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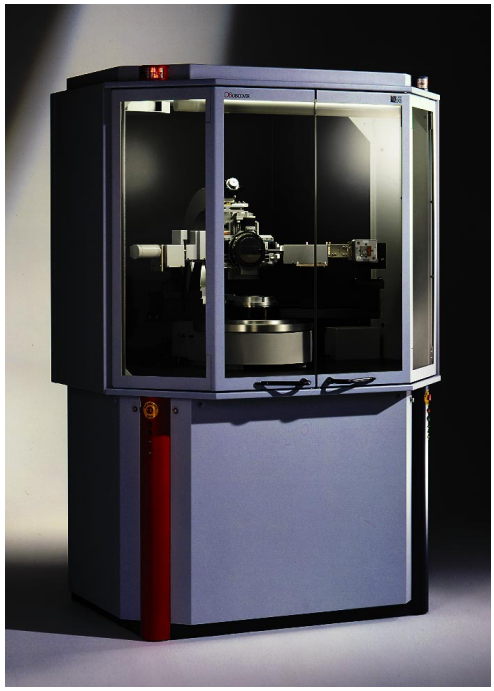
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D8 X-ray Diffractometer Vol. II

D8 ADVANCE



D8 DISCOVER



D8 X-ray Diffractometer Vol. II

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1 9 Position Sample Stage

Introduction

The 9 Position Sample Stage is meant for routine reflection and/or transmission measurement of up to 9 samples of different types (fig. 1-4, fig. 1-5). The sample rings of this stage are compatible with the standard sample stages. The 9 position stage consists of 2 parts: The stage and the magazine. The installation and alignment is similar to the rotary stage. It is compatible with vertical θ–θ and θ–2θ goniometers.

General Features

Reproducibility in 2θ of identical sample (σ)	better +/-0.005°
Reproducibility of standard quartz (σ)	better +/- 0.01°
Precision of surface position of sample rings	better +/- 0.025 mm
Measurement height 150 mm	up to 9 samples
Measurement height 214 mm	up to 7 samples (magazine and stage are identical for 214 mm and 150 mm height, only a limit switch is inserted)
Rotation	0 or 30 rpm
Measurement circle diameter	435 mm, 500 mm, 600 mm, or any intermediate value
θ range (θ–2θ)	at least –3° ... 183°
2θ range (θ–2θ)	at least –3° ... 165°
tube range (θ–θ)	at least -3° 168°
detector range (θ–θ)	at least -3° 168°
2θ range (θ–θ)	at least -3° 165°
Transmission θ–θ	short tracks, 435 mm diameter, 150 mm height, no counter balance on detector circle, standard secondary slit system

Necessary Equipment

Hardware

1. 9 Position Sample Stage (C79298-A3244-B250 which includes cable set C79298-A3130-A81: Interface cable to D8 frame and Connection Cable D8 frame to 9 Position Sample Stage)
2. I/O Board with version 4 or higher (C79298-A3220-B103)
3. Vertical D8 goniometer, measuring circle height 150 mm recommended (9 samples possible), 214 mm also possible (up to 7 samples)
4. Sample magazine (C79298-A3244-B251 or C79298-A3244-B252)
5. Sample rings (e.g. basic set C79298-A3244-D80, at least quartz and glass slit are necessary for alignment)

(No 4axis or 2axis board required!)

Firmware

Release: 31.5.1999 or newer

Software

1. D8Adjust: 31.5.1999 or newer
2. D8Meas: 26.4.1999 or newer
3. D8Stndbye: 26.4.1999 or newer
4. Edit Job: 27.5.1999 or newer
5. Config: 18.5.1999 or newer

Additional features of software and firmware

Firmware

There are four additional direct commands:

1. IN17: Initialise 9 Position Sample Stage (translate magazine to sample 1)
2. SA1, ... SA9: Put sample 1 ... 9 to measurement position, rotate sample by one revolution
3. SA0: Release sample and translate sample magazine to position for magazine change (move to sample 7)
4. SR1, SR0: Switch on/off sample rotation

After performing SA0 the 9 Position Sample Stage is not initialised anymore and a IN17 is necessary before other commands are accepted.

Configuration

To put the 9 Position Sample Stage into operation it is necessary to change the configuration (fig. 1-6). The “Multi-Position” sample stage has to be selected. Depending on measuring circle height the maximum number of samples has to be defined: “7x” for 214 mm height and

"9x" for 150 mm height. The configuration program takes care that the other software (D8Adjust, Editjob) does not allow positions with collisions possible.

Be aware that it is not possible to configure several sample stage at the same time, e.g. rotary stage together with 9 Position Sample Stage.

D8Adjust

There are four additional features (fig. 1-7)

1. **Sample Position**: Type in the number of the sample to be measured. The numbers are labelled on the magazines. Depending on configuration samples 1 ... 7 or 1 ... 9 are allowed. With "Data Collection" the sample is put to the measurement position and rotated exactly one turn to guarantee the correct positioning of the surface of the sample to goniometer centre.
2. **Magazine release:** Click on button and the magazine will move to release position (sample 7). Afterwards it is necessary to initialise the 9 Position Sample Stage before measuring is possible again (use e.g. "initialise all button").
3. **Sample Rotation:** Click on "rotation on". The next measurement will be performed with 30 revolution per minute. The rotation is automatically switched off in case of sample changing. To switch off the rotation click on "rotation on" to activate the "rotation off" button. Then click on "rotation off". The zero position of the rotation is lost if the rotation has been used (i.e. do not use the rotation feature if the absolute rotary position is important, e.g. for measurement with the glass slit)
4. **Initialisation:** The "initialise all" command includes the initialisation of the 9 Position Sample Stage

Edit Job

In Edit Job the number of the sample to be measured must be inserted (fig. 1-8). Edit Job takes care that only allowed numbers are taken (in case of correct configuration). If "enter" is used after typing in the first line, the next line is filled out automatically with sample position increased.

Status Display

The status display gives a graphical information about the state of the 9 Position Sample Stage and the conditions of the sample (fig. 1-9): green = measurement finished, blue = active measurement, red = not measured, yellow = aborted, dark yellow = interrupted, white = skipped, grey = no sample.

D8 Standby

If the D8 goes to standby values, the 9 Position Sample Stage moves the sample magazine to magazine release position (sample 7).

Installation

First Switch off D8. Install 9 Position Sample Stage to goniometer in the same manner as described for the rotary and standard sample stage (chapter 3 D8 user manual vol. I): Fasten softly with 3 fixing screws, centre by fixing centring screw rigidly, fasten rigidly 3 fixing screws. Loosen centring screw and softly hit it on the top for detaching centring wedge to make future removal of the stage easier (fig. 1-10).

Take out I/O board and check version (4 or higher). Change I/O board if necessary. Install interface cable and reinstall I/O board. Fix loose end of interface cable to D8 frame. (fig. 1-11, 1-12, 1-13).

Guide connecting cable through radiation labyrinth and connect it to D8 frame and 9 Position Sample Stage (fig. 1-12, 1-4).

Fig. 1-13 shows the cabling in case a FDC controller is installed together with the 9 Position Sample Stage.

Check firmware state (floppy drive of D8). If the firmware is older than 31.5.99 upgrade the firmware. Check release dates of DiffracPlus software (compare “necessary equipment”) and upgrade if necessary.

In case of measuring height 214 mm 7 samples are accessible. Position 8 and 9 lead to collision with the front doors of the enclosure. Therefor light barrier actuators have to be installed on the magazines at position 7 to avoid movements beyond sample 7 (fig. 1-3). These actuators disturb the reflection of light back to the photo sensors of the light barrier for sample translation (fig. 1-18).

Switch on D8.

Change configuration of D8 (select “Multi-Position” sample stage and “7x” or “9x” for maximum number of samples, deactivate the sample stage which was in use before, fig. 1-6). Download new configuration.

Warning:
When installing the 9 Position Sample Stage, be sure that the mounting thread of the actuators for the safety switches match the value 110 mm and 70 mm, measured from the goniometer's rear edge. If this requirement is not fulfilled, cut the thread M5 at this location (see figure on the following page).

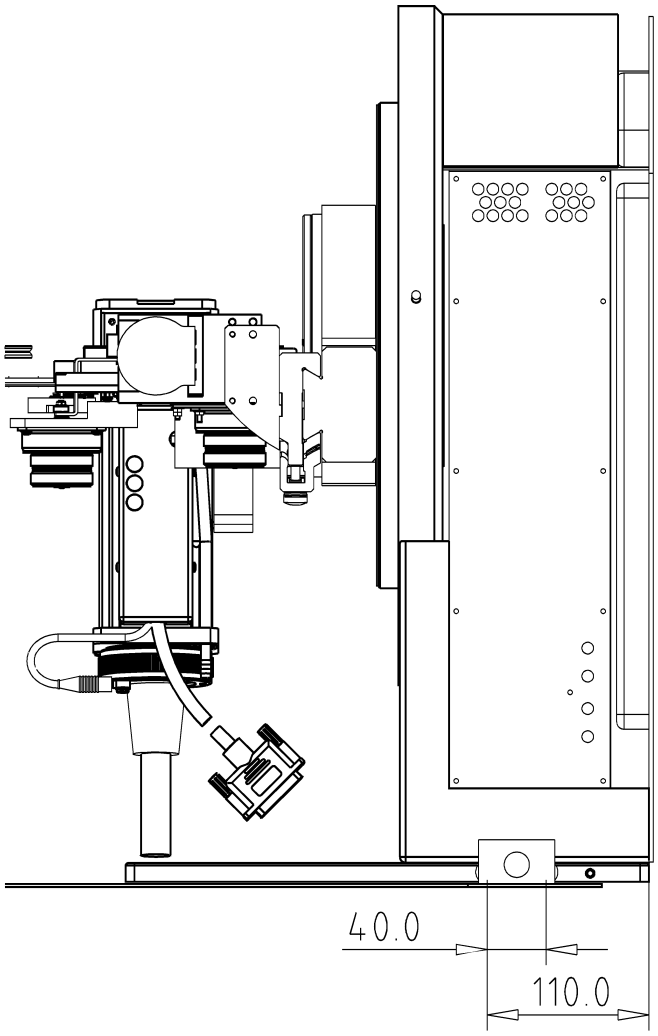


Fig. 1-1: The actuators mounting thread must be mounted properly

Change of sample rings

Push sample release, push sample ring against support rings, release actuator (fig. 1-14).

