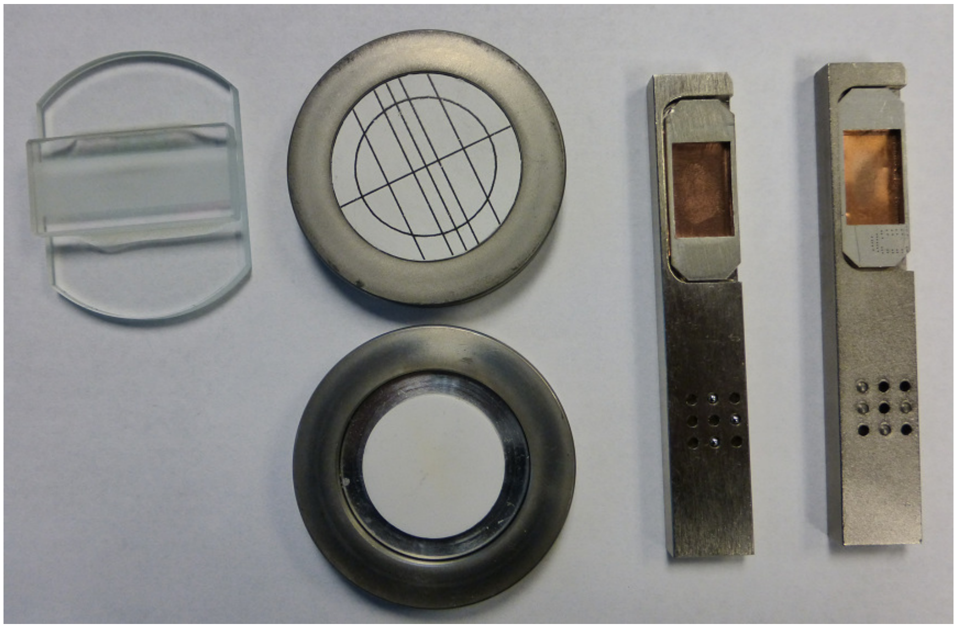
System Alignment

The goal of the system alignment is to adjust all reference values for motorized drives so that the X-ray goes through the center of the goniometer for Bragg-Brentano geometry. Additional optics with different take-off angles that are close to zero (typically below +-1 such as a Goebel mirror) can be adjusted using deflection angles, which can be associated with any optic in the configuration plugin.



To get a system with twin-twin optic aligned the tools in the image below are necessary.

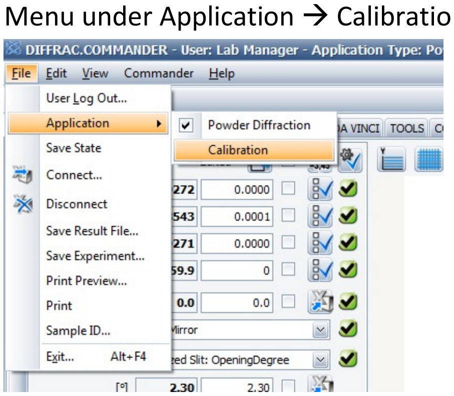
1) A glass slit which mounts in the sample cup and defines the reference plane at theta=0 and 2 theta=0
2) A fluorescent screen to check the mirror reflection
3) 0.1mm and 0.2mm copper foil absorbers
4) The SRM1976a Corundum plate



Alignment steps:

1) Göbel mirror optimization:
   1.1 Optimize the mirror intensity by adjusting the beam focus height, check mirror intensity and width of reflection
   1.2 Check mirror quality (intensity and width of reflection)
   1.3 Check, if the radiation safety slit is centered for all slits. If not, adjust height of Goebel mirror and repeat step 1.1
   1.4 Visually check with fluorescent screen that the Goebel mirror reflection is within 1mm of the center. If not adjust beam rotation on DOF
2) Goniometer reference angle Determination
   2.1 Primary Goniometer reference determination
   2.2. Secondary Goniometer reference angle determination
3) Centering the LynxEye detector strips to the slit of the secondary optic bench
4) Determine deflection angle for primary Twin Mirror
5) Alignment of Mirror primary beam stop
6) Divergence slit alignment
   3.1 Slit position alignment - centering the Divergence slit
   3.2 Opening reference determination – Determination of “Var. Closed “property
7) Anti-Scatter slit alignment
   4.1 Slit position alignment- centering the Anti-Scatter slit
   4.2 Opening reference determination –Determination of “Var. Closed “property
8) Equatorial Soller Slit alignment
   8.1 Soller Alignment
   8.2 Deflection angle determination
9) Calibrate LynxEye Detector in Detector Plugin

Most of the required scan types are only possible with a point detector or the LynxEye detector in 0d mode. To get the correct settings for the stepper motors in the primary and secondary Twin optic the scan types are only available in Calibration mode, which is accessible in the Diffrac Commander File Menu under Application → Calibration.



1. Mirror alignment

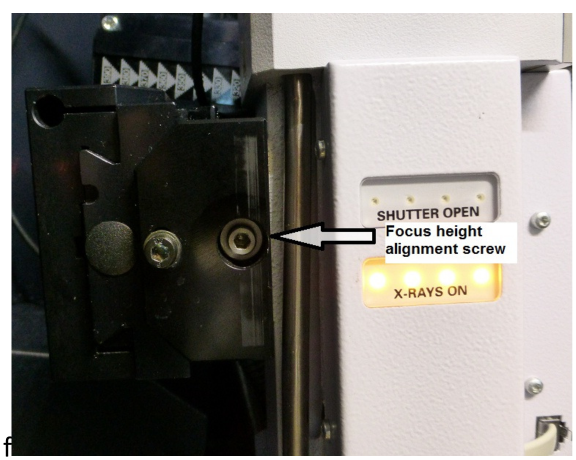
The Goebel mirror of the primary twin optic was aligned in the factory and in most cases no alignment of the tilt screw should be necessary. Optimizing the focus height translation should be tried first before changing any alignment screws on the mirror.

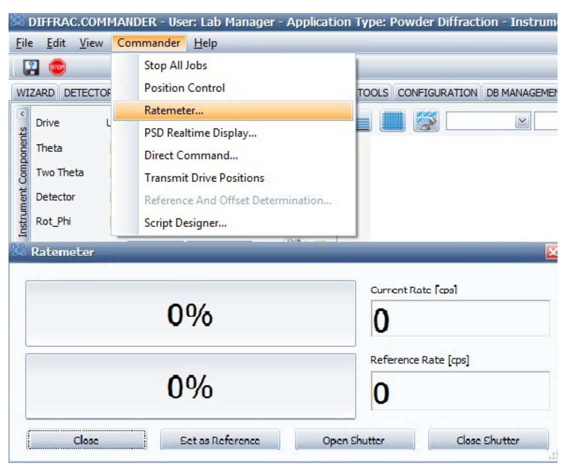
1.1 Optimizing of Beam focus height

An alignment of the X-ray tube focus position may be necessary under the following conditions:
a.) First set up of the D8 at customer side
b.) Change of the adapter plate
c.) Change of the x-ray tube
d.) Change from point focus to line focus

To check the mirror intensity, perform a TwoTheta scan through the direct beam with 0.2mm Cu foil absorber and check the intensity. If it is lower than 200000 cps park the detector in the direct beam and optimize the Intensity using the rate meter and the focus height alignment screw

Generator	40kV40mA
Start position	Tube at 0°, Detector at 0°
Primary Optic	Twin Primary set to Goebel Mirror
Radiation safety slit	None
Primary Axial Soller Slit	None
Sample	None
Secondary Optic	None
Secondary Slit	Absorber 0.2mm Cu foil
Secondary Axial Soller Slit	None
Detector	LynxEye 0D with 6mm opening
Measurement	TwoTheta scan from -1 to 1, increment 0.01deg, step time 0.1s
Evaluation	Determine the peak intensity (peak max) Criterion: I>200.000cps Park your detector at the maximum and optimize intensity with focus height alignment screw and Ratemeter





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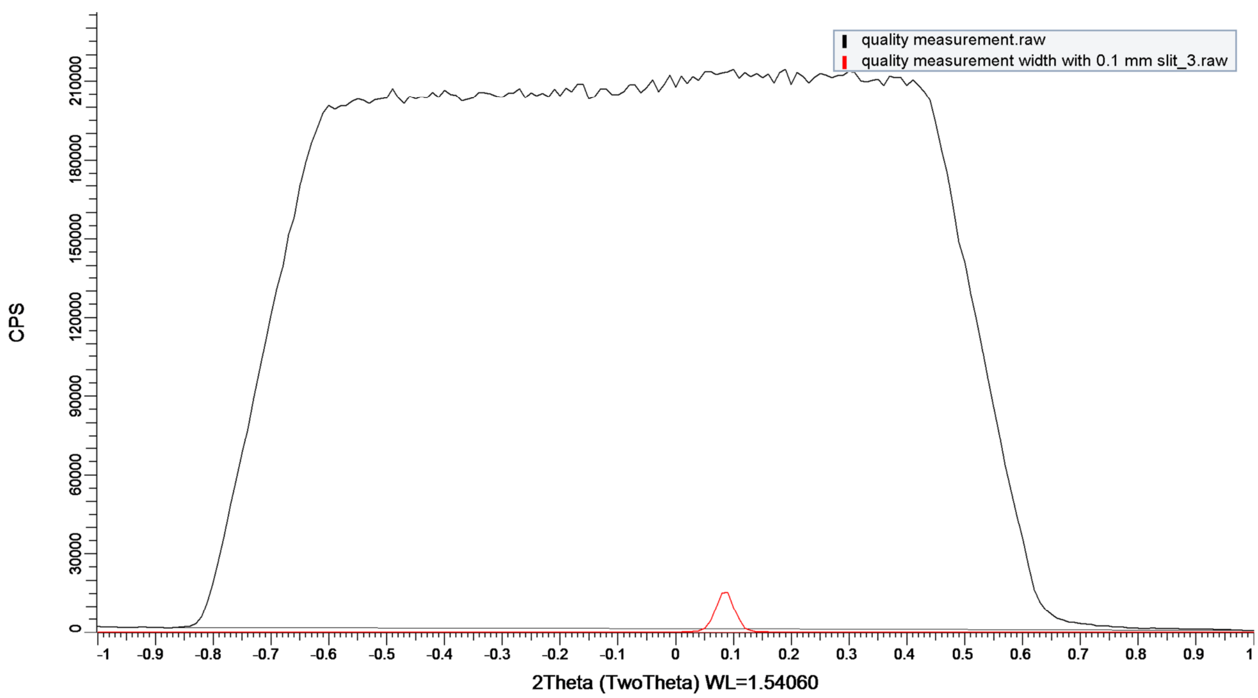
1.2 Quality measurement

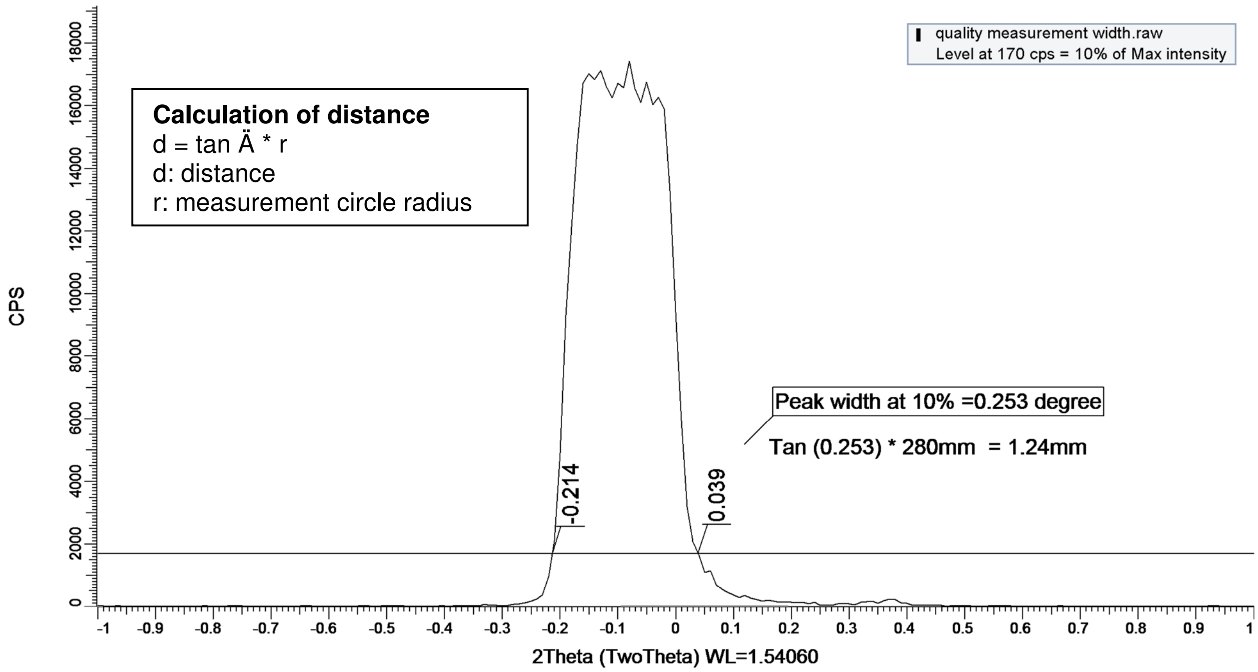
To verify the quality of the mirror alignment the mirror reflection should be measured directly using two absorber foils (0.2mm copper foils). All slits and Soller Slits should be removed and a TwoTheta scan through the mirror reflection is performed.

Generator	40kV40mA
Start position	Tube at 0°°, Detector at 0°
Primary Optic	Twin Primary set to Goebel
Radiation safety slit	None
Primary Axial Soller Slit	none
Sample	Empty
Secondary Optic	Empty
Secondary Slit	Absorber with 0.2 mm Cu foil
Secondary Axial Soller Slit	None
Detector	LynxEye 0D with 1) 6mm opening for intensity check 2) 0.075mm opening for peak width check
Measurement	TwoTheta continuous scan from -1° to 1° with 0.01 steps and 0.1sec/step
Evaluation	Determine the peak intensity (peak max) Criterion: I>200.000cps
	If the Count rate is insufficient, adjust focus height If that not help, try optimizing Bragg angle or Goebel mirror If still not enough counts consider a new tube, if it is older



1.2.2 Quality measurement (Peak Width)

To determine the width of the reflection, collect a scan with 0.075mm opening in the LynxEye detector. Criteria: The FWHM at 10% of the intensity should be larger than 0.8mm



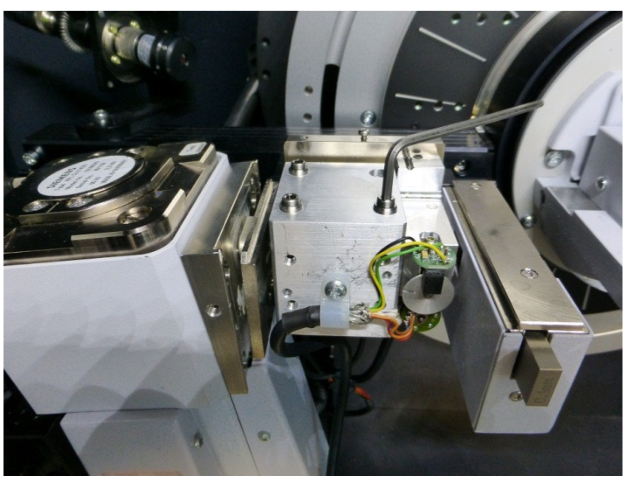
1.3 Radiation safety slit alignment

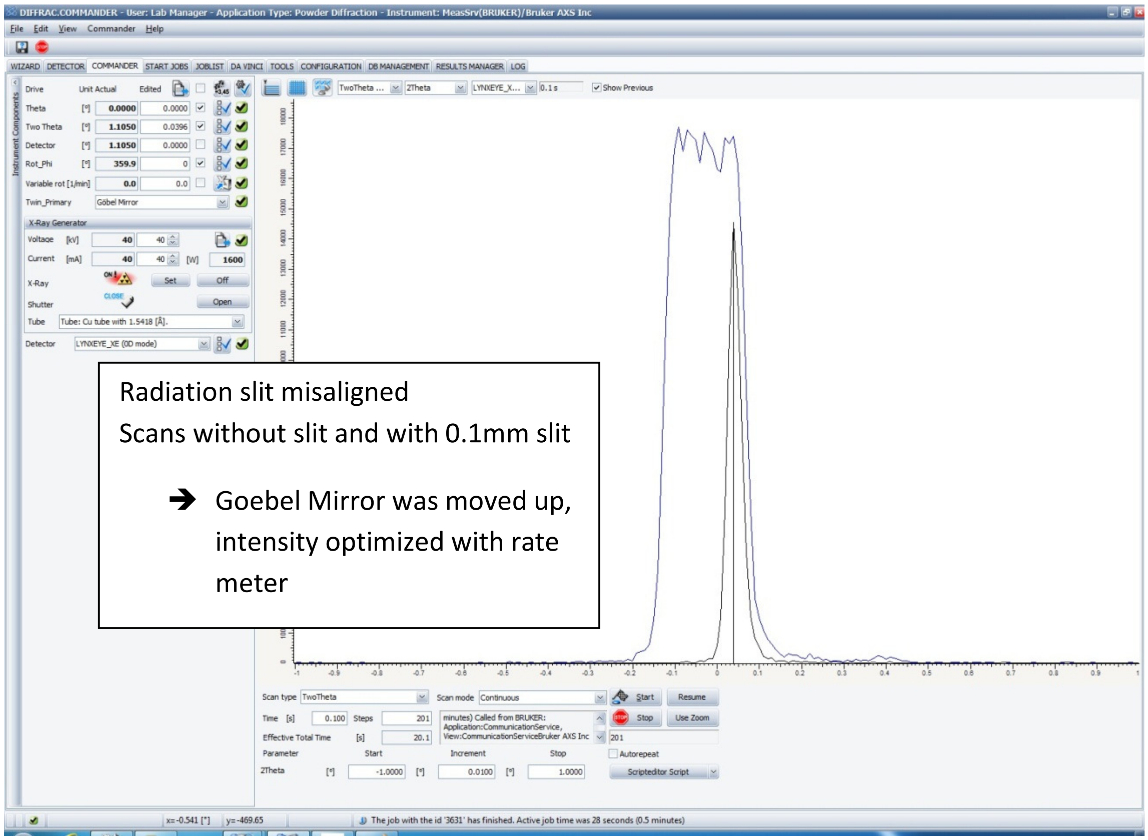
This slit position cannot be mechanically aligned. Instead the mirror height has to be adjusted to this slit. If it is not perfectly centered to the beam, intensity loss will occur when small slits are used with the mirror. This is important for SAXS measurements, GID measurements at low incidence angles or any measurement where a narrow beam is needed.