Release of magazine

Move magazine to release position, if necessary: Sample magazine horizontal and magazine at position of sample 7 (fig. 1-15, use magazine release button of D8 Adjust). Push red “translation release” actuator to the right on 9 Position Sample Stage to give free translation of magazine (fig.1-16). Move out magazine as far as possible. Use red “Magazine release” actuator on magazine to take out magazine. (fig. 1-17).

⚠ **Caution:**
Do not bend linear guide of 9 Position Sample Stage!

⚠ **Caution:**
Do not touch light barrier (fig. 1-18)!

Insertion of magazine

Red “translation release” actuator of sample translation must be in out position (right, fig. 1-16). Move out linear guide of 9 Position Sample Stage as far as possible – **do not bend!** Shift magazine against bolt (fig. 1-17, 1-18). Push red “magazine release” actuator of magazine and release it when magazine is in position, i.e. it has fixed the locking pin of the magazine (fig. 1-15). Translate magazine roughly to position of sample 7 and push red “translation release” actuator of sample stage back to working position (left, fig. 1-19). Initialise 9 Position Sample Stage.

Caution: ⚠
Do not touch light barrier! (cf. fig. 1-18)

Alignment

In this manual the standard alignment for a θ–2θ goniometer in Bragg-Brentano geometry will be described. As the alignment is quite similar to the standard sample stage refer to the respective parts of the D8 users manual for questions regarding alignment of the different monochromators and for a θ–θ goniometer.

☞ The standard D8 alignment with a standard sample stage as described in chapter 3 of the D8 manual vol. I should be performed first: all slits, the optics, the detector.

Put glass slit in magazine position 1 and quartz reference sample in magazine position 2 (fig. 1-20). The position of the sample surface is defined by three wheels. They are factory aligned in such way that the surface is automatically in the centre of the goniometer if the installation has been performed as described above. It is only necessary to define the zero degree position of θ and 2θ.

1. Perform rocking curve with glass slit, divergence slit 1 mm, anti scattering slit 1 mm, no detector slit, 0.1 mm Cu absorber, 40 kV, 40 mA, step size 0.004°. Determine maximum position.
2. If maximum of scan (1) differs by more than 0.03° from 0.0° set new ZI value of θ and repeat rocking curve described in (1). (fig. 1-21).
3. Without change of sample (to get highest precision possible) move θ to maximum of rocking curve. Perform a 2θ scan (0.004° step size) with glass slit still inserted to define the zero position of the beam and an additional detector slit of 0.1 mm compared to rocking curve conditions in (1). Define new ZI value of 2θ at the maximum of this scan. This gives the 0° position of the instrument. (fig. 1-22)
4. For safety reasons repeat the rocking curve without 0.1 mm detector slit. Check again if maximum position does not differ by more than 0.03° from 0.0° and move to maximum. Repeat 2θ scan with additional 0.1mm slit. The result can be accepted if the maximum position of 2θ does not differ by more than 0.004° from 0.0°.

Perform a locked coupled scan (step size 0.004°) with the reference quartz sample (position 2), 1 mm divergence slit, 1 mm anti scattering slit, 0.1 mm detector slit, 40 kV, 40 mA. The 100% quartz reflection should be at 26.64° +/- 0.01° (ICDD 46-1045) and have a countrate of above 10 000 cps. (Cu long fine focus tube, less than 1000h of usage, 435 mm diameter measuring circle. (compare fig. 1-23).

Finally the position of the hardware and software limit switches should be checked and tested. The position of the hardware switches should be set slightly less limiting than the software limits (at least 0.5°). The positions listed are valid for 435 mm measuring circle diameter. For larger diameters larger ranges are possible and can be fine tuned if necessa-

ry. In case of a θ–θ goniometer with 435 mm diameter, 150 mm height, short tracks, no counter balance on the detector arm, and standard secondary slit system the detector can be moved down to –93°.

limit switch θ–θ	**position**
lower limit θ	-3°
upper limit θ	183°
lower limit 2θ	-3°
upper limit 2θ	165°

limit switch θ–2θ	**position**
lower limit tube	-3°
upper limit tube	168°
lower limit detector	-3°
upper limit detector	168°
collision limit	$2\theta = 165°$

Transmission Measurements

Take sample rings for transmission measurement (C79298-A3244-D81). Instead of performing “locked coupled” scans use “unlocked coupled” scans with start value of theta increased by 90° compared to the “locked coupled” scan. Reflection and transmission measurements might be mixed, but be aware that reflection samples might fall out depending on sample preparation. The accessible 2θ range is larger than 0° ... 90°.

Accessories

Basic Equipment (C79298-A3244-B250)

9 Position Sample Stage

connection cables to I/O Board

No magazine!

Basic Set Sample Rings (C79298-A3244-D80)

1 glass slit for 9 Position Sample Stage

1 quartz reference sample for 9 Position Sample Stage

6 standard sample rings (PMMA, reflection, 25 mm diameter)

3 standard sample rings (PMMA, reflection, 40 mm diameter)

Magazines

Standard (reflection and transmission) , sample thickness 8.5 mm:

C79298-A3244-B251

Fig. 1-2

Thick samples (reflection only), sample thickness 20 mm:

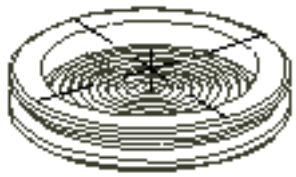
C79298-A3244-B252

Fig. 1-3

Sample Rings

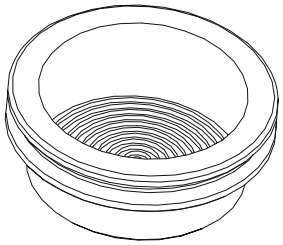
Standard Sample Ring

Part Number 9 Sample Rings	Ø Sample	Sample Height	Ring Height	Material
C79298-A3244-D82	25	1	8.5	PMMA
C79298-A3244-D83	40	6	8.5	PMMA
C79298-A3244-D84	25	1	8.5	Steel
C79298-A3244-D85	40	6	8.5	Steel



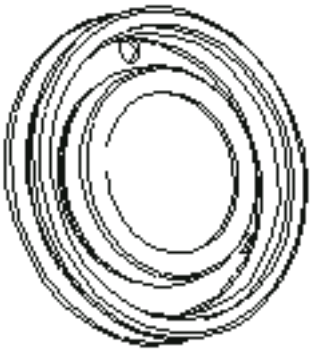
Thick Samples

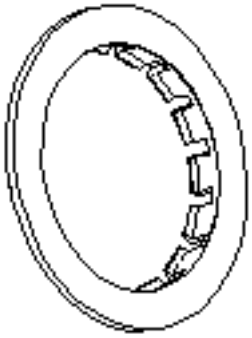
Part Number 9 Sample Rings	Ø Sample	Sample Height	Ring Height	Material
C79298-A3244-C254	40	17	20	Steel



Air Sealed

Part Number 9 Sample Rings	Ø Sample	Sample Height	Ring Height	Material
C79298-A3244-D97	25	1	8.5	PMMA
C79298-A3244-D98	25	4	8.5	PMMA

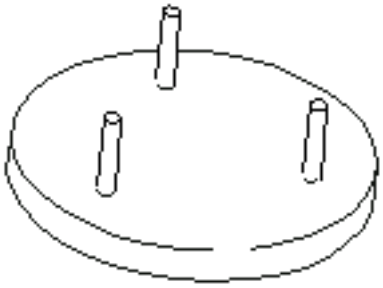




Preparation Tool

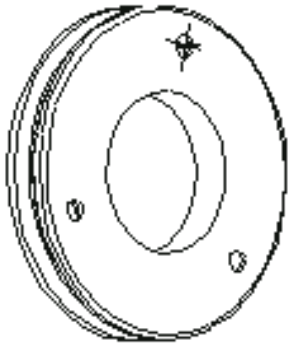
Preparation of back loading, air sealed, transmission, side loading sample rings)

C79298-A3244-B259



Back loading Sample Ring

Part Number 9 Sample Rings	Ø Sample	Sample Height	Ring Height	Material
C79298-A3244-D88	25	6.5	8.5	PMMA



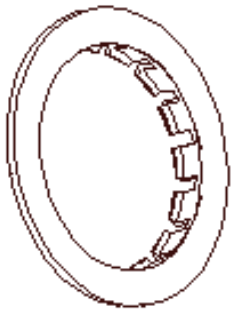


Transmission Sample Ring

Transmission measurements (e.g. Polymers), monochromator recommended (e.g. Goebel mirror, $K\alpha_1$ Transmission)

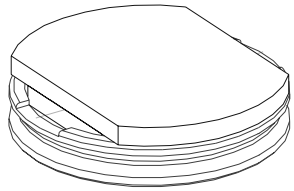
Part Number 9 Sample Rings	Ø Sample	Sample Height	Ring Height	Material
C79298-A3244-D81	11		8.5	PMMA





Glas Slit

Alignment
C79298-A3244-B255



Quartz Sample

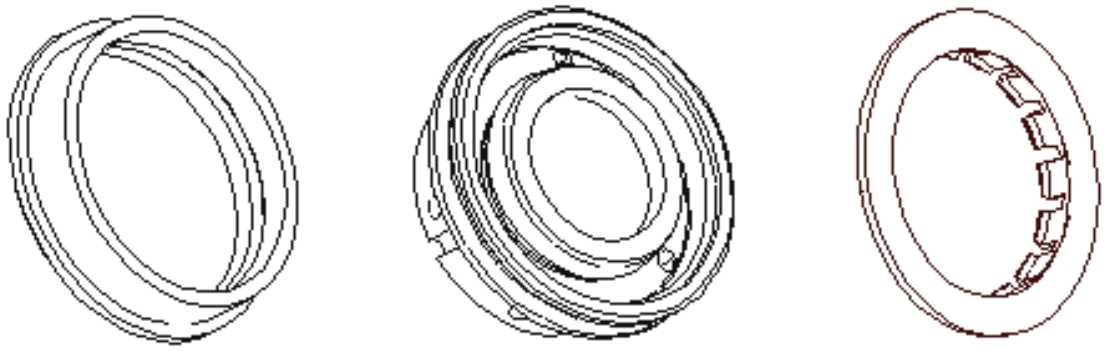
Reference Measurement, pressed quartz

C79298-A3244-B254



Air sealed, compatible with Matic

Part Number 9 Sample Rings	Ø Sample	Sample Height	Ring Height	Material
C79298-A3244-D96	25	4	20	Steel



Silicon single crystal sample ring (low background sample ring)