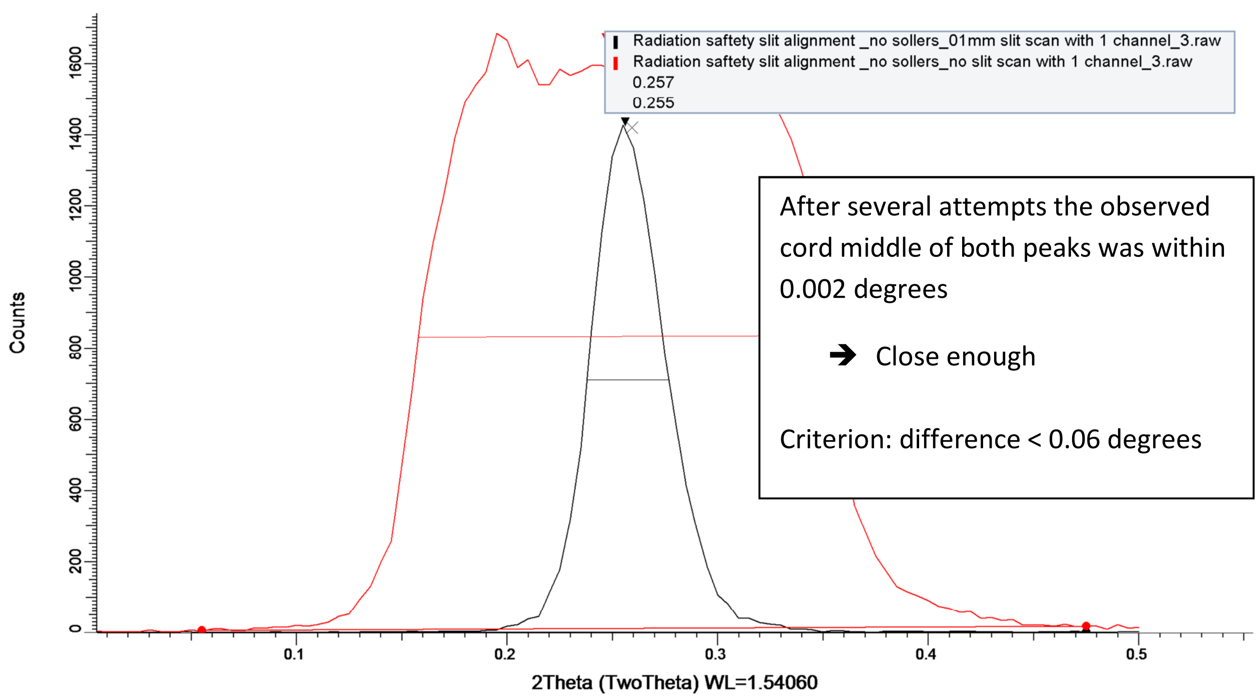
To check the factory alignment, perform a TwoTheta scan through the direct beam without a slit and with a 0.1mm slit. If the observed peak center do not line up within 0.06° the mirror height needs to be adjusted to the radiation slit assembly.

Generator	40kV40mA
Start position	Tube at 0°°, Detector at 0°
Primary Optic	Twin Primary set to Goebel
Radiation safety slit	1) None 2) 0.1mm slit
Primary Axial Soller Slit	None
Sample	Empty
Secondary Optic	Empty
Secondary Slit	Absorber with 0.2 mm Cu foil
Secondary Axial Soller Slit	None
Detector	LynxEye 0D with 0.075mm opening
Measurement	TwoTheta continuous scan from -1° to 1° with 0.01 steps and 0.1sec/step
Evaluation	Determine the peak gravity (cord middle at 10% height) for both measurements
Criterion	If |x-xo| > 0.06deg which corresponds to 0.3mm, align all GM alignment screws to new height and maximize GM Bragg screw to maximum intensity with rate meter.

If the mirror height needs to be adjusted, move all three screws approx. a quarter turn (in this example counterclockwise to move the mirror up)

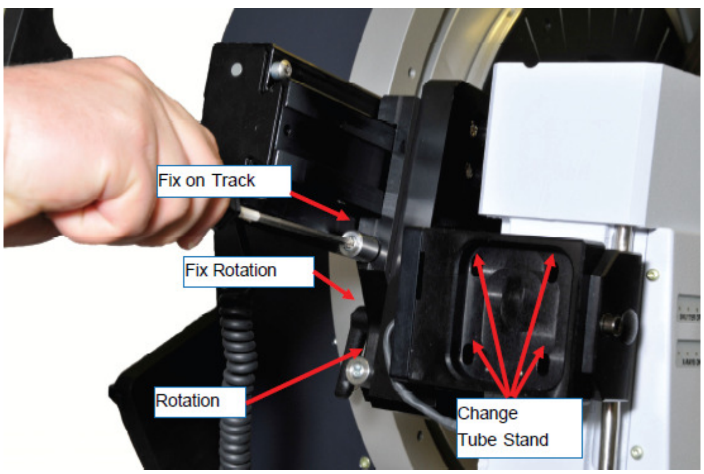


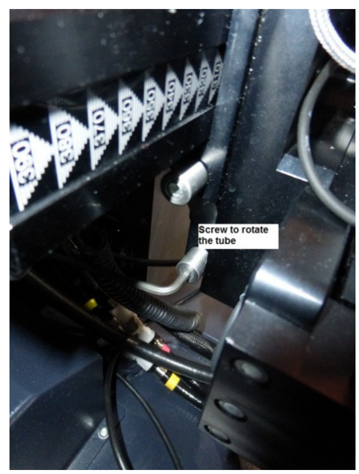

DIFFRAC.COMMANDER - User: Lab Manager - Application Type: Powder Diffraction - Instrument: MeasSrv(BRUKER)/Bruker AXS Inc
Radiation slit misaligned
Scans without slit and with 0.1mm slit
➔ Goebel Mirror was moved up, intensity optimized with rate meter
The job with the id '3631' has finished. Active job time was 28 seconds (0.5 minutes)

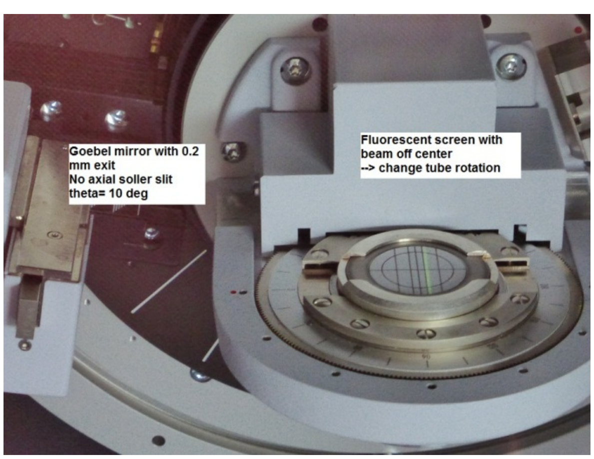

Radiation saftety slit alignment _no sollers_01mm slit scan with 1 channel_3.raw
Radiation saftety slit alignment _no sollers_no slit scan with 1 channel_3.raw
0.257
0.255
After several attempts the observed cord middle of both peaks was within 0.002 degrees
➔ Close enough
Criterion: difference < 0.06 degrees
Counts
0
200
400
600
800
1000
1200
1400
1600
0.1
0.2
0.3
0.4
0.5
2Theta (TwoTheta) WL=1.54060

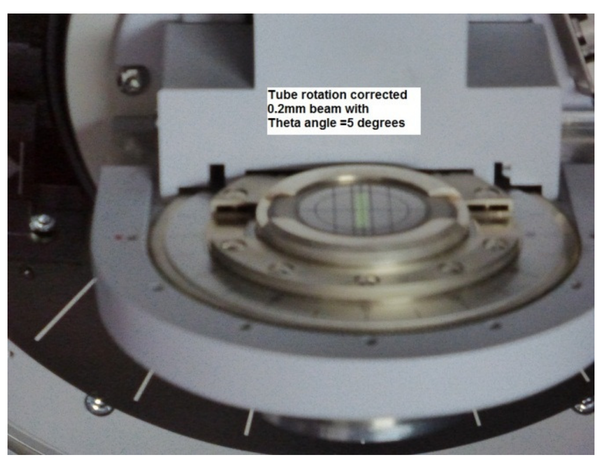
1.4 Centering the Goebel mirror by adjusting the tube rotation

Generator	40kV4mA
Start position	Tube at 5°, Detector at 0°
Primary Optic	Set to Goebel mirror
Radiation safety slit	0.2mm
Primary Axial Soller Slit	None for more intensity
Sample	Fluorescent sample
Secondary Optic	Empty
Secondary Slit	Absorber with 0.1 mm Cu foil
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 0D with 0.075mm opening
Measurement	Turn off the lights and visually check if the mirror reflection is centered on the screen
Evaluation	If beam is not centered loosen fix rotation lever and change rotation on rotation beam steering plate





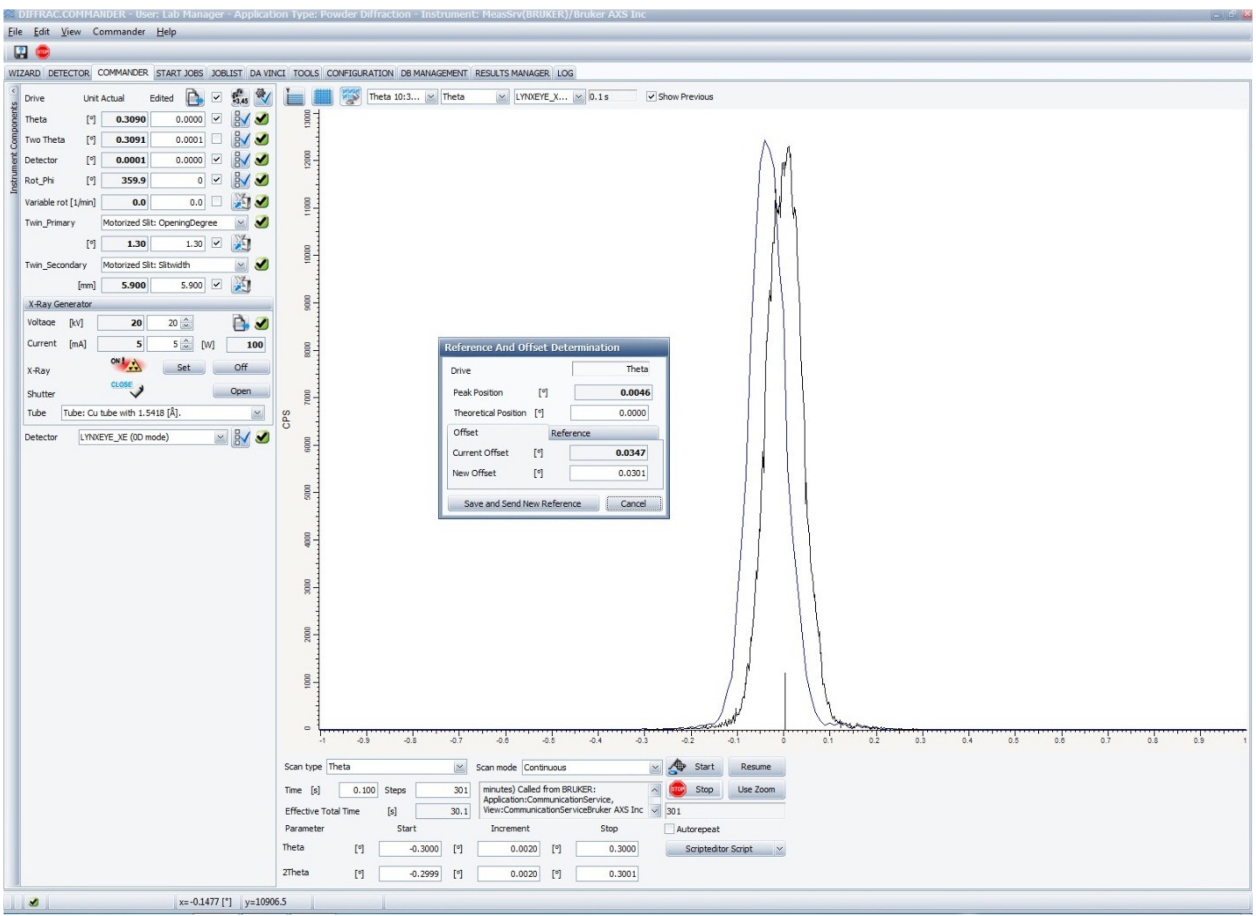




2. Goniometer reference angle Determination

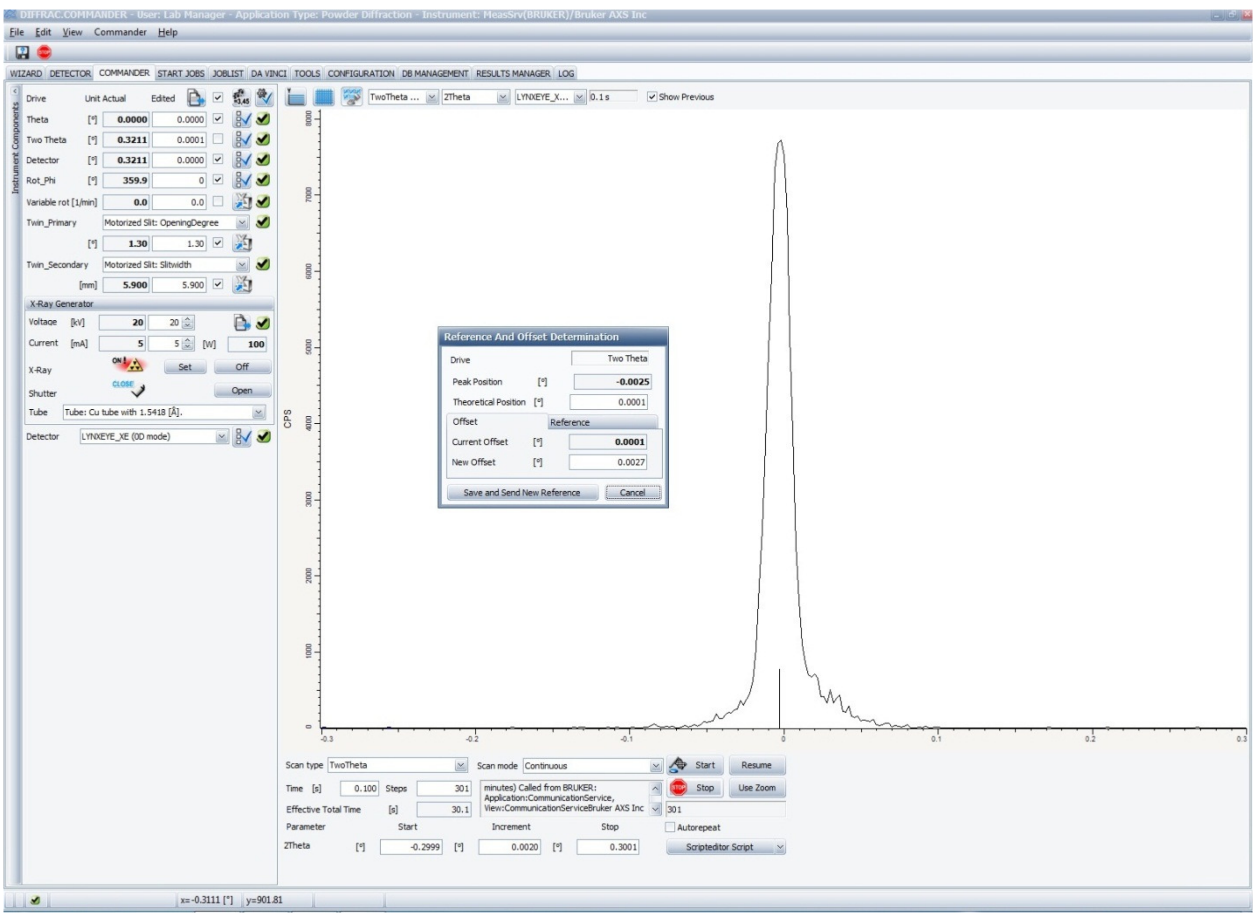
2.1 Primary Goniometer reference angle determination

Generator	20kV5mA
Start position	Tube at 0°, Detector at 0°
Primary Optic	Twin Primary set to max. opening
Radiation safety slit	None
Primary Axial Soller Slit	2.5°
Sample	Glass slit
Secondary Optic	Empty
Secondary Slit	Absorber with 0.1 mm Cu foil
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 0D with 10mm opening
Measurement	Theta continuous scan from -1° to 1° with 0.01 steps and 0.1sec/step
Evaluation	Go to COMMANDER menu and press REFERENCE AND OFFSET DETERMINATION. Criterion: If |0-X| > 0.005deg, adjust the reference position by pressing ok Repeat scan in a smaller range with small increment to verify



2.2 Secondary Goniometer reference angle determination

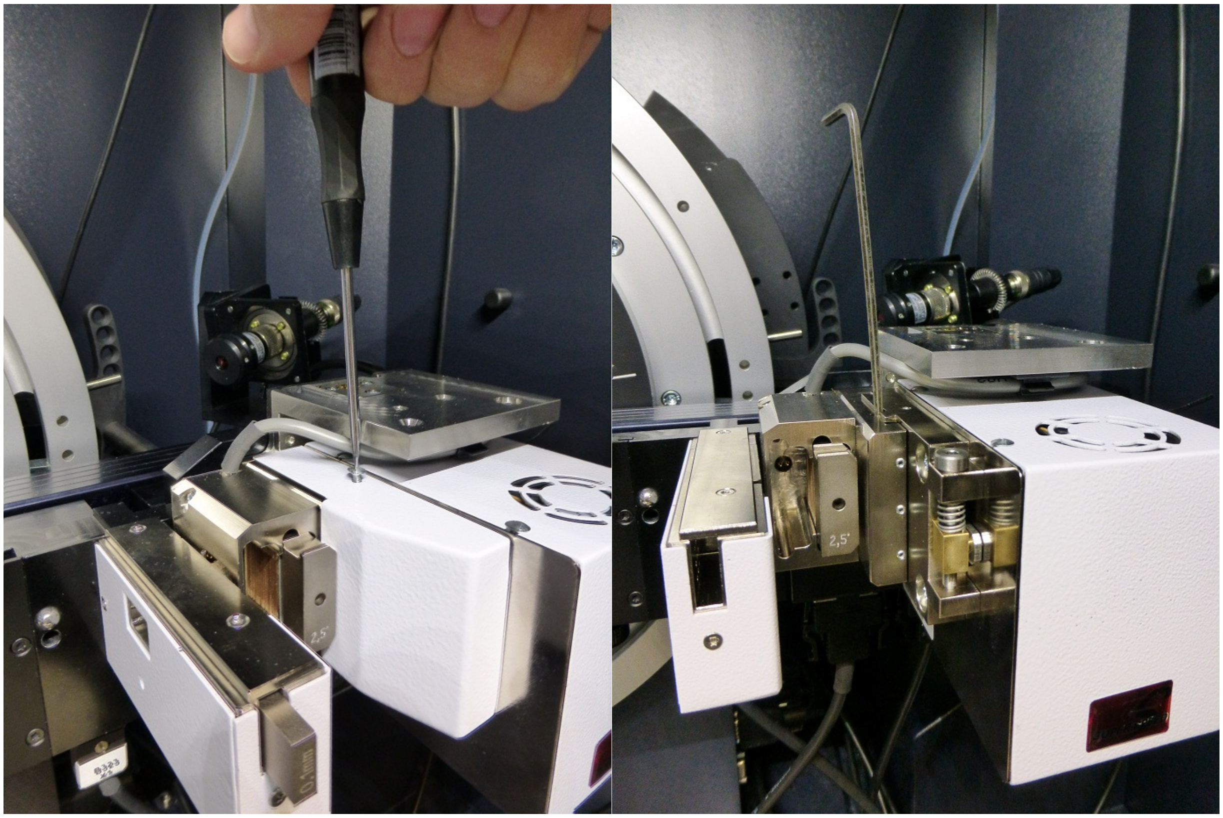
Generator	20kV5mA
Start position	Tube at 0°, Detector at 0°
Primary Optic	Twin Primary set to max. opening
Radiation safety slit	Absorber with 0.1 mm Cu foil
Primary Axial Soller Slit	2.5°
Sample	Glass slit
Secondary Optic	Secondary Twin wide open
Secondary Slit	0.1mm slit
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 0D with 10mm opening
Measurement	TwoTheta continuous scan from -1° to 1° with 0.01 steps and 0.1sec/step
Evaluation	Go to COMMANDER menu and press REFERENCE AND OFFSET DETERMINATION. Criterion: If |0-X| > 0.005deg, adjust the reference position by pressing ok Repeat scan in a smaller range with small increment to verify



3. Centering the LynxEye detector strips to the slit position of the secondary optical bench

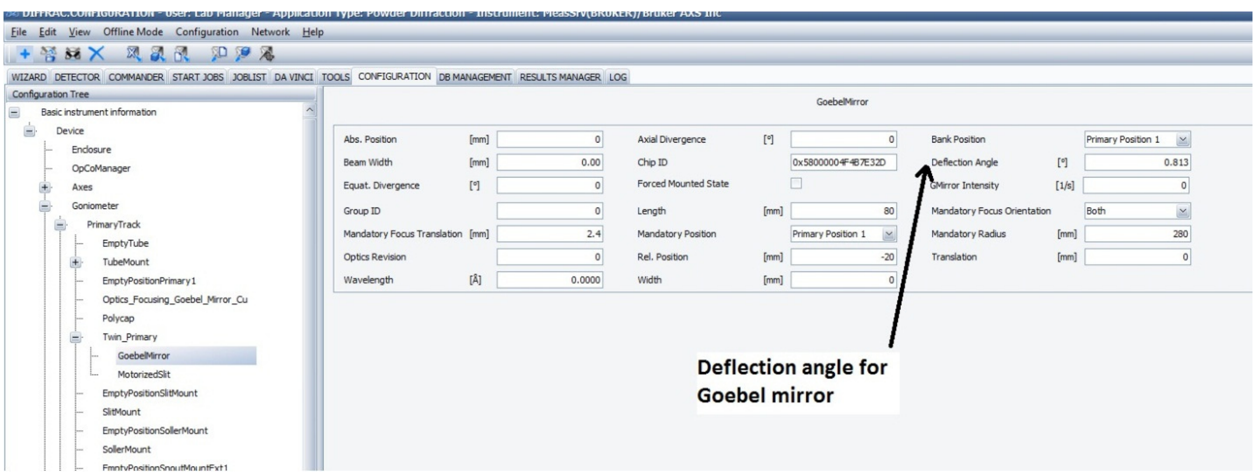
Remove the front cover of the LynxEye XE detector with a small flat head screwdriver (see image below). Adjust the position of the detector strips with an Allen wrench so that the center strip is lined up with a small slit in the secondary optical bench.

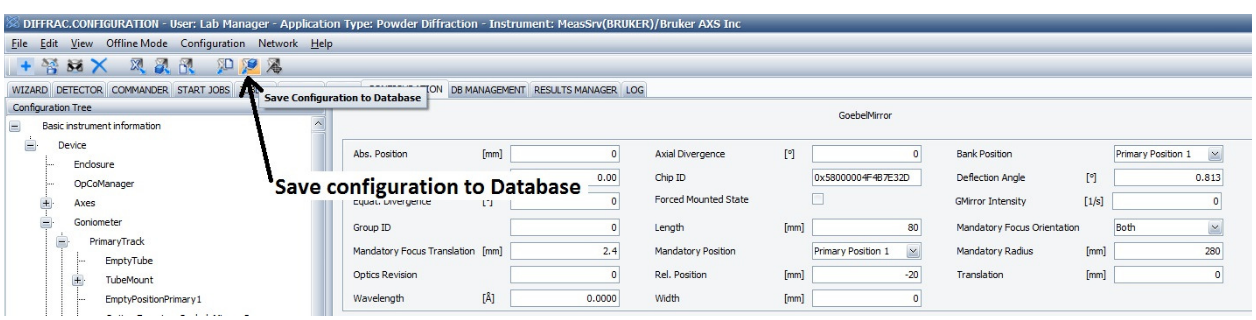
Generator	20kV5mA
Start position	Tube at 0°, Detector at 0°
Primary Optic	Twin Primary set to max. opening
Radiation safety slit	Absorber with 0.1 mm Cu foil
Primary Axial Soller Slit	2.5°
Sample	Glass slit
Secondary Optic	Secondary Twin wide open
Secondary Slit	0.1mm slit
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 0D with 0.1mm opening
Measurement	TwoTheta continuous scan from -0.3° to 0.3° with 0.002 steps and 0.1sec/step
Evaluation	Adjust strip position for maximum intensity with the rate meter in Commander Menu



4. Determine Deflection Angle for Primary Twin Goebel Mirror

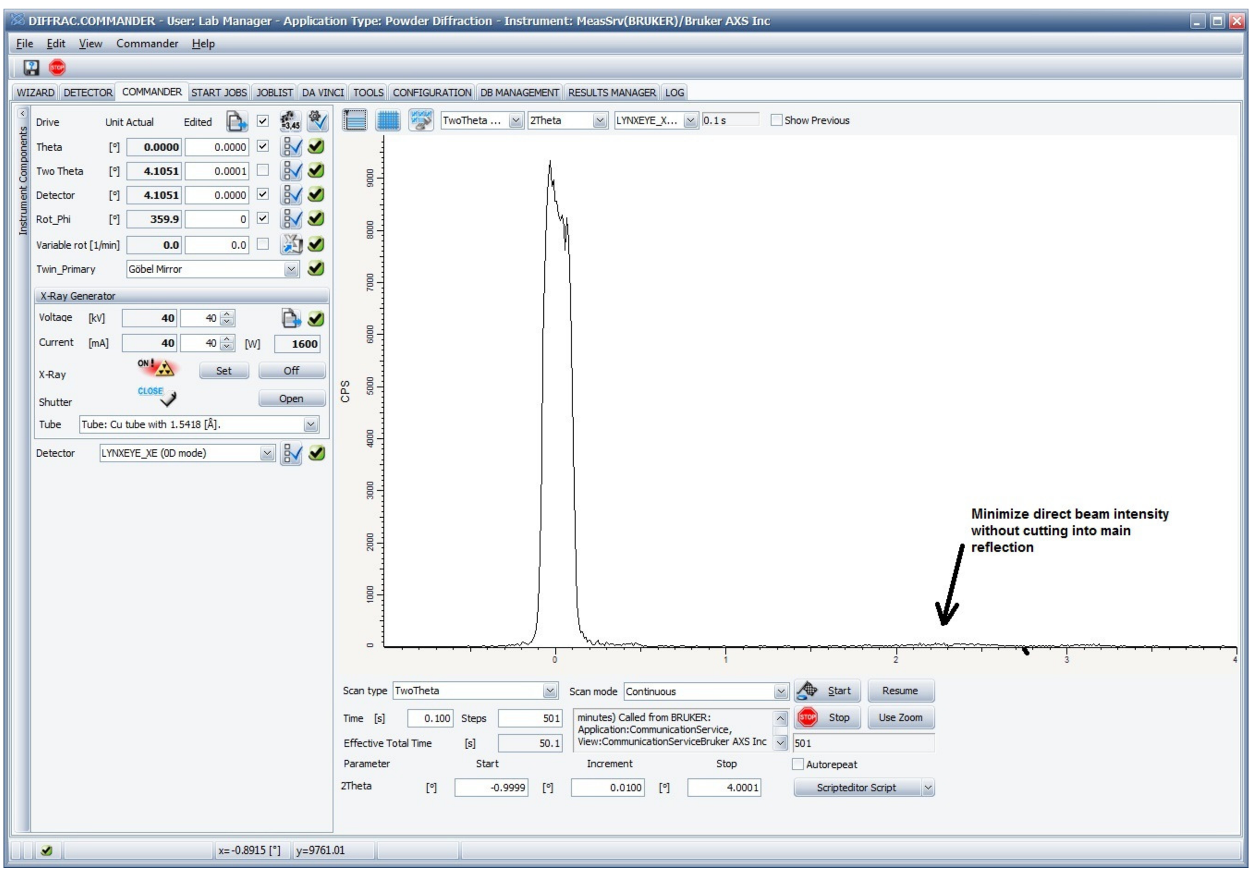
Generator	40kV40mA
Start position	Tube at 0°, Detector at 0°
Primary Optic	Twin Primary set Goebel mirror
Radiation safety slit	Absorber with 0.2 mm Cu foil
Primary Axial Soller Slit	2.5°
Sample	No sample
Secondary Optic	Secondary Twin wide open
Secondary Slit	
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 0D with 0.075mm opening
Measurement	TwoTheta continuous scan from -1° to 1° with 0.002 steps and 0.1sec/step
Evaluation	Scan through the direct beam, evaluate peak position and correct deflection angle for primary twin optic gobel mirror in config plugin, save and activate your configuration change





5. Alignment of Mirror Primary Beam Stop

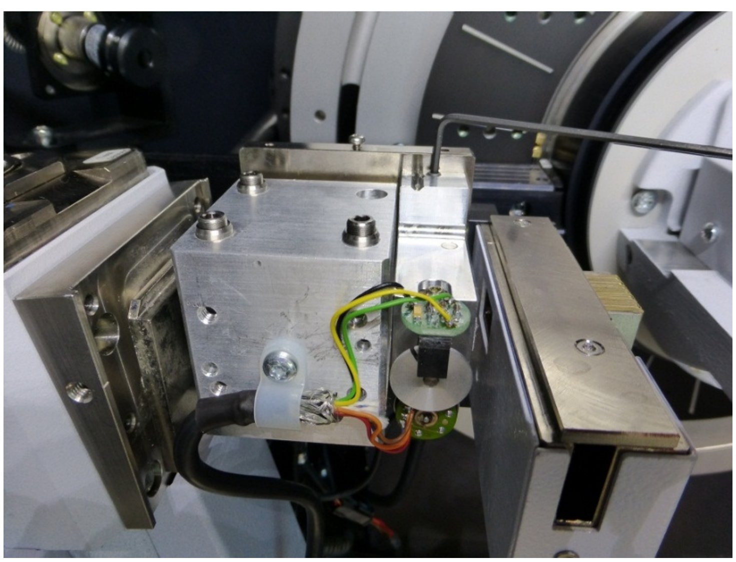
Generator	40kV40mA
Start position	Tube at 0°, Detector at 0°
Primary Optic	Twin Primary set Goebel mirror
Radiation safety slit	Absorber with 0.2 mm Cu foil
Primary Axial Soller Slit	2.5°
Sample	No sample
Secondary Optic	Secondary Twin wide open
Secondary Slit	
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 0D with 0.075mm opening
Measurement	TwoTheta continuous scan from -1° to 4° with 0.01 steps and 0.1sec/step
Evaluation	Determine the peak intensity of the direct beam at 2.5°. Align the beam stop manually to minimize direct beam without cutting into the main reflection. The beam stop is visible after removing the housing of the primary twin and has to be adjusted manually by trial and error.