C79298-A3244-B249



Figures

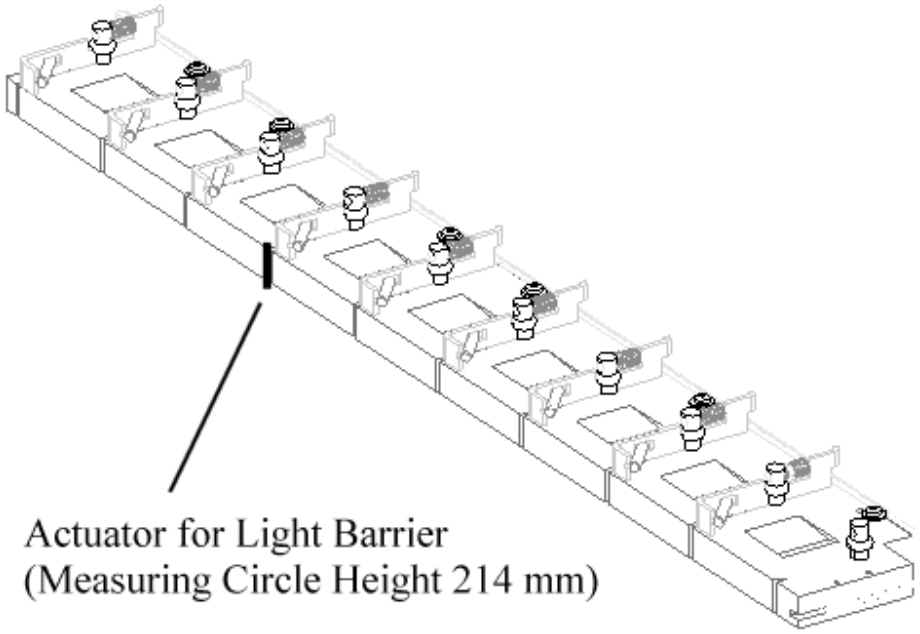


Fig. 1-2: Standard magazine for reflection and transmission

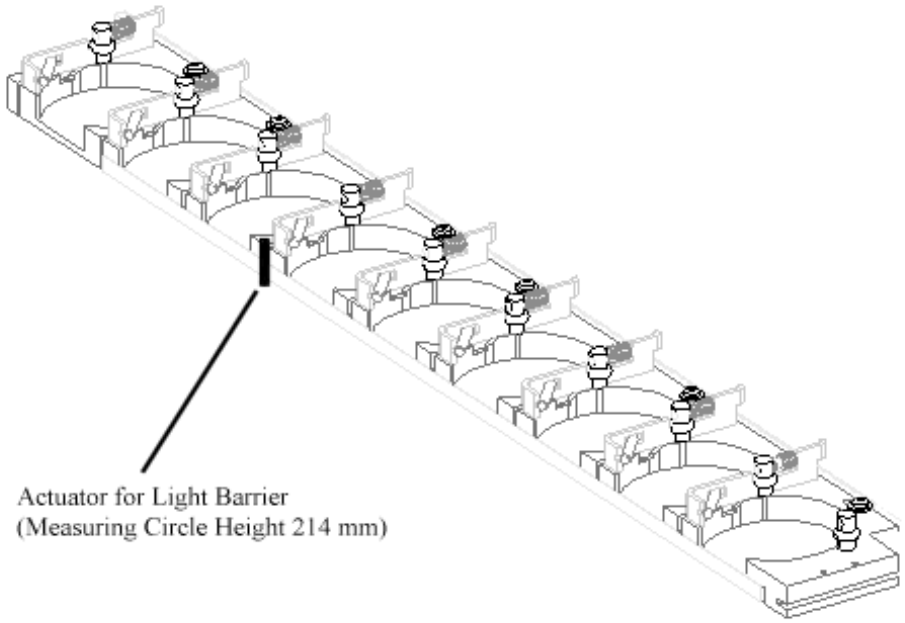


Fig. 1-3: Magazine for reflection measurements on thick samples

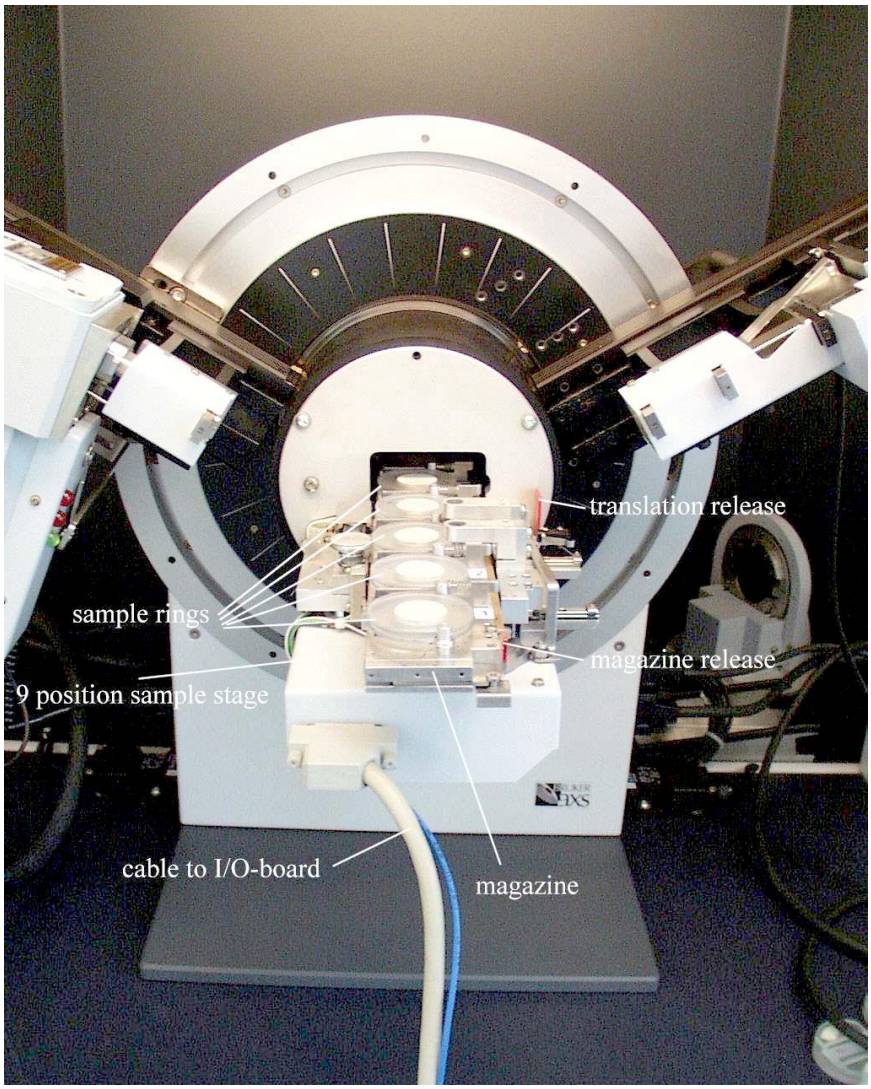


Fig. 1-4: 9 Position Sample Stage in reflection mode