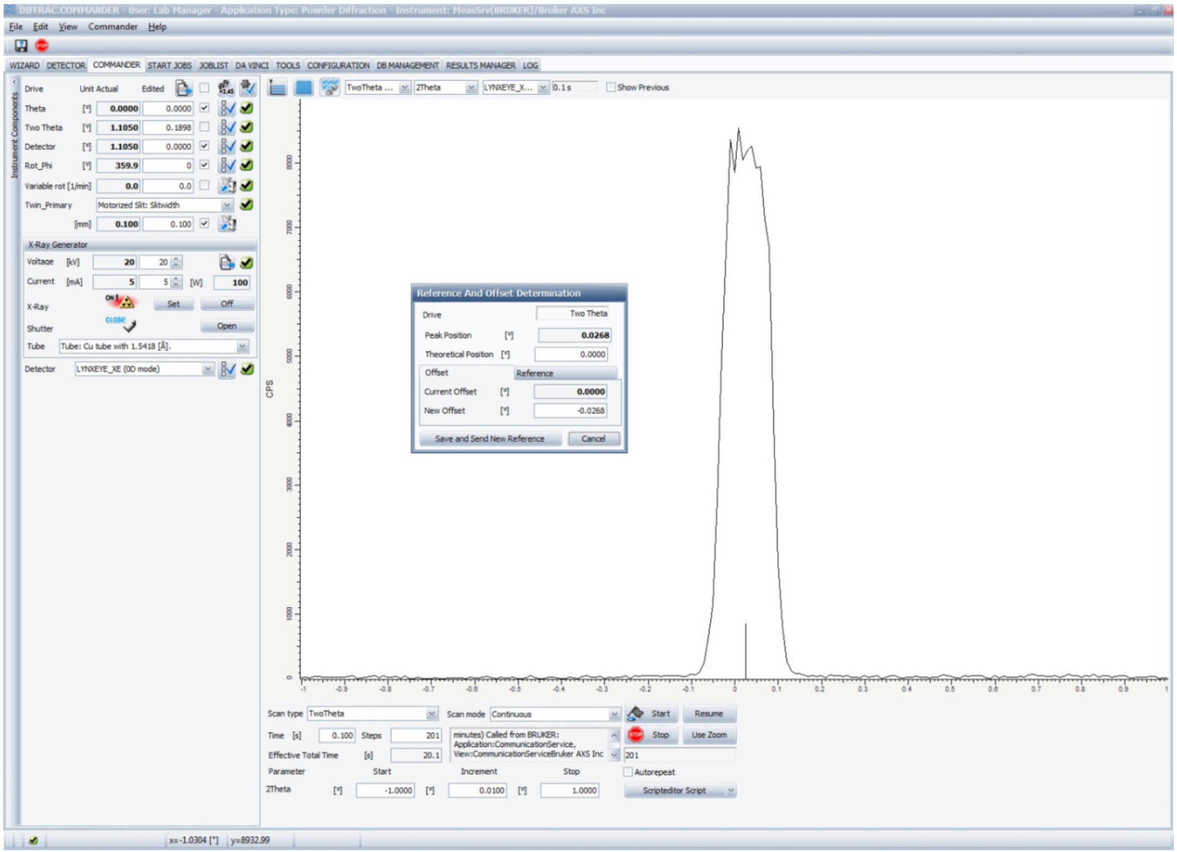


6. Divergence slit alignment

To adjust the position of the divergence slit the cover of the primary twin optic has to be taken off with a T8 Torx screw driver. There are two Allen wrench (M2) screws above and below the motorized divergence slit assembly that can be used to move the variable slit assembly. The goal is to perform a detector scan with a small divergence slit and then adjust the resulting peak position mechanically by loosening one adjustment screw and tightening the opposite screw.

Generator	20kV5mA
Start position	Tube at 0°°, Detector at 0°
Primary Optic	Twin Primary set to 0.1mm
Radiation safety slit	None
Primary Axial Soller Slit	2.5°
Sample	Empty
Secondary Optic	Empty
Secondary Slit	Absorber with 0.1 mm Cu foil
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 0D with 0.075mm opening
Measurement	TwoTheta continuous scan from -1° to 1° with 0.01 steps and 0.1sec/step
Evaluation	Go to COMMANDER menu and press REFERENCE AND OFFSET DETERMINATION. Criterion: If |0-X| > 0.01deg, align the slit manually



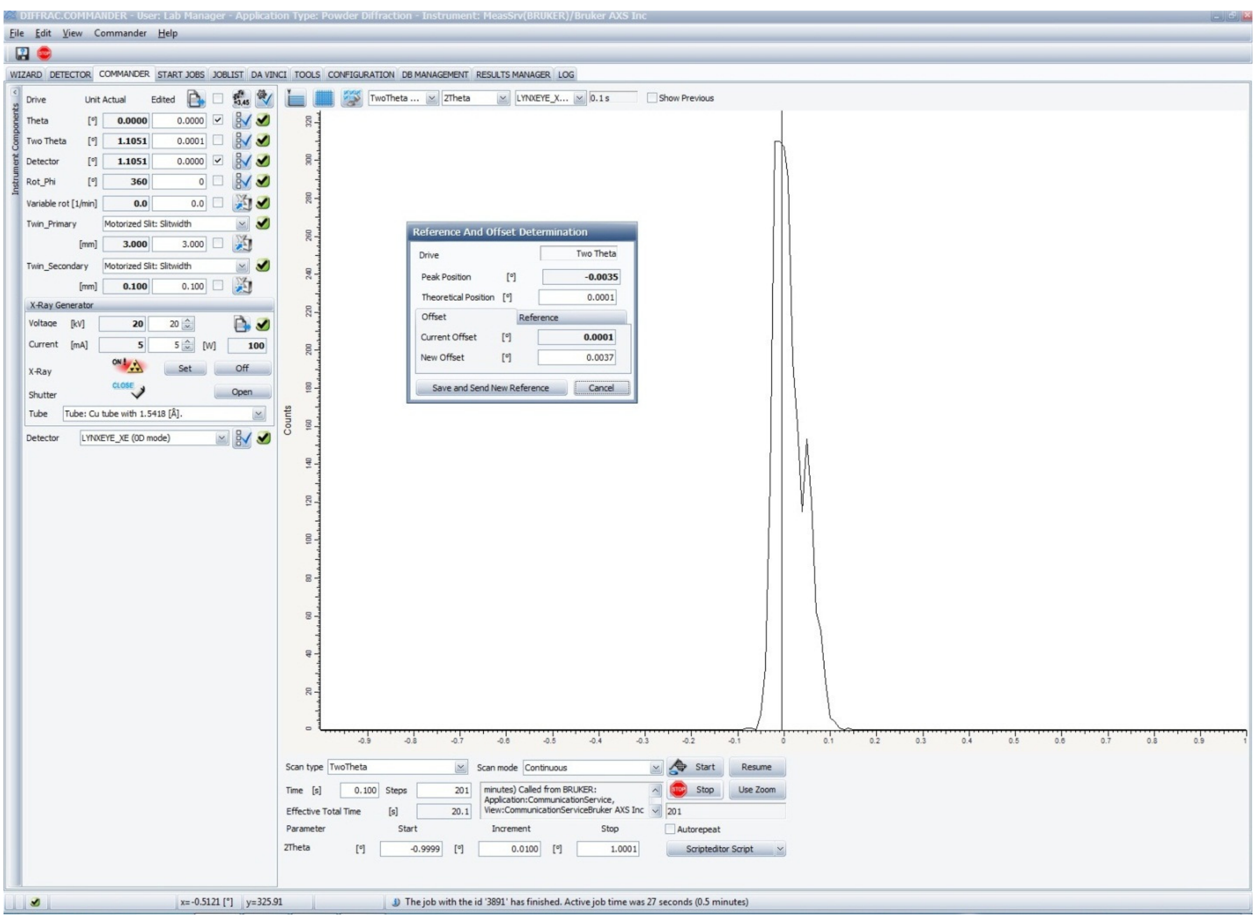


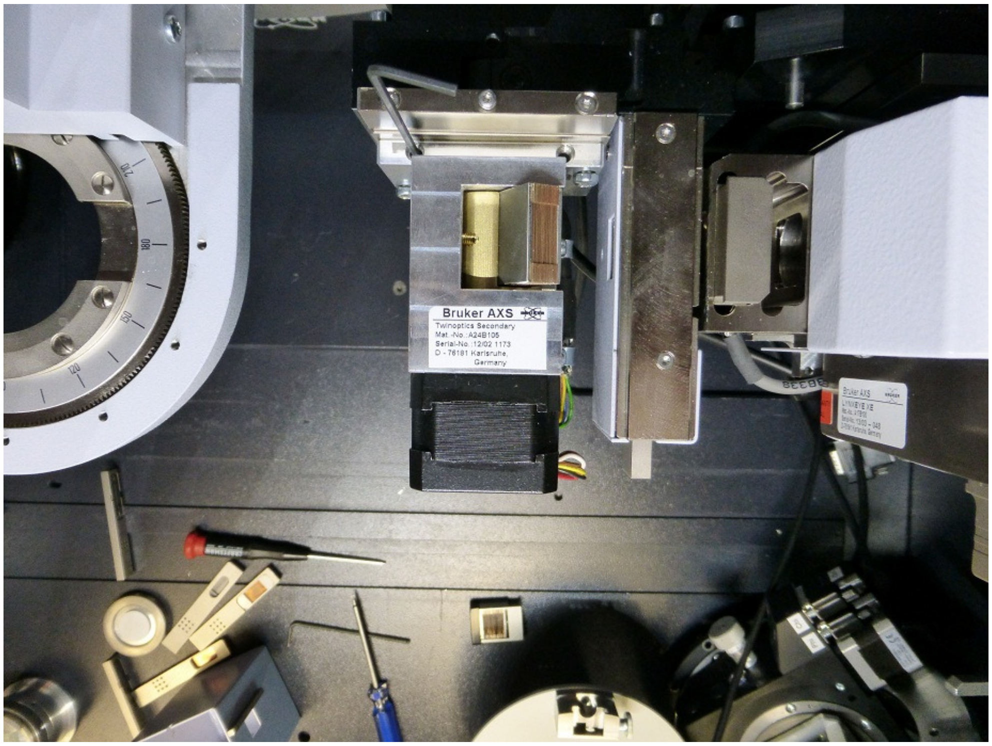
If the peak is too high as in the screenshot above, tighten the screw below the assembly and loosen the top screw slightly.

7. Anti-Scatter Slit Alignment

7.1 Alignment of Slit position

Generator	20kV5mA
Start position	Tube at 0°°, Detector at 0°
Primary Optic	Twin Primary set to 3mm
Radiation safety slit	None
Primary Axial Soller Slit	2.5°
Sample	Glass slit
Secondary Optic	Set to 0.1mm
Secondary Slit	Absorber with 0.1 mm Cu foil
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 0D with 10mm opening
Measurement	TwoTheta continuous scan from -1° to 1° with 0.01 steps and 0.1sec/step
Evaluation	Go to COMMANDER menu and press REFERENCE AND OFFSET DETERMINATION. Criterion: If |0-X| > 0.01deg, align the slit manually

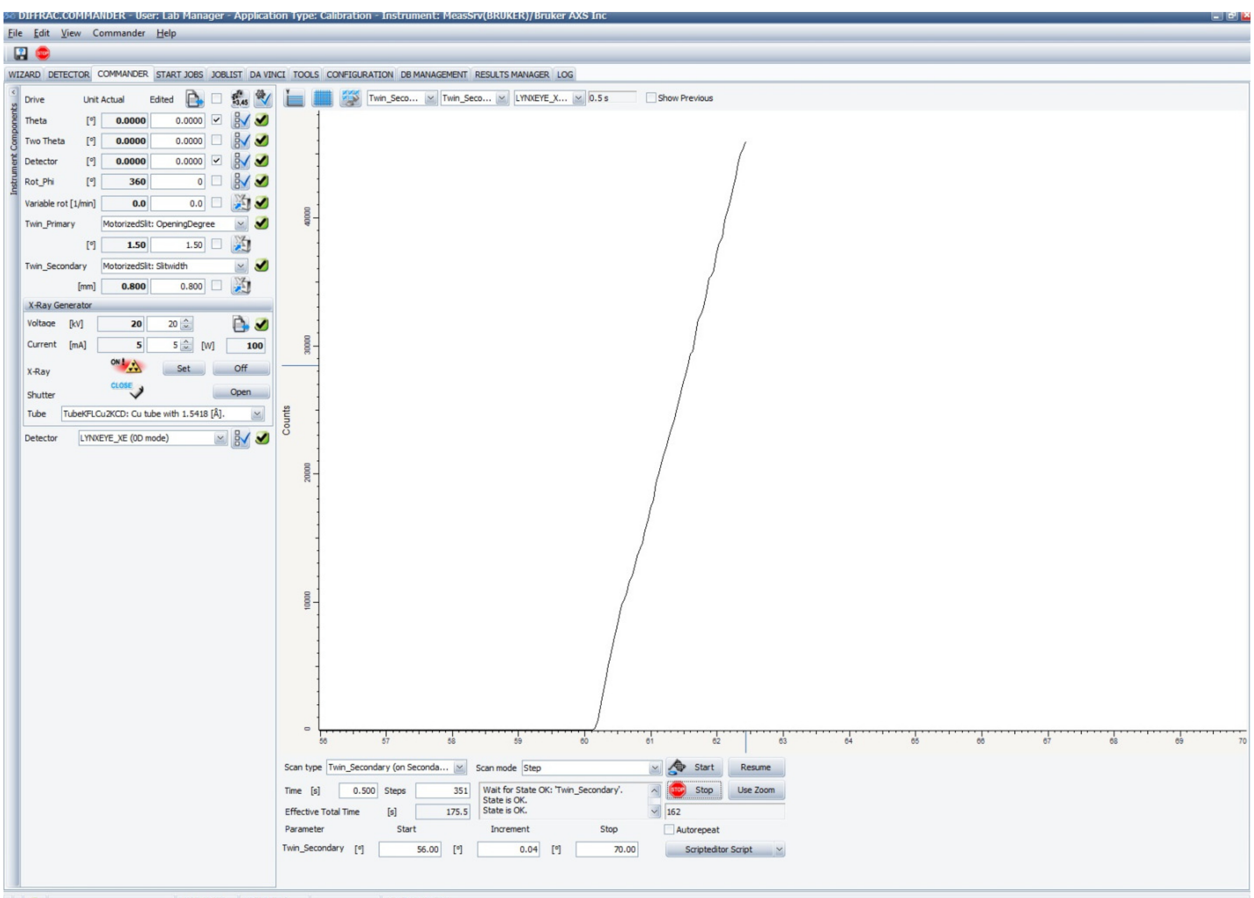



Bruker AXS
Twinoptics Secondary
Mat.-No.:A24B105
Serial-No.:12/02 1173
D - 76181 Karlsruhe,
Germany

7.2 Opening of reference angle determination

The scan type necessary for this alignment is only available in Calibration mode

Generator	20kV5mA
Start position	Tube at 0°, Detector at 0°
Primary Optic	Twin Primary set to 3mm
Radiation safety slit	None
Primary Axial Soller Slit	2.5°
Sample	None
Secondary Optic	Set to 0.1mm
Secondary Slit	Absorber with 0.1 mm Cu foil
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 0D with 10mm opening
Measurement	TWIN Antiscatter Slit motor scan from -1 ° to 1 ° around Var. Slit Closed, with 0.02 °/step and 0.5s/step
Evaluation	Determine abscissa of the measurement using the linear part of the signal slope If |x-xo| > 0.05deg, correct the var. slit open property in the CONFIGURATION plugin, save and activate the new configuration. Else continue.