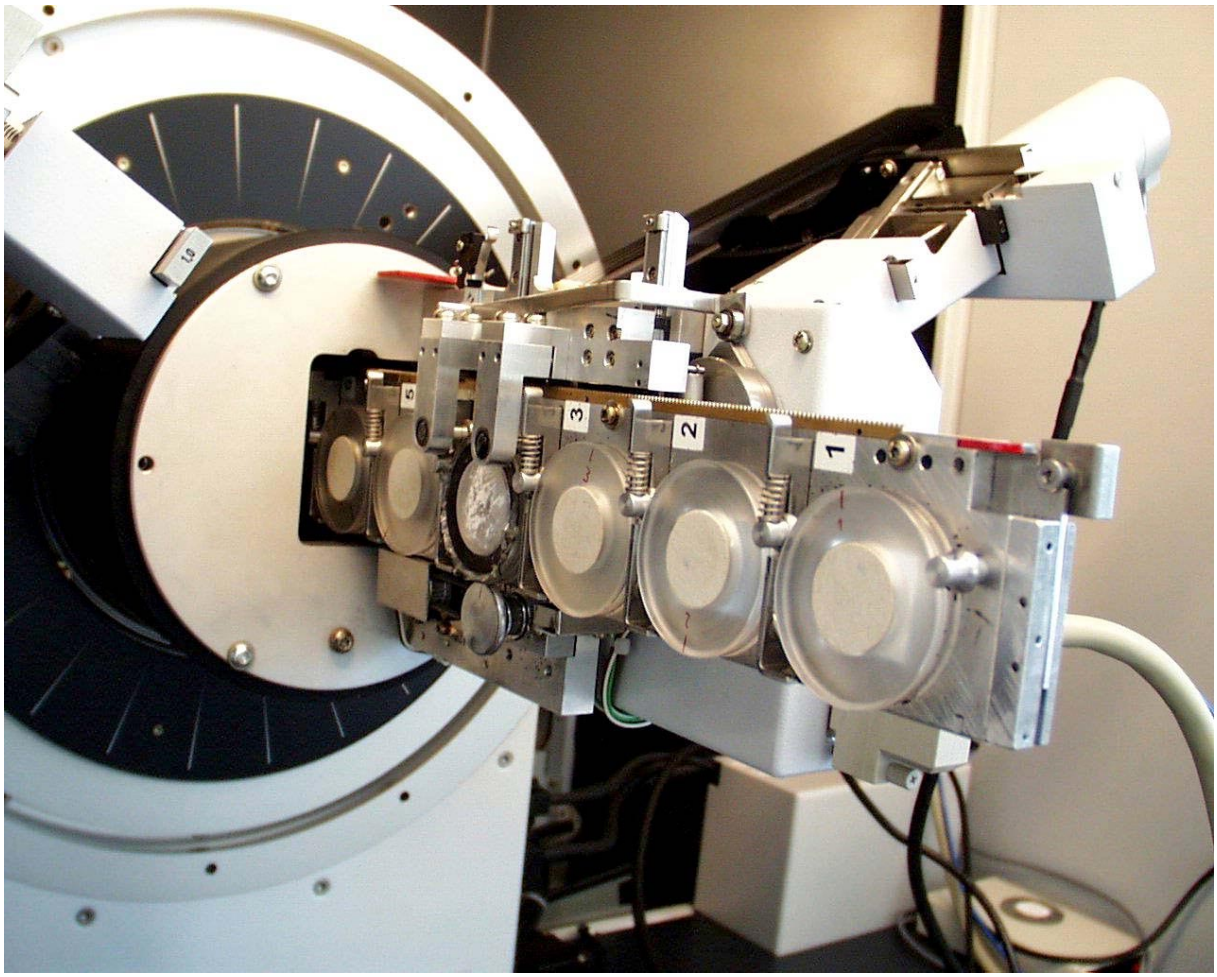


Fig. 1-5: 9 Position Sample Stage in transmission mode

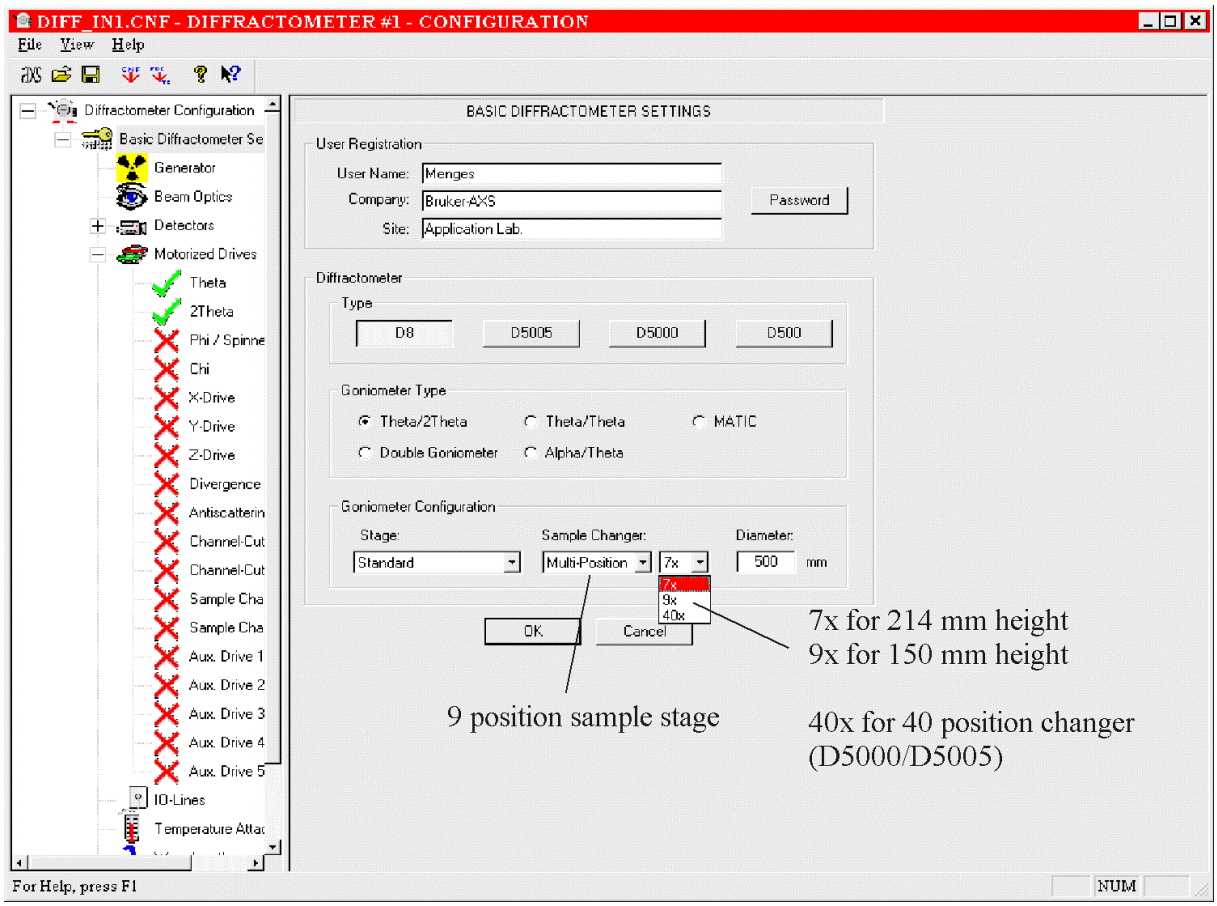


Fig. 1-6: Configuration of 9 Position Sample Stage

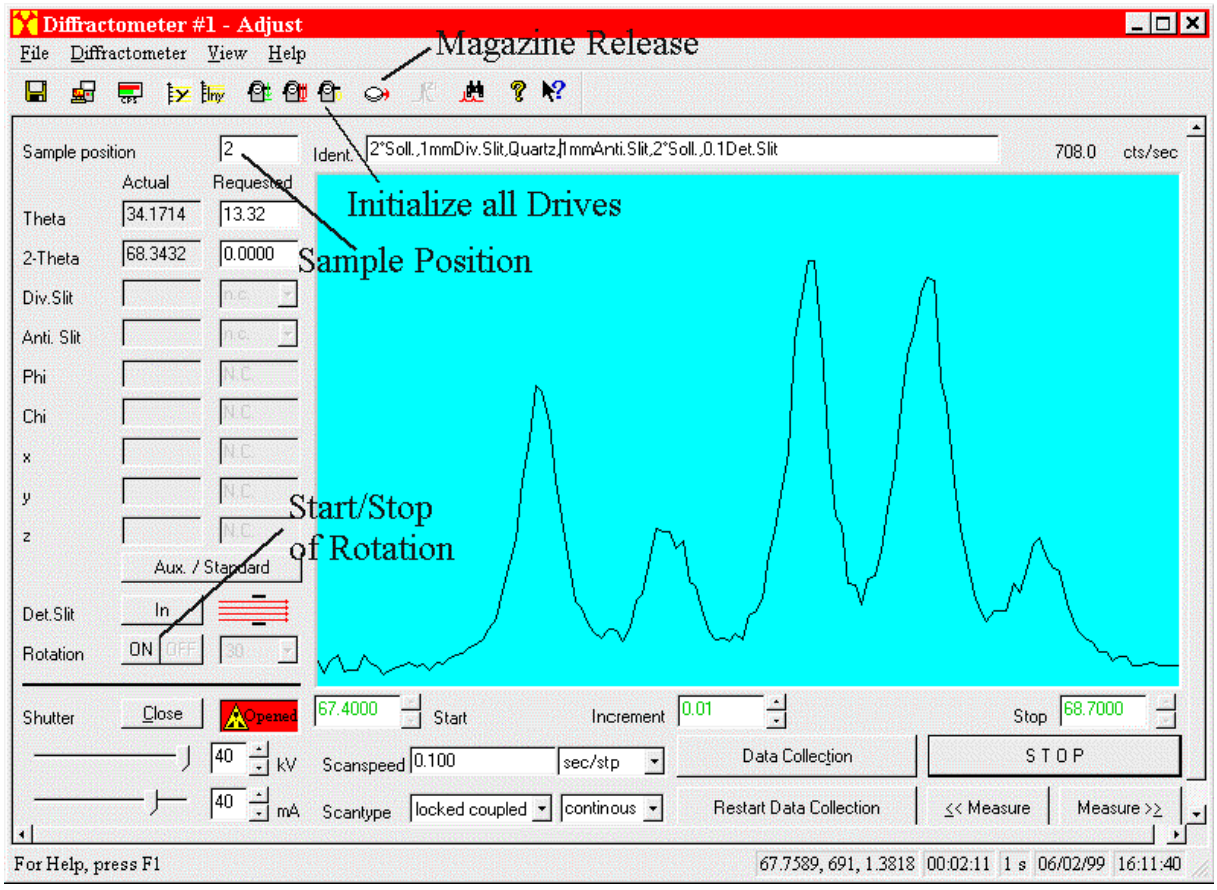


Fig. 1-7: Usage of 9 Position Sample Stage within the Adjust program

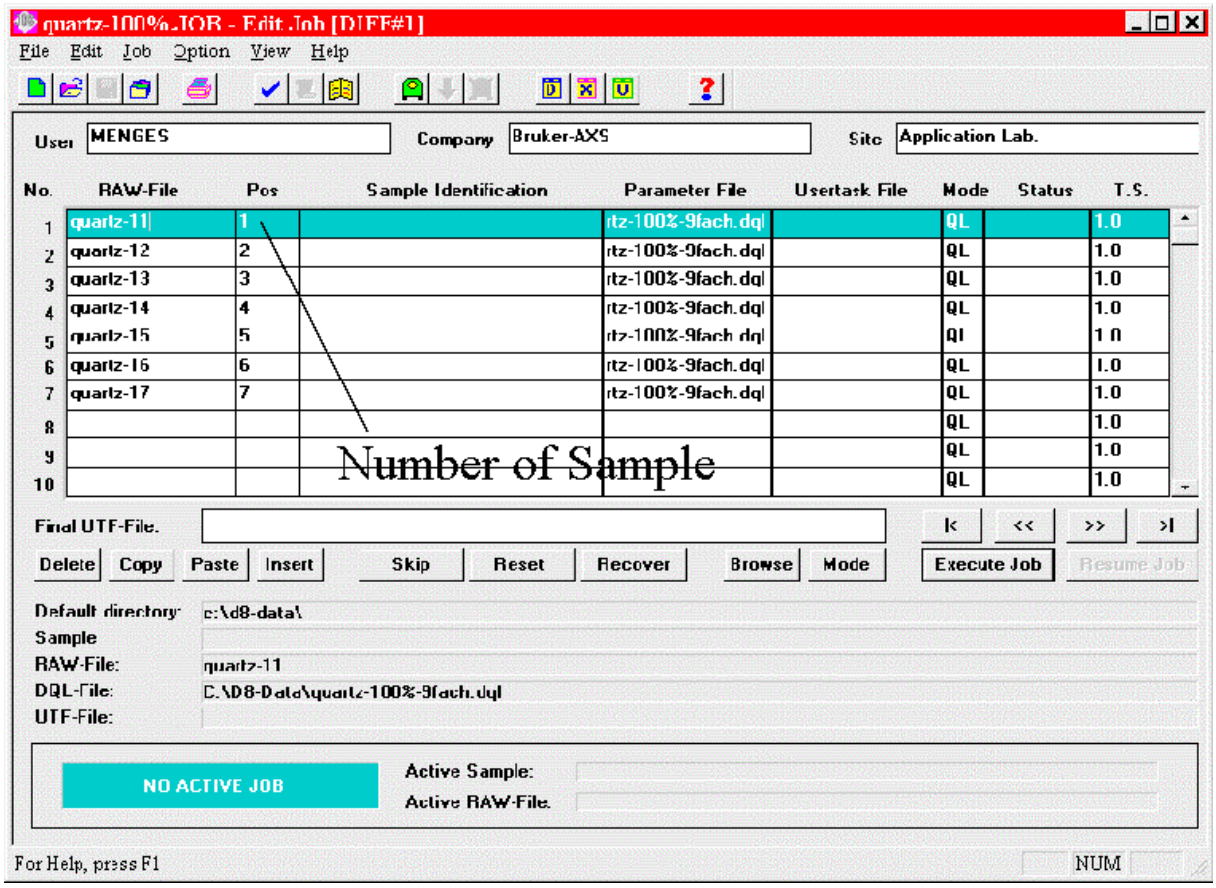


Fig. 1-8: Change of samples in Edit Job

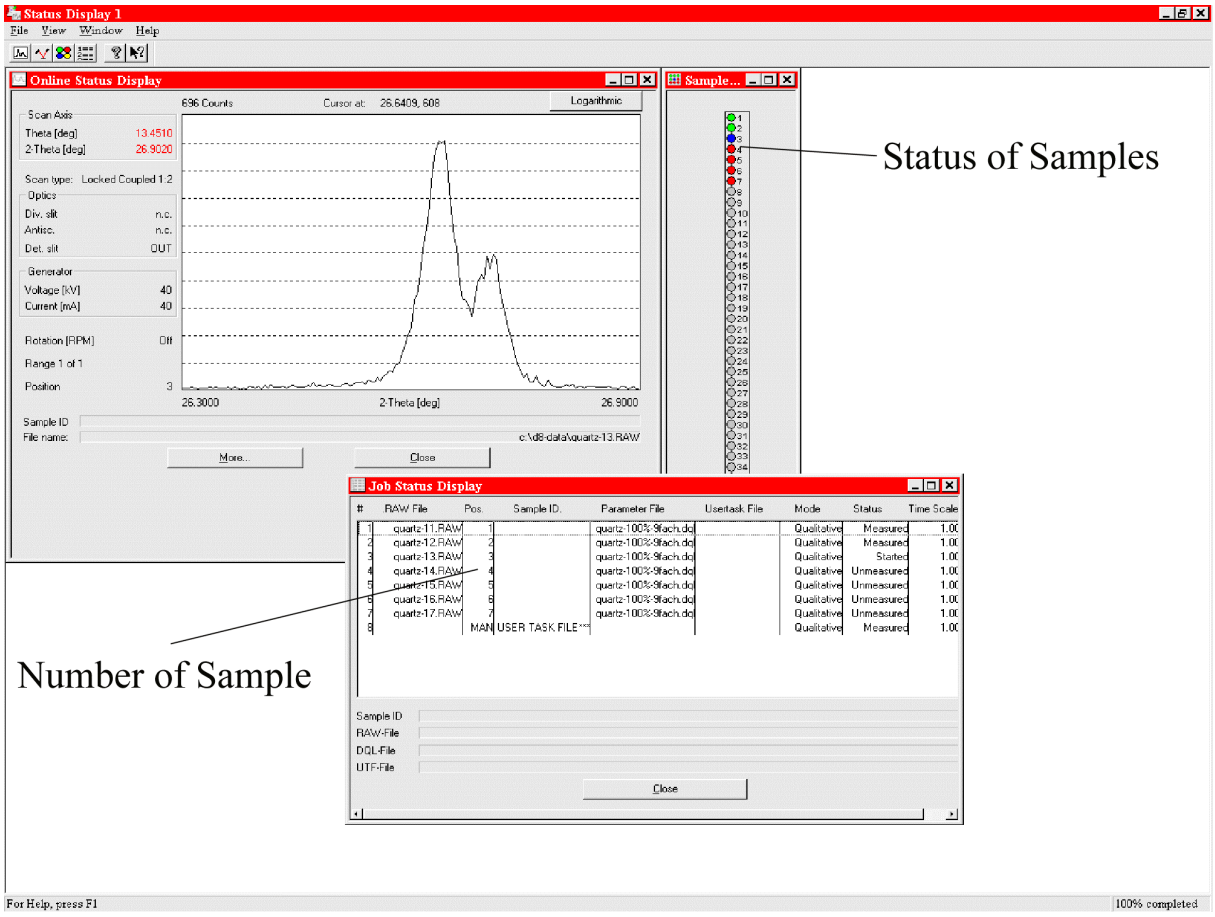


Fig. 1-9: Status display of 9 Position Sample Stage

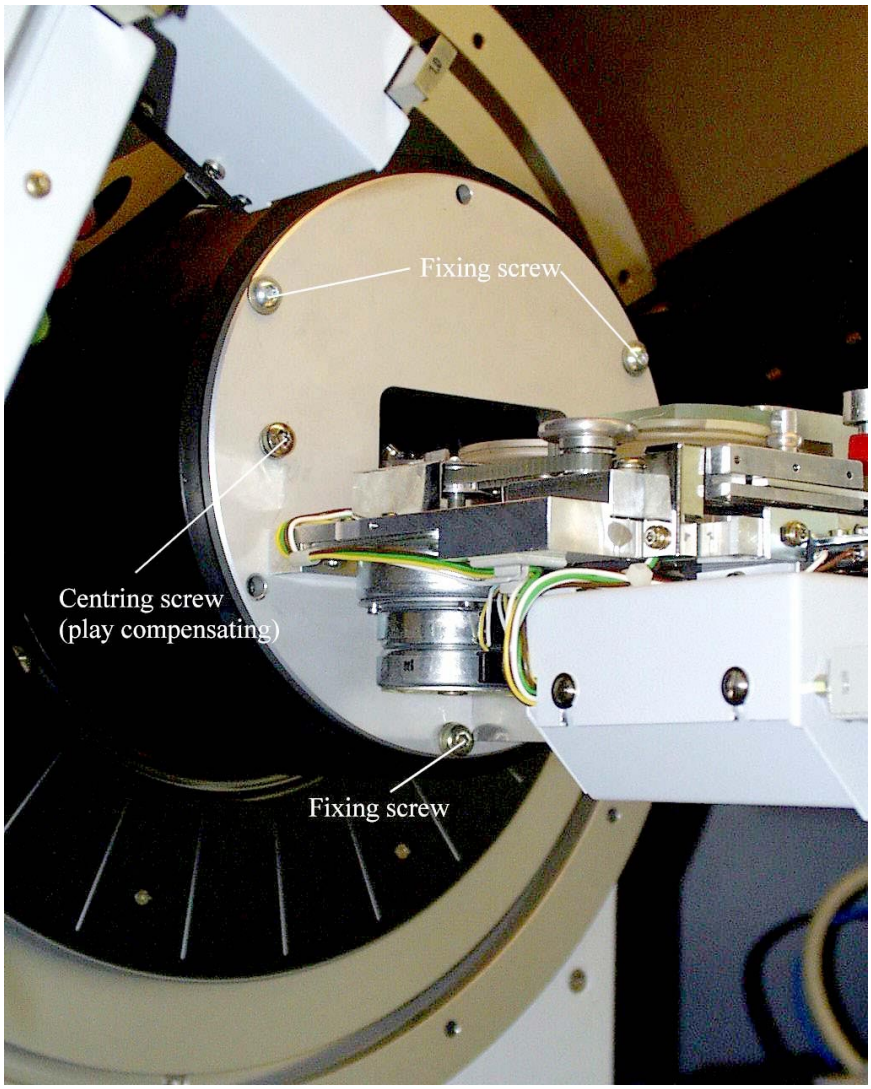


Fig. 1-10: Mechanical installation of 9 Position Sample Stage

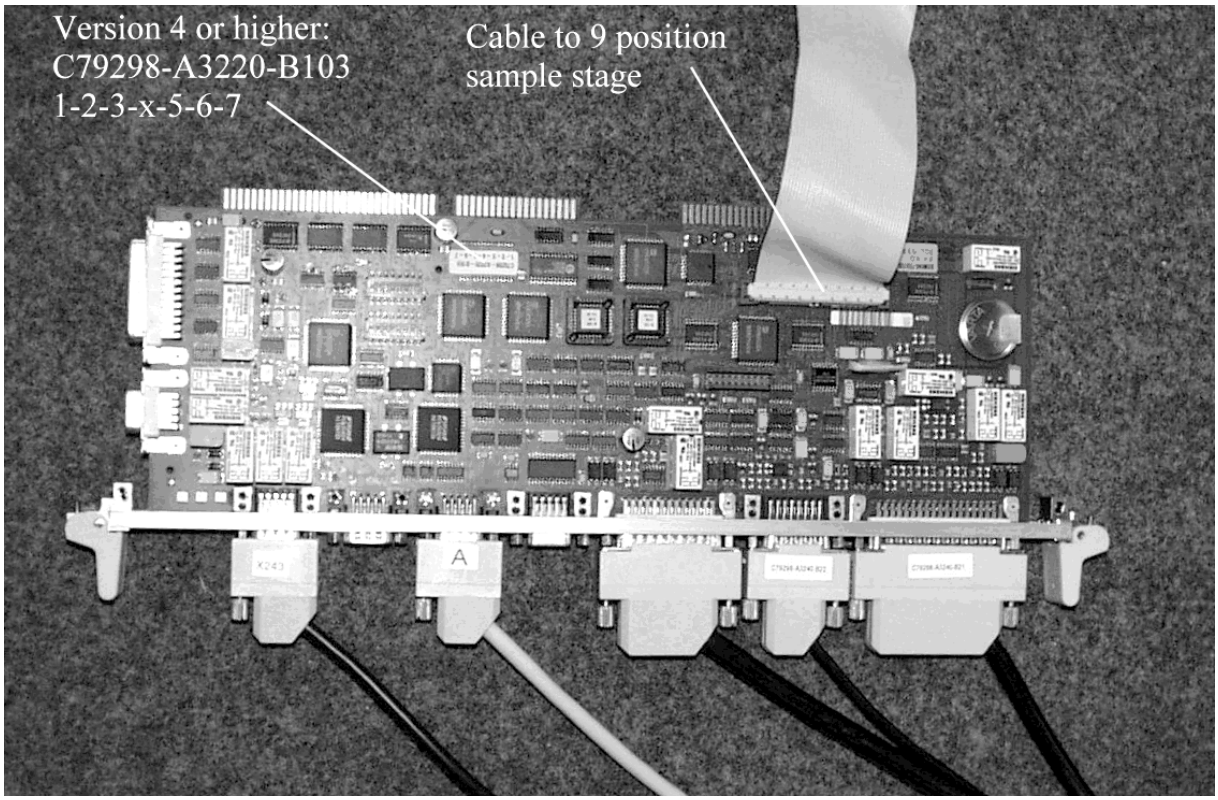