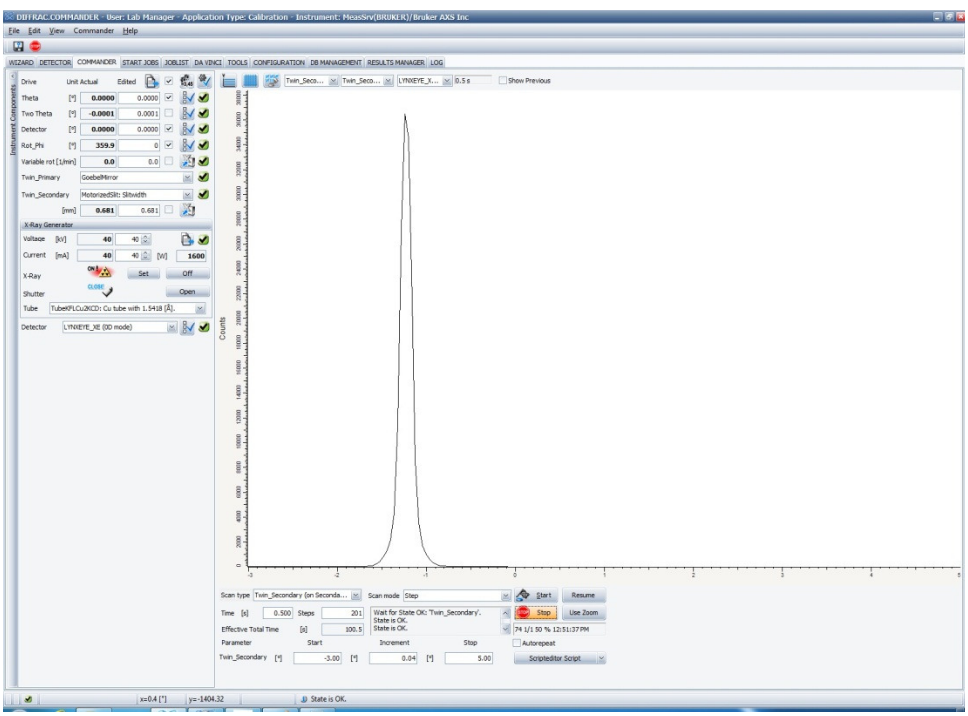


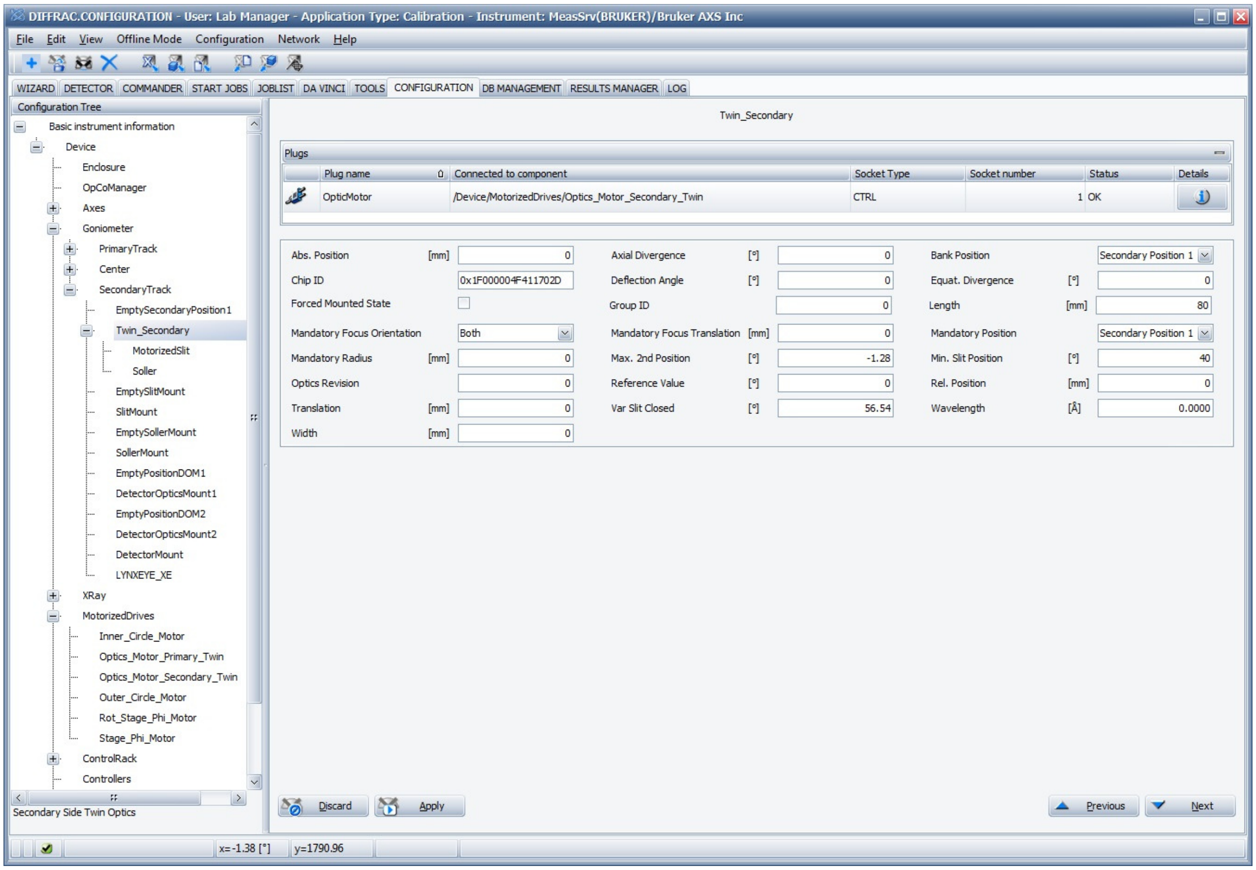
8. 1 Equatorial Soller Slit alignment

The goal of this alignment is to line up the two Soller slit parts that make up the equatorial Soller slit. This is achieved by determining the correct stepper motor position where the two Soller slit parts line up perfectly. The value is entered in the Config plugin under Secondary Twin optic as “Max. 2nd Position” property. The necessary stepper motor scan type is only available in Calibration mode.

Before starting this scan, make sure that the deflection angle for the Secondary Twin Soller was set to zero and the value was “saved and activated” before determining the Max .2nd position property.

Generator	40kV40mA
Start position	Tube at 0°, Detector at 0°
Primary Optic	Twin Primary set to Goebel Mirror
Radiation safety slit	0.2 mm Cu absorber foil
Primary Axial Soller Slit	2.5°
Sample	No sample
Secondary Optic	Soller 0.2
Secondary Slit	None
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 0D with 14mm opening
Measurement	TWIN Antiscatter Slit step scan from -1 ° to 1 ° around 2 $_{nd}$ position property, with 0.02 °/step and 0.5s/step
Evaluation	Determine peak maximum and enter the corresponding value on the x-axis into the configuration file under Twin Secondary into the Max. 2nd position



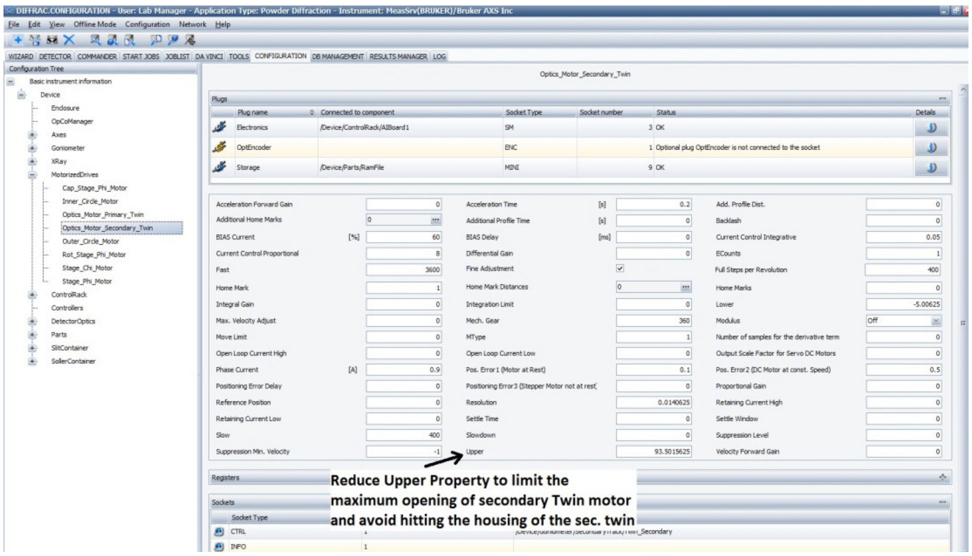


8.2 Determination of the Deflection angle for the Secondary Twin-Equatorial Soller

Generator	40kV40mA
Start position	Tube at 0°, Detector at 0°
Primary Optic	Twin Primary set to Goebel Mirror
Radiation safety slit	1mm
Primary Axial Soller Slit	2.5°
Sample	SRM1976a
Secondary Optic	Soller 0.2
Secondary Slit	None
Secondary Axial Soller Slit	None
Detector	LynxEye 0D with 14mm opening
Measurement	Coupled TwoTheta/theta continuous scan from -30° to 40° with 0.03 steps and 0.1sec/step
Evaluation	Determine angle of main corundum peak, adjust the deflection angle for secondary the Twin-Soller in the configuration plug-in.

If the variable anti-scatter slit is open to the maximum value the equatorial Soller may hit the housing of the sec. twin optic. In that case the reproducibility of the Max. 2nd position may not be given. Adjust the maximum "Upper" parameter in the configuration of the secondary twin optic to a slightly lower value to limit the maximum opening.

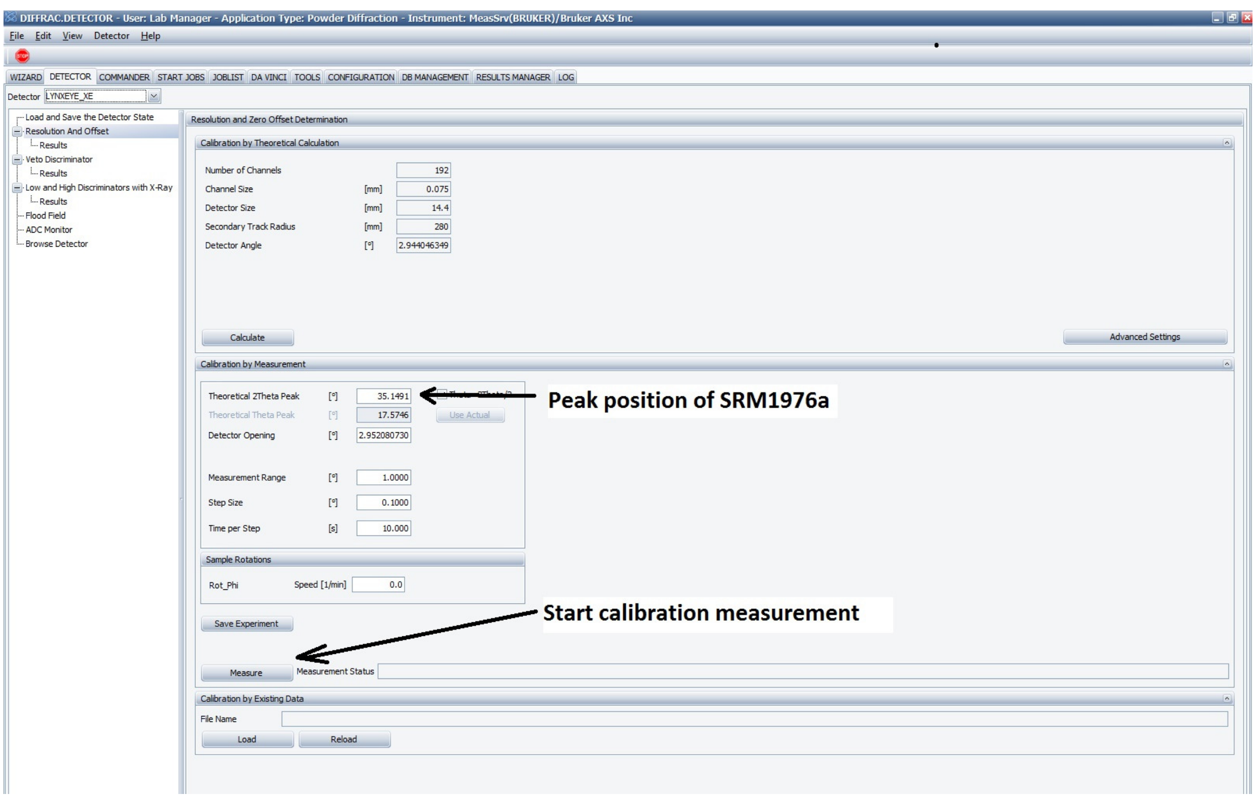
Collect multiple data sets in parallel beam (with Goebel mirror and equatorial Soller) and with Bragg Brentano and full opening to verify that both scans collect reproducible data.

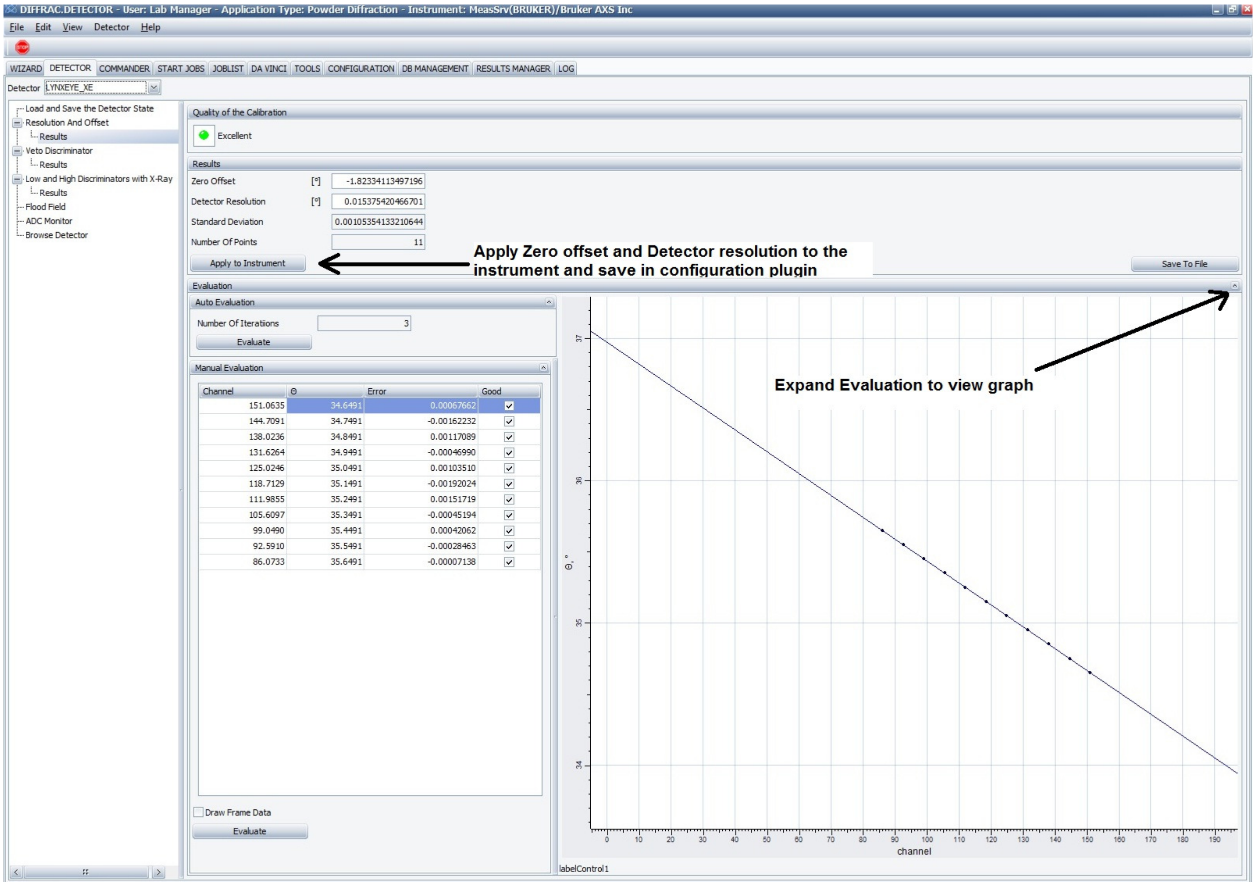


9. LynxEye Detector Calibration

So far all alignment measurements were taken with the LynxEye detector in 0d mode, essential using it as a point detector. To be able to use it in 1d mode, resolution and offset values have to be determined to account for parallax errors. This is done in the detector plugin of the Diffrac.measurement software.