Fig. 1-11: Check of IO-board version and cable connection

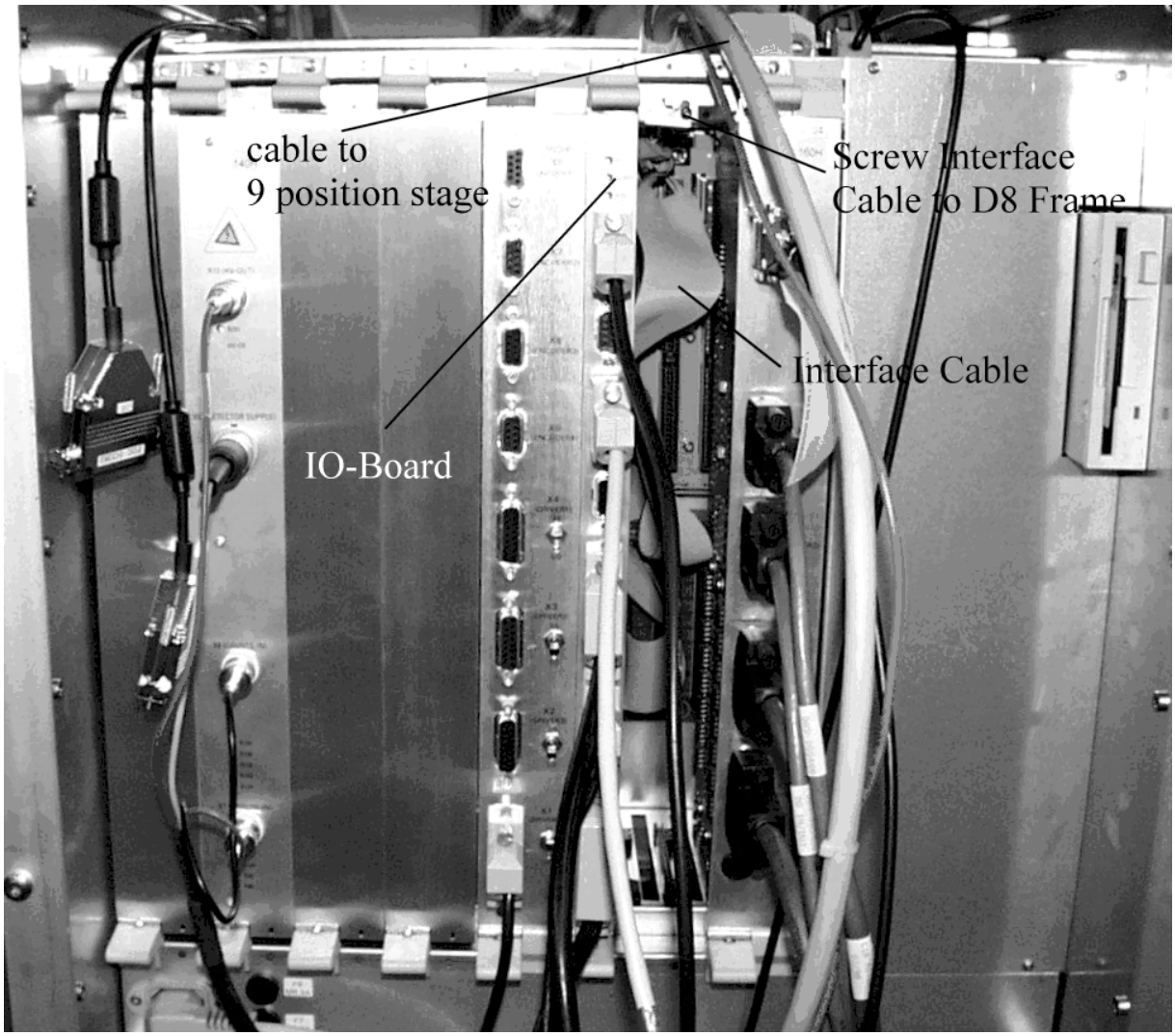


Fig. 1-12: IO-board mounted in D8 frame with all cables connected

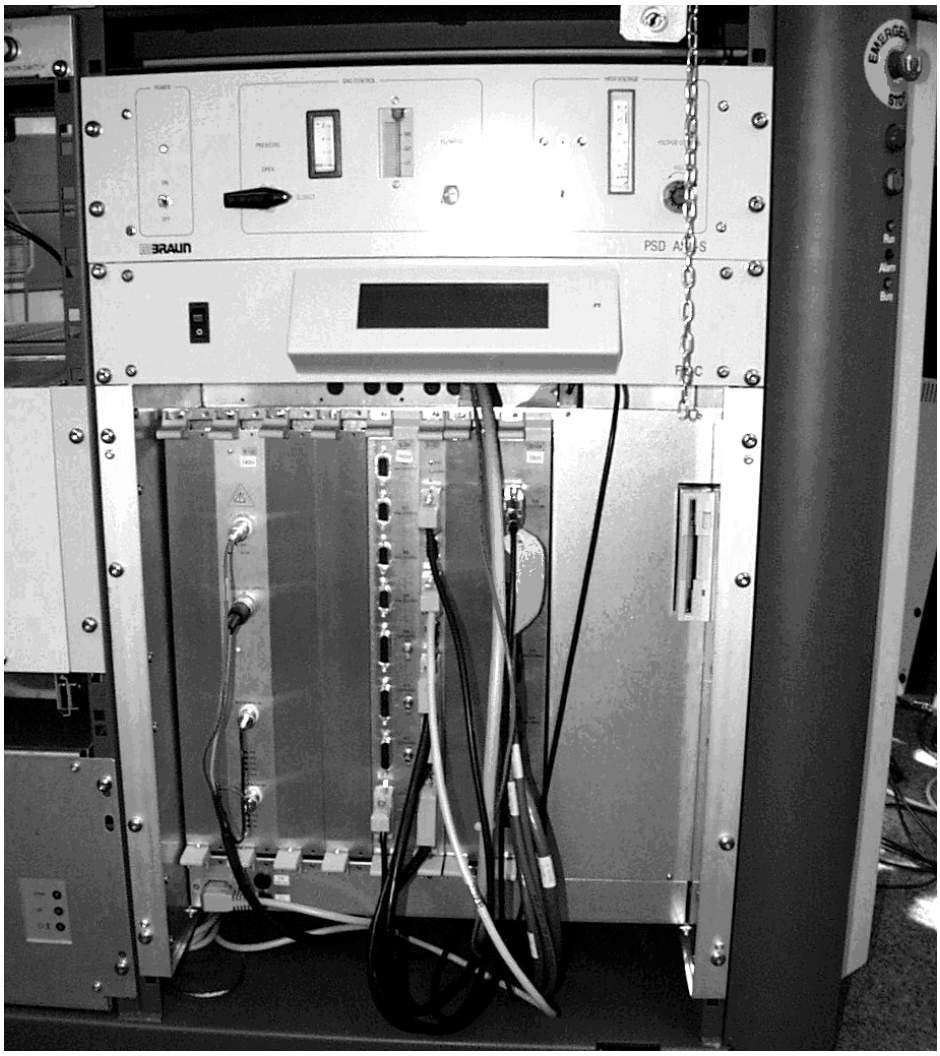


Fig. 1-13: Cable connection of IO-board to 9 Position Sample Stage if a FDC controller is installed