Generator	40kV40mA
Start position	Changes for varies PSD fixed scans
Primary Optic	Twin Primary set to 0.3° opening
Radiation safety slit	None
Primary Axial Soller Slit	2.5°
Sample	SRM1976a
Secondary Optic	Wide open
Secondary Slit	None
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 1D with maximum opening
Measurement	Measure directly in Detector plugin
Evaluation	Wait for all pdf fixed scan measurement to finish and inspect results





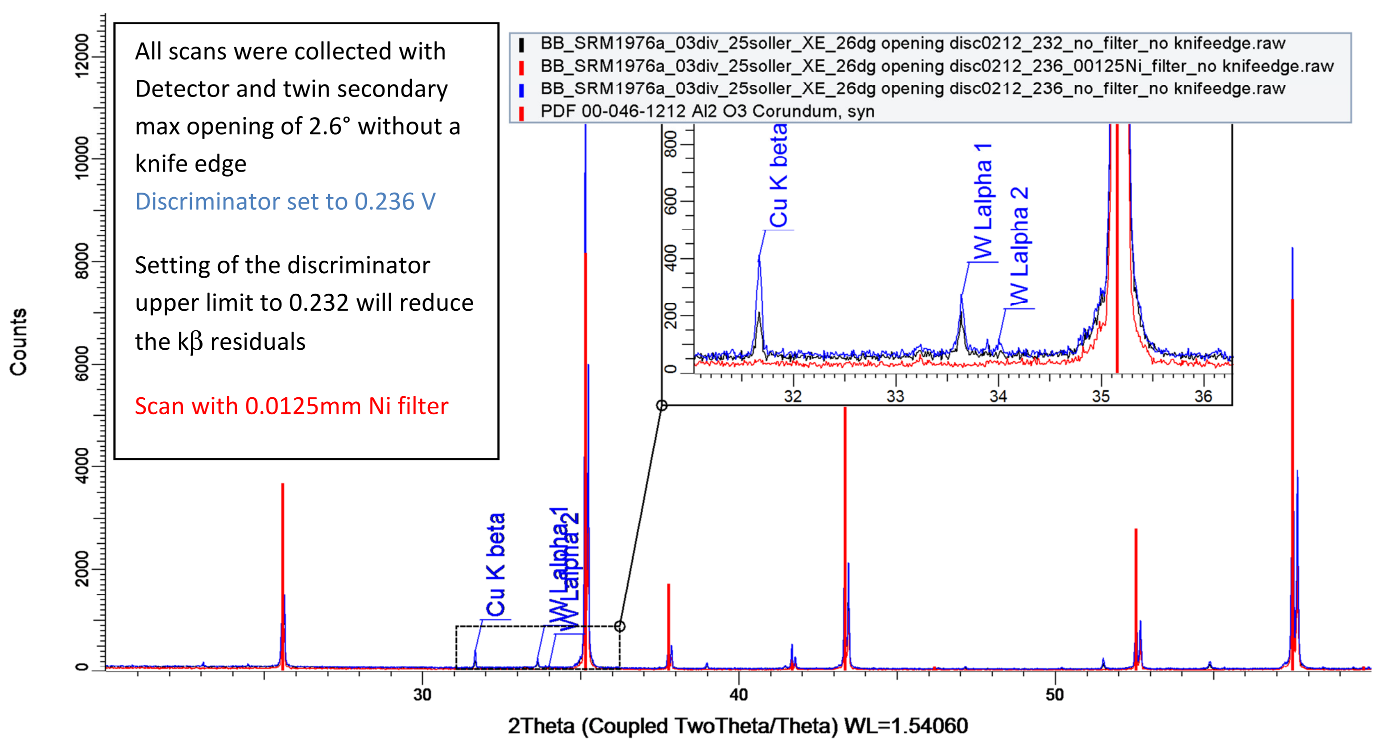
After the calibration run the results should indicate excellent quality. Apply the determined Zero Offset and resolution values to the configuration plugin. There is no download necessary.

The system is ready for some test scans on standard samples.

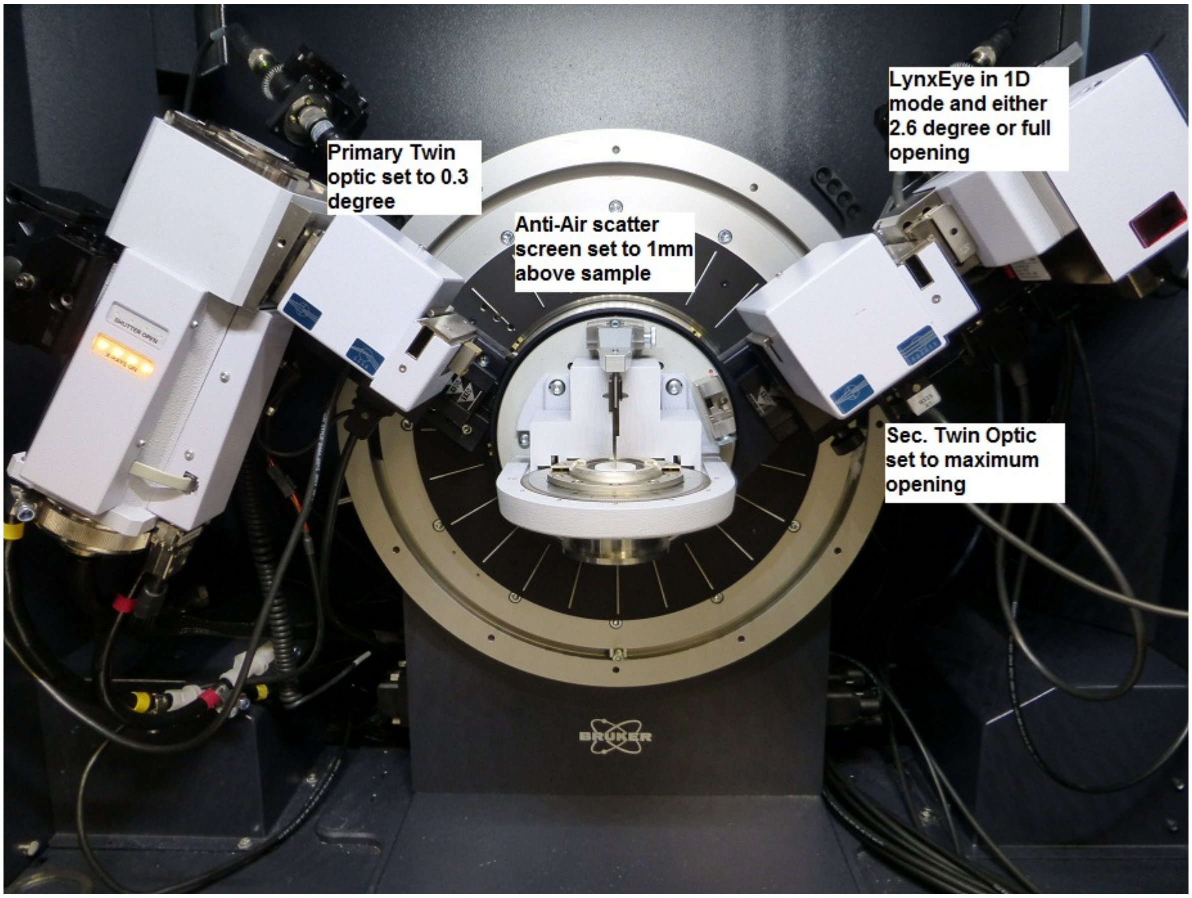
Example scans on Standard Samples

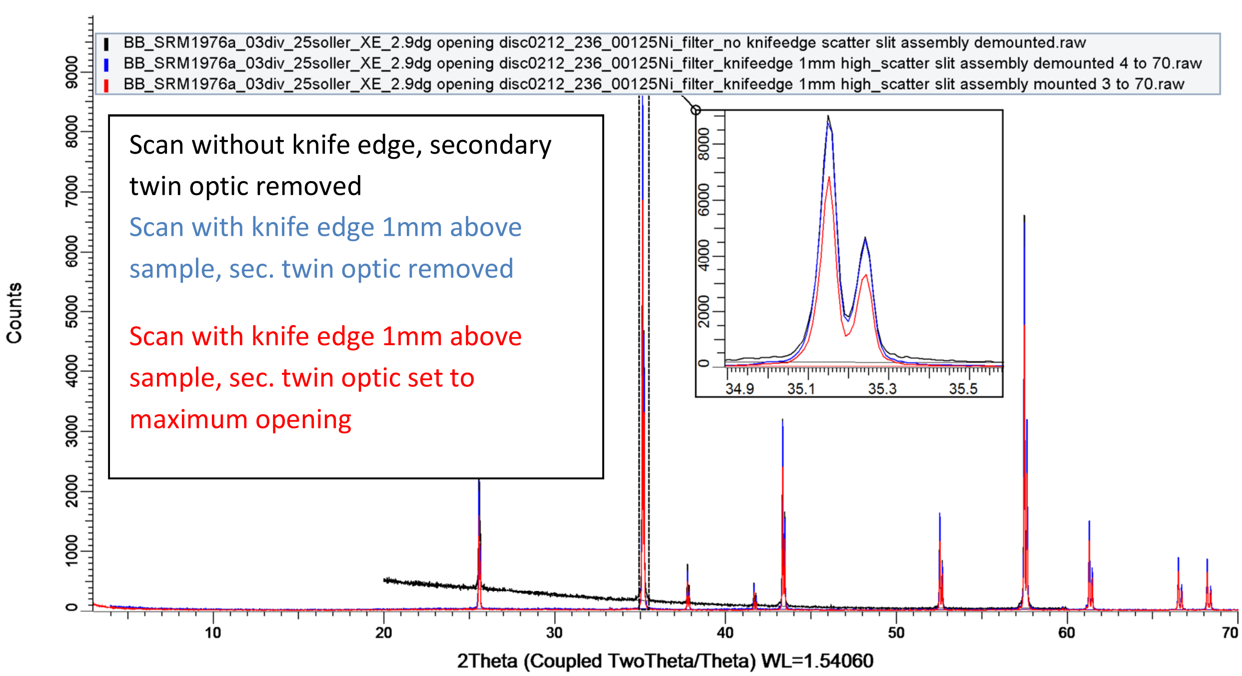
Bragg-Brentano Scans on SRM1976a

Generator	40kV40mA
Primary Twin Optic	Twin Primary set to 0.3° opening
Radiation safety slit	None
Primary Axial Soller Slit	2.5°
Sample	SRM1976a
Knife Edge	Removed or set to 1mm above the sample
Secondary Twin Optic	Set to maximum (2.6°) or completely demounted for 25% higher count rate
Secondary Slit	None
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 1D set to 2.6° or maximum angle if secondary Twin was removed
Measurement	Coupled TwoTheta/theta continuous scan from 3° to 70° with 0.01 steps and 0.1sec/step
Example scans	in subdirectory \ scans after alignment\BB



Measuring lower 2 theta angles





Typical measurement ranges for pharmaceutical samples range from 3 to 40° 2theta and for mineral samples from 3 to 70° 2theta. These ranges can be measured with the full opening of the LynxEye detector using all channels. The background from air scatter can be controlled by using the knife edge above the sample as well the secondary twin optic.

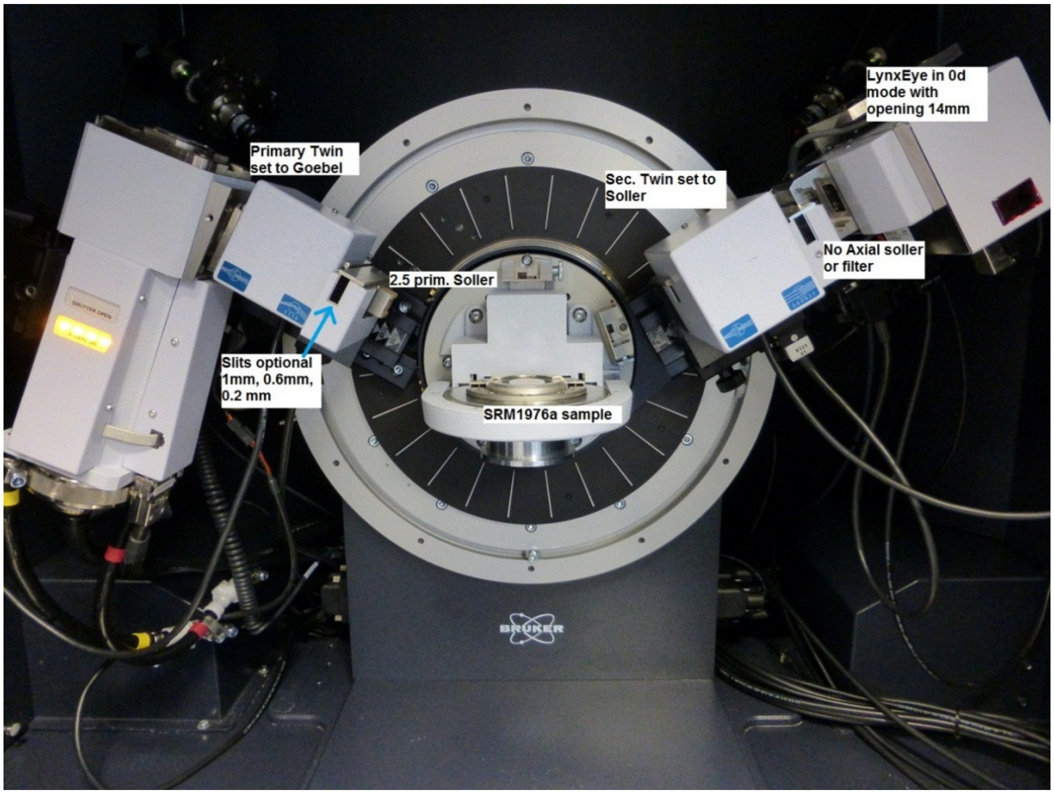
The secondary twin optic and even the empty secondary optical bench will shade part of the detector opening and removing these components will result in approximately 25% higher intensity. (Compare the red and blue scan in the plot above). The knife edge will have the largest effect on reducing the air scatter. The optimum height of the knife edge will depend on the desired measurement range. A knife edge that is adjusted too low will result in intensity loss at high angles, because it will cut into the incident beam. The height of the knife in these example scans was adjusted to exactly 1mm above the sample (using the thickness of a glass slide as a spacer between corundum plate and knife edge). Without the secondary twin optic that spacing will result in a usable 2 theta range between 4 and 70 degrees without odd background shapes at low angles or intensity loss at high angles.

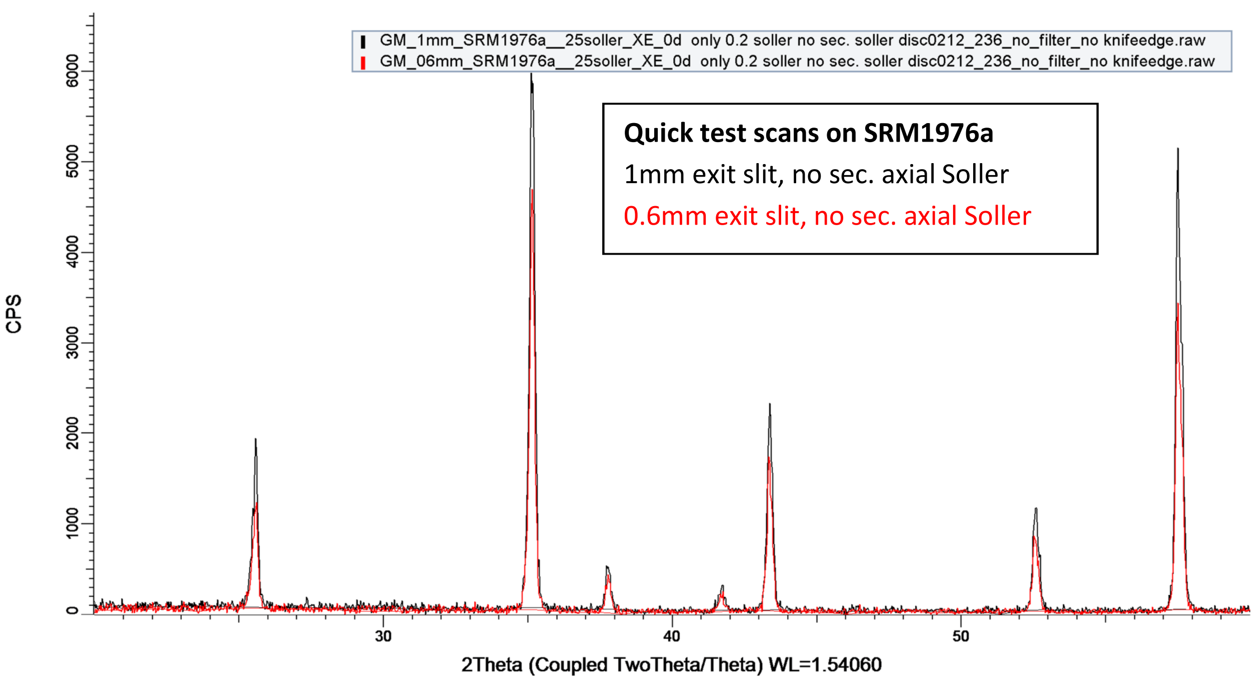
With the secondary twin optic mounted and the detector opening decreased, lower angles than 3 degrees can be a measured.

For scans where more intensity is required for better statistics to detect minor impurity phases the standard 2.5° axial Soller slits can be replaced by 4° Solller slits or the secondary axial Soller can be removed completely, if the peaks width is limited by crystallite size broadening.

Parallel Beam geometry on SRM 1976a (for GID or irregular shaped samples)

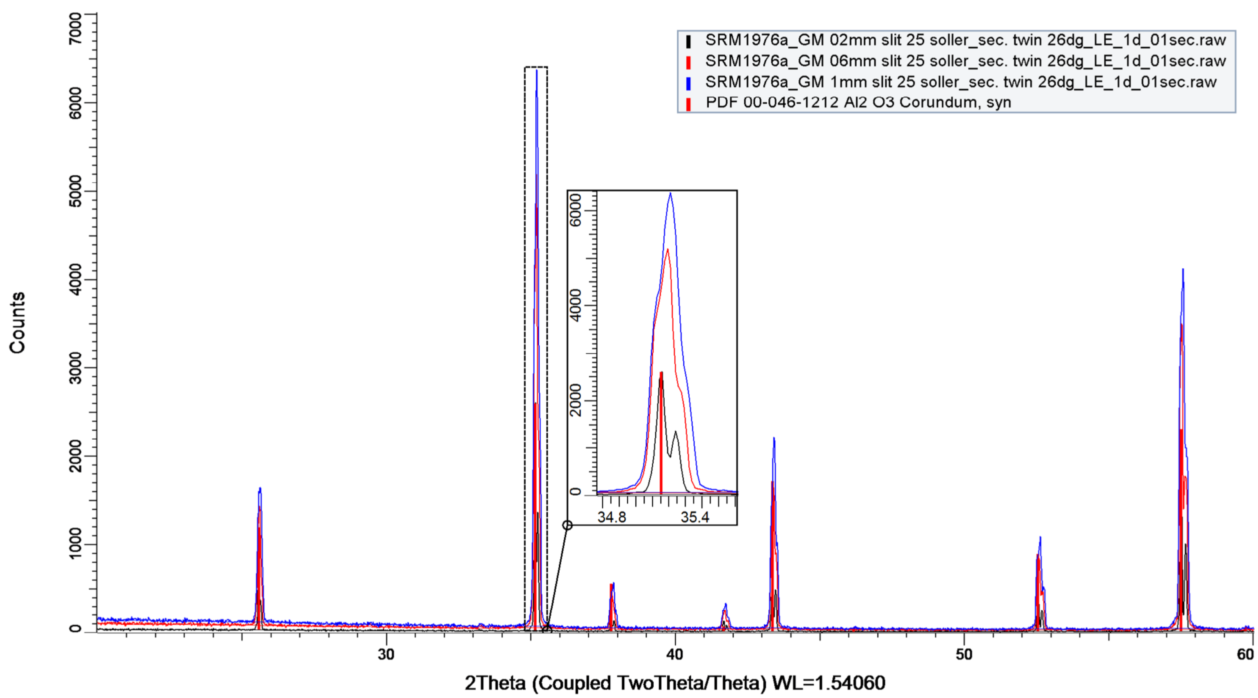
Generator	40kV40mA
Primary Twin Optic	Twin Primary set to Goebel mirror
Radiation safety slit	Optional 1mm, 0.6mm or smaller depending on desired beam width
Primary Axial Soller Slit	2.5° (or 4° for more intensity)
Sample	SRM1976a
Knife Edge	Usually not necessary
Secondary Twin Optic	Set to Soller 0.2°
Secondary Slit	None
Secondary Axial Soller Slit	2.5° or none for more intensity
Detector	LynxEye 0D set to maximum opening
Measurement	Coupled TwoTheta/theta continuous scan from 20° to 60° with 0.03 steps and 0.1sec/step
Example Scan files	\ scans after alignment\pb)


LynxEye in 0d mode with opening 14mm
Primary Twin set to Goebel
Sec. Twin set to Soller
No Axial soller or filter
2.5 prim. Soller
Slits optional 1mm, 0.6mm, 0.2 mm
SRM1976a sample
BRUKER

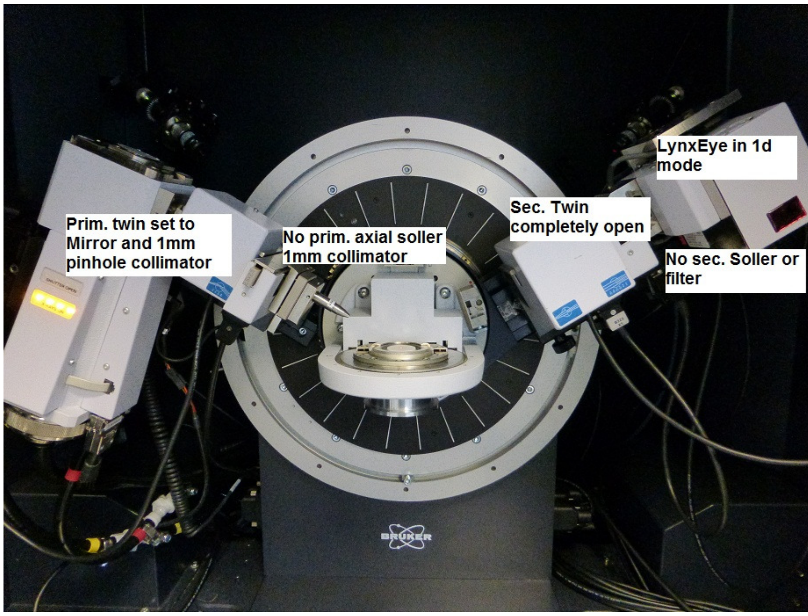

GM_1mm_SRM1976a__25soller_XE_0d only 0.2 soller no sec. soller disc0212_236_no_filter_no knifeedge.raw
GM_06mm_SRM1976a__25soller_XE_0d only 0.2 soller no sec. soller disc0212_236_no_filter_no knifeedge.raw
Quick test scans on SRM1976a
1mm exit slit, no sec. axial Soller
0.6mm exit slit, no sec. axial Soller
CPS
0
1000
2000
3000
4000
5000
6000
30
40
50
2Theta (Coupled TwoTheta/Theta) WL=1.54060

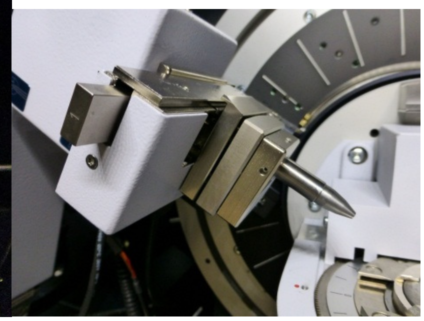
Parallel Beam geometry on SRM1976a
(for capillaries or in combination with pinholes for micro diffraction)

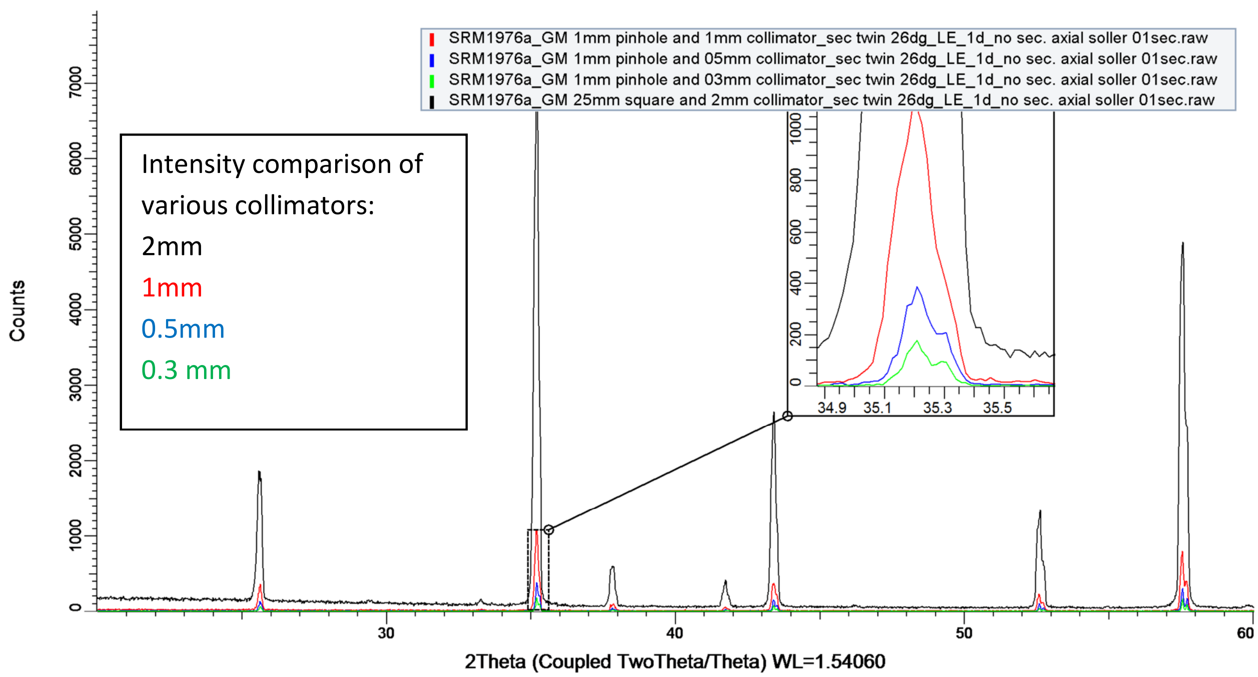
Generator	40kV40mA
Primary Twin Optic	Twin Primary set to Goebel mirror
Radiation safety slit	Optional 1mm, 0.6mm or 0.2mm depending on desired beam width and resolution
Primary Axial Soller Slit	2.5° (or 4° for more intensity)
Sample	SRM1976a
Knife Edge	Set to 1mm above sample Two knife edges above and below sample available for capillary stages
Secondary Twin Optic	Set to maximum opening or removed
Secondary Slit	None
Secondary Axial Soller Slit	2.5° (or 4° for more intensity)
Detector	LynxEye 1D set to maximum opening
Measurement	Coupled TwoTheta/theta continuous scan from 20° to 60° with 0.016 steps and 0.1sec/step
Example scan files	in subdirectory \ scans after alignment\cap



The size of the exit slit will determine peak resolution and peak intensity. For very crystalline samples an exit slit of 0.2mm will lead to better peak resolution.



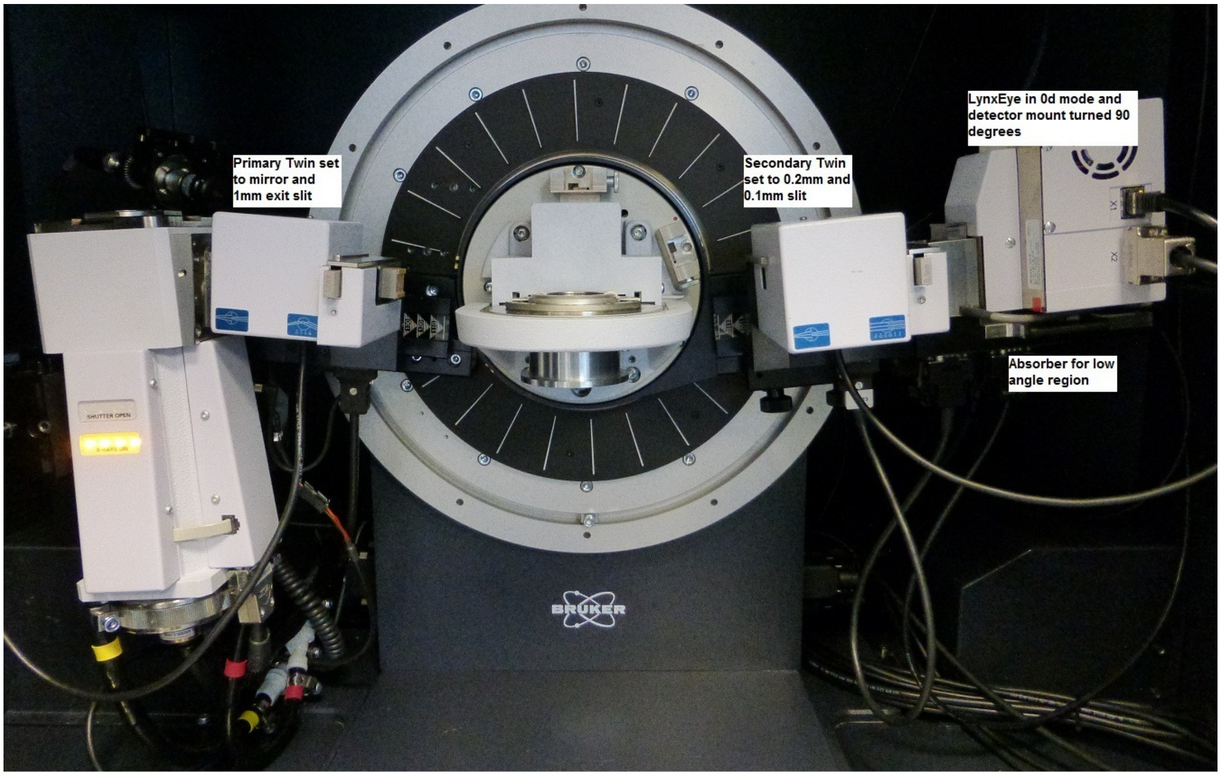




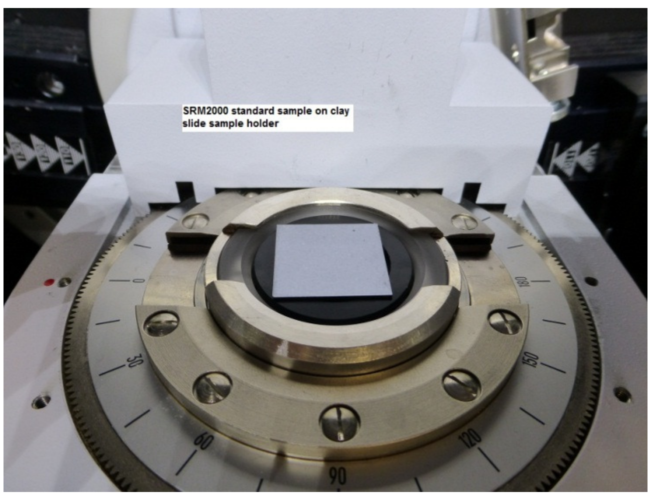
(Example scans in subdirectory \ scans after alignment\microdiffraction)

The 2mm collimator size would be suitable for texture measurements in combination with a compact cradle

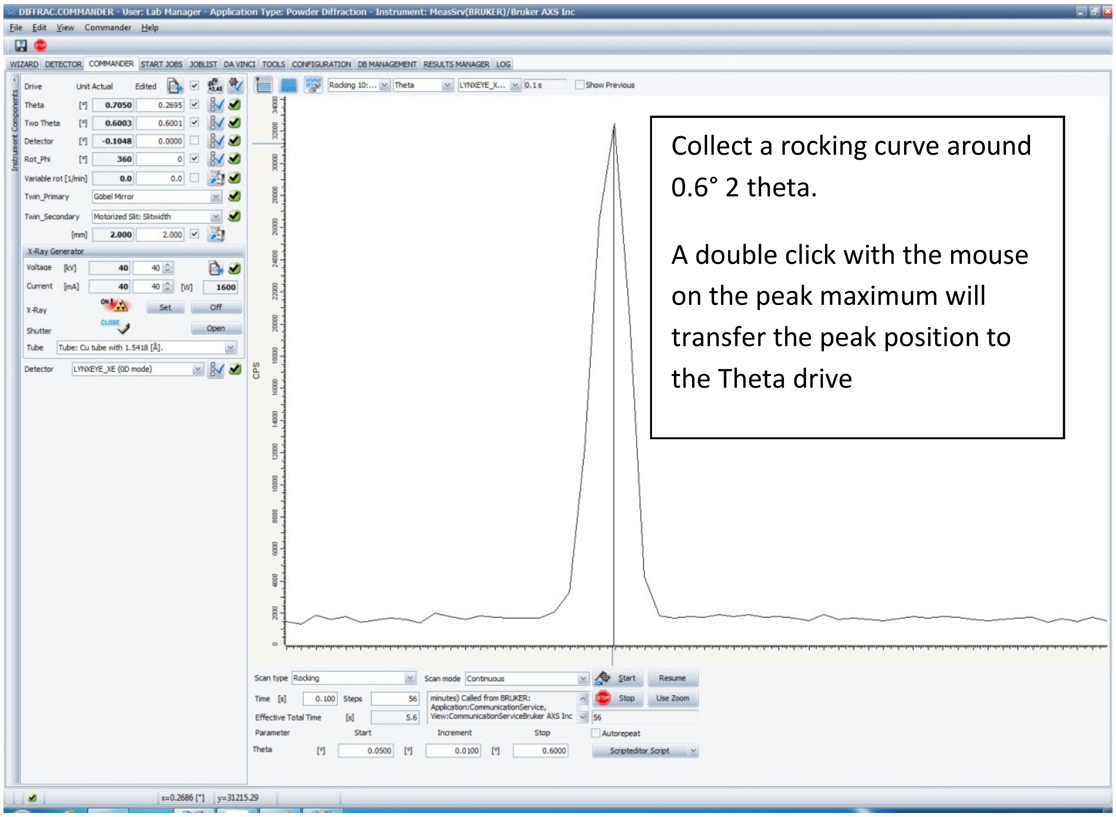
Parallel Beam geometry, XRR on SRM2000 sample

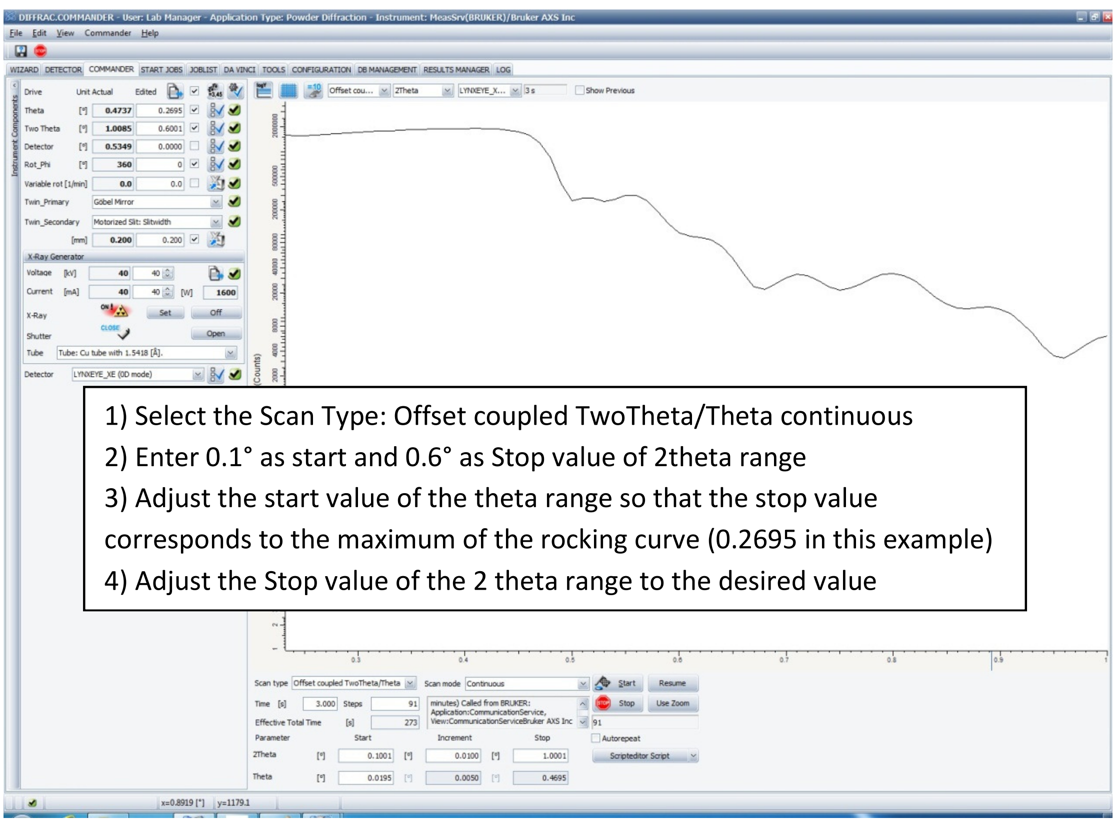


The NIST SRM2000 standard sample was mounted on a height adjustable clay slide sample holder.



To adjust the position of the sample to the beam a rocking curve around 0.6°2 theta was measured using a 0.1mm Cu absorber foil. Usually the peak occurs somewhere near the expected position of 0.3° but not exactly. If the peak is far off the expected position, remount the sample and try again. On a rotation stage the sample cannot be adjusted through z or chi scans as with a cradle system or a dedicated XRR stage. That means that mounting the sample at the right height and without tilt is critical to obtain good data.

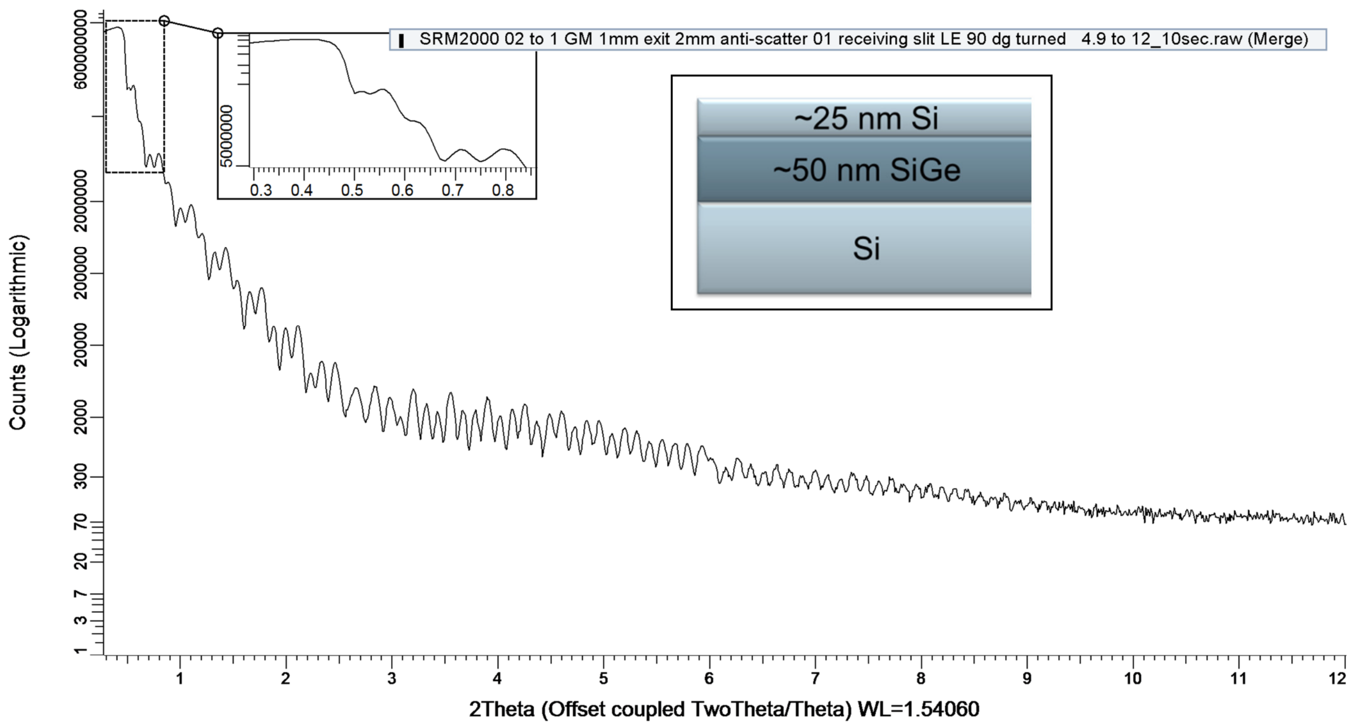

Collect a rocking curve around 0.6° 2 theta.
A double click with the mouse on the peak maximum will transfer the peak position to the Theta drive


1) Select the Scan Type: Offset coupled TwoTheta/Theta continuous
2) Enter 0.1° as start and 0.6° as Stop value of 2theta range
3) Adjust the start value of the theta range so that the stop value corresponds to the maximum of the rocking curve (0.2695 in this example)
4) Adjust the Stop value of the 2 theta range to the desired value

The SRM2000 data below was collected in three ranges

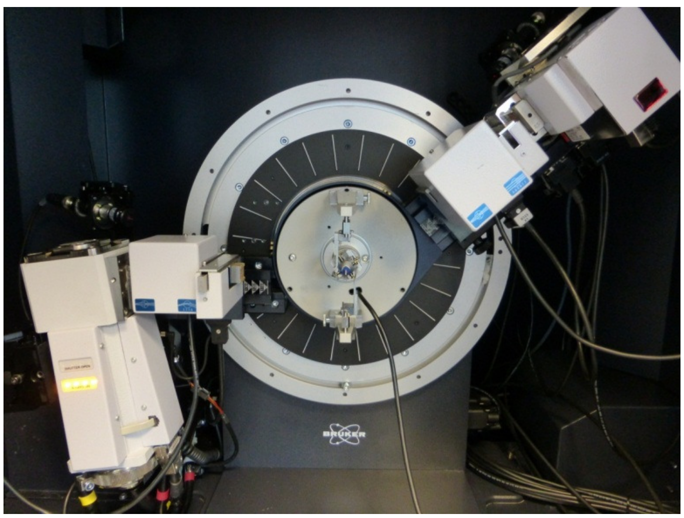
0.2 to 1° 2 theta	0.1 Cu absorber 0.01° steps, 3sec/step
0.9 to 5° 2 theta	0.01° steps, 3sec/step
4.9 to 12° 2 theta	0.01° steps, 10sec/step

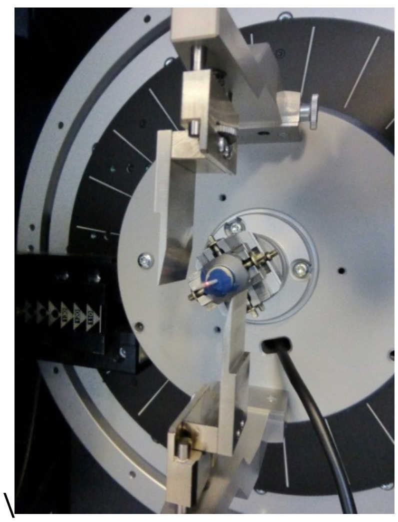
(Example scans in subdirectory \ scans after alignment\XRR)

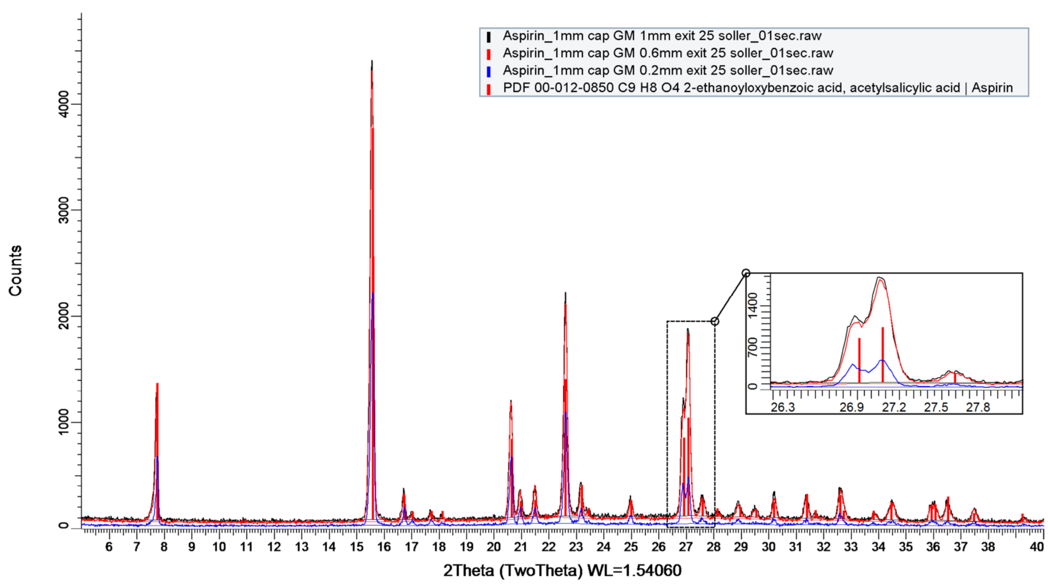


Parallel Beam geometry on 1mm Aspirin glass capillary

Generator	40kV40mA
Primary Twin Optic	Twin Primary set to Goebel mirror
Radiation safety slit	1mm, 0.6mm and 0.2mm
Primary Axial Soller Slit	2.5°
Sample	1mm glass capillary with Aspirin mounted with clay on goniometer head
Knife Edge	Two knife edges, above and below sample
Secondary Twin Optic	Set to maximum opening
Secondary Slit	None
Secondary Axial Soller Slit	2.5°
Detector	LynxEye 1D with 2.5° opening
Measurement	TwoTheta continuous scan from 5° to 4° with 0.02 steps and 0.1sec/step
Example scan files	subdirectory \ scans after alignment\Cap

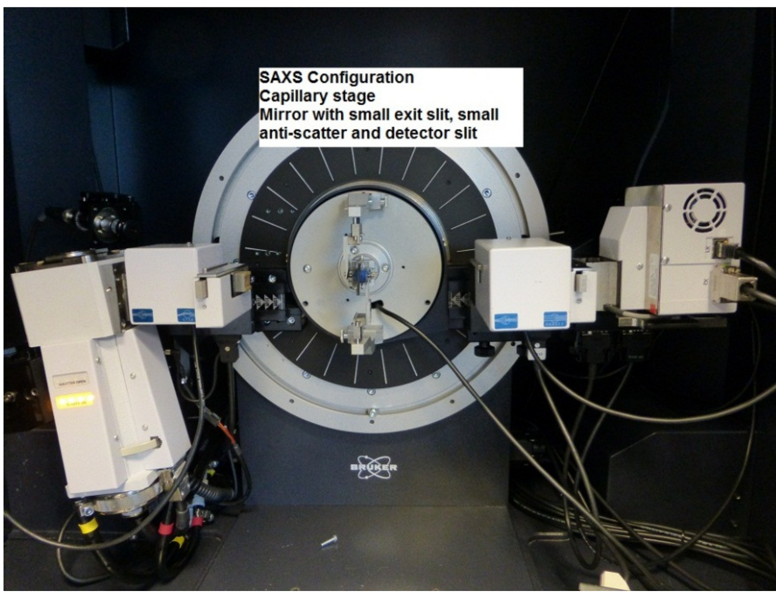


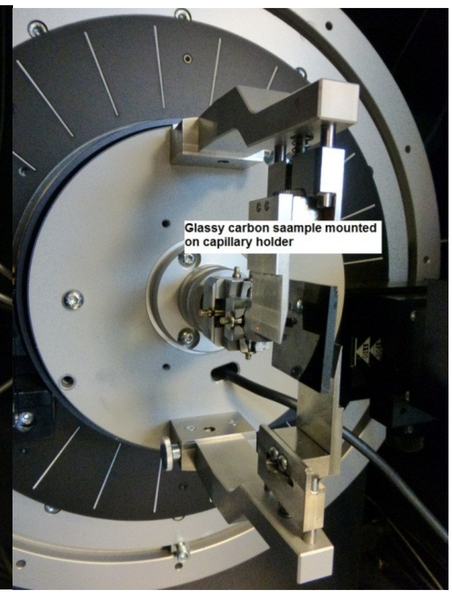




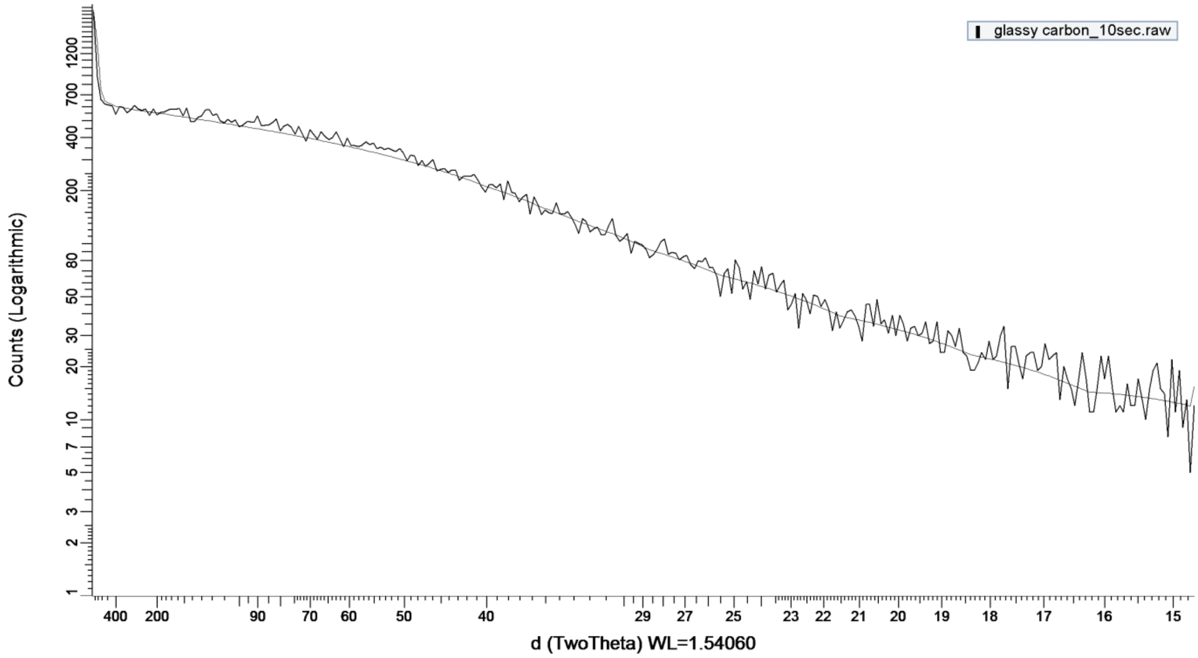
SAXS on glassy carbon sample

Generator	40kV40mA
Primary Twin Optic	Twin Primary set to Goebel mirror
Radiation safety slit	0.1mm or 0.05mm
Primary Axial Soller Slit	2.5°
Sample	Glassy carbon with pores
Knife Edge	Two knife edges above and below sample available for capillary stages
Secondary Twin Optic	Set to 0.1mm
Secondary Slit	0.1mm
Secondary Axial Soller Slit	None
Detector	LynxEye 0D turned 90 degree
Measurement	TwoTheta continuous scan from 0.1° to 8° with 0.02 steps and 10sec/step
Scan files	in subdirectory \ scans after alignment\SAXS





Scatter profile of glassy carbon sample



SAXS simulation in Nanofit