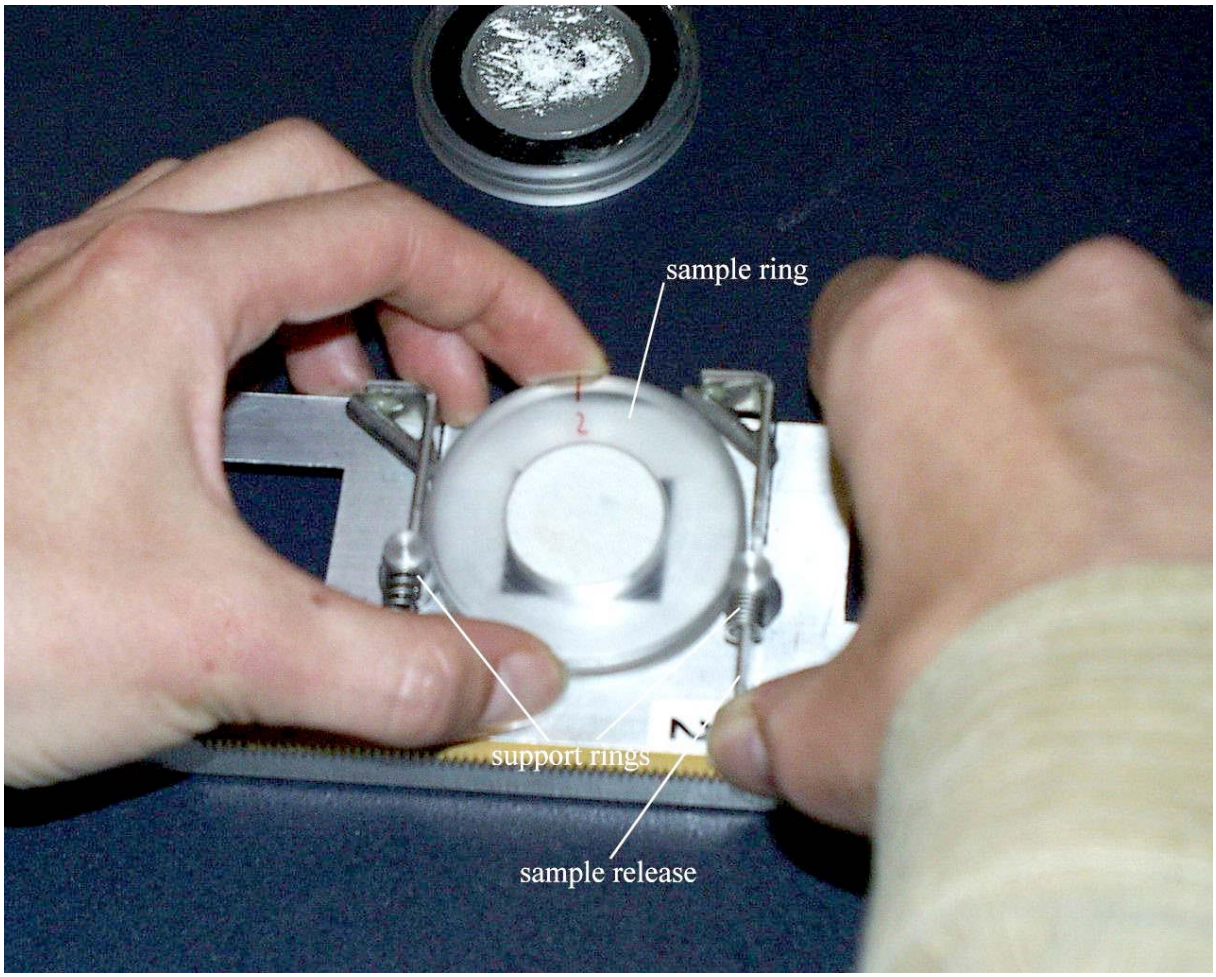


Fig. 1-14: Changing of sample rings

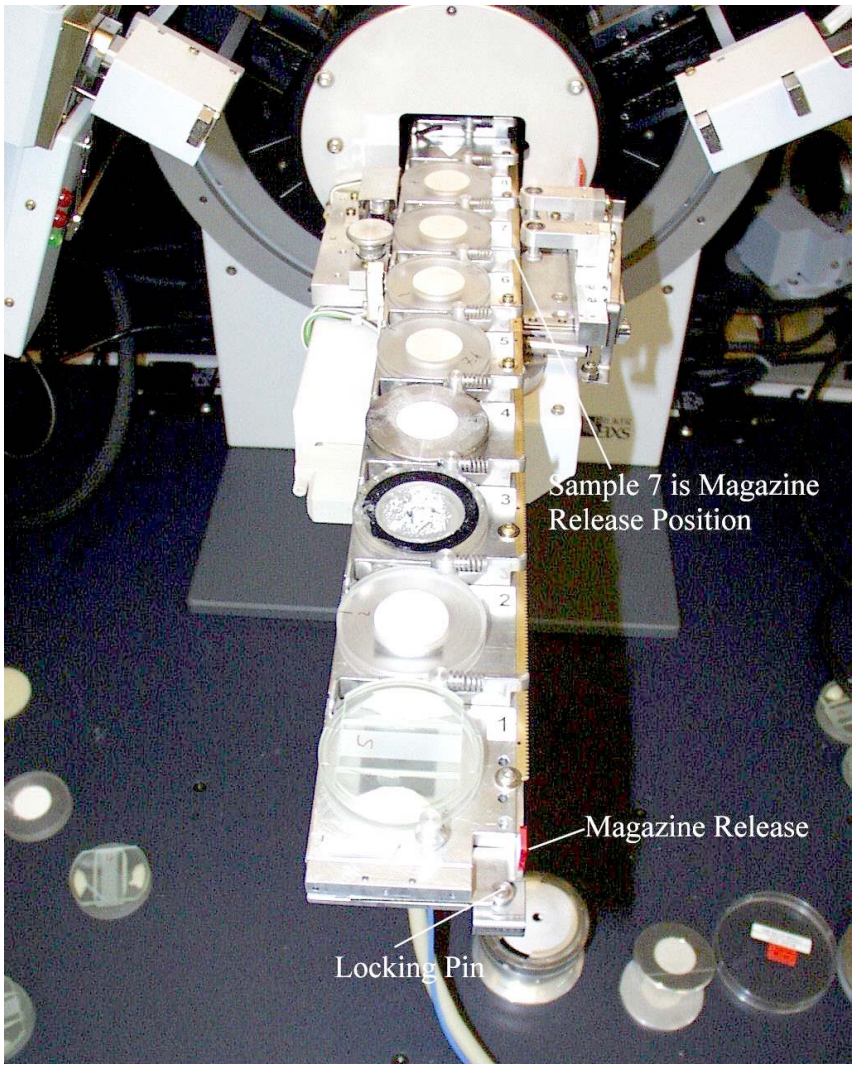


Fig. 1-15: 9 Position Sample Stage in release position

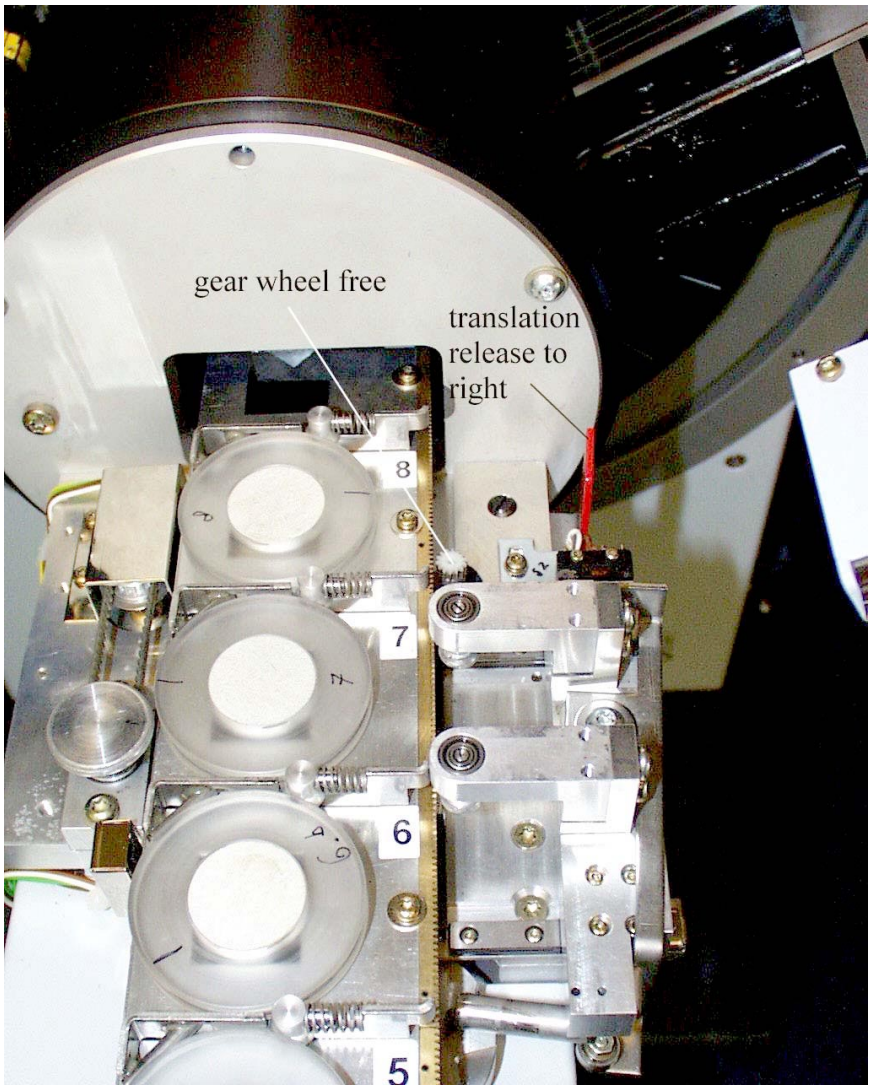


Fig. 1-16: Magazine translation released

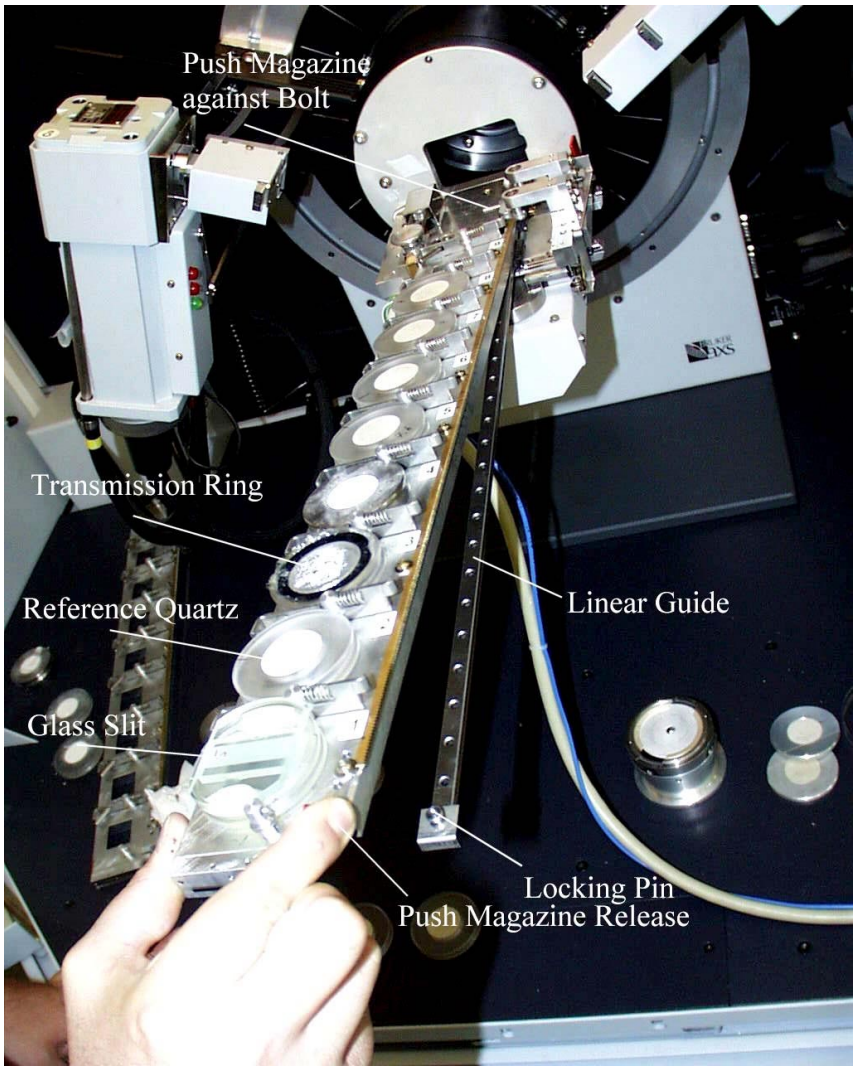


Fig. 1-17: Changing of sample magazine

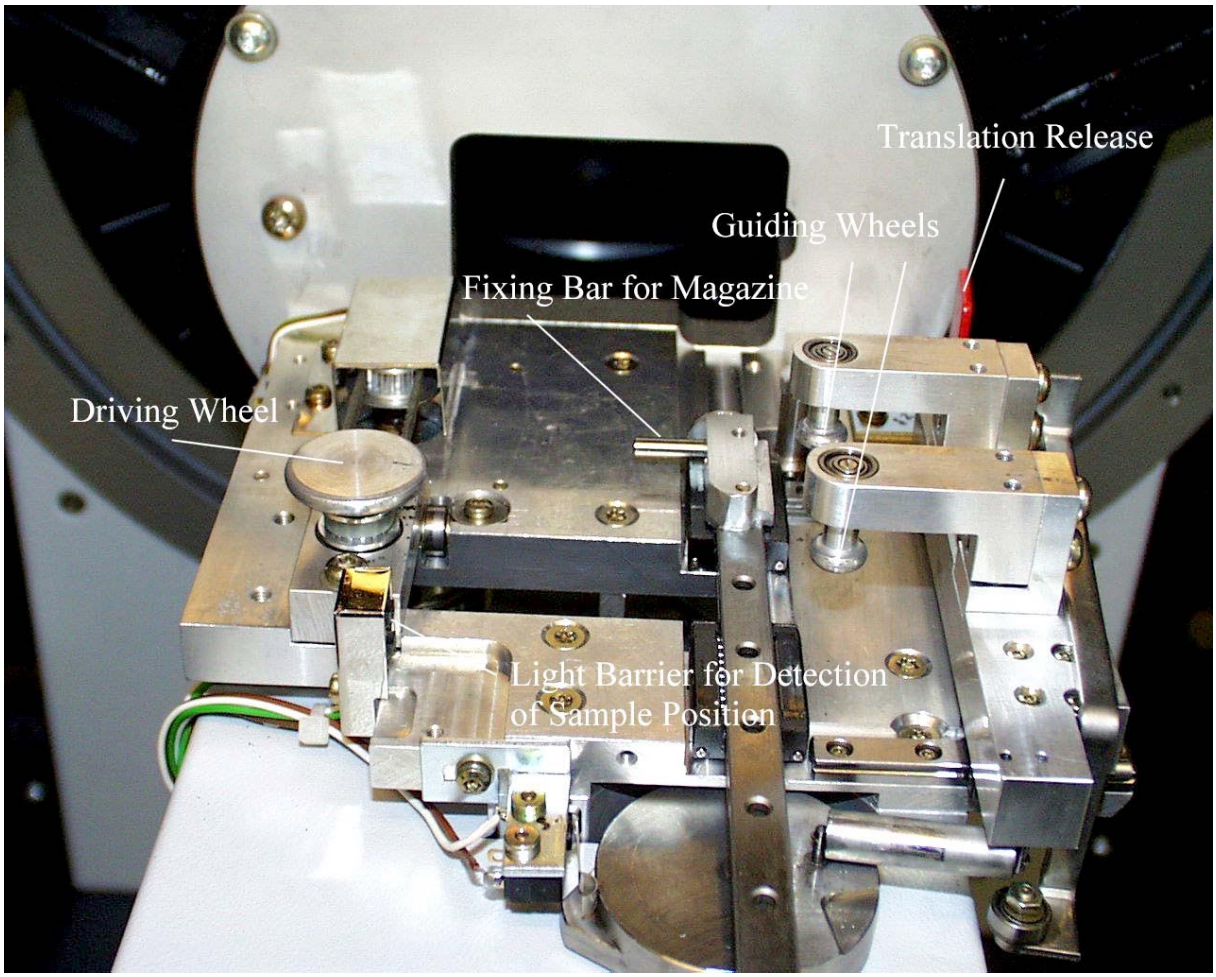


Fig. 1-18: 9 Position Sample Stage without magazine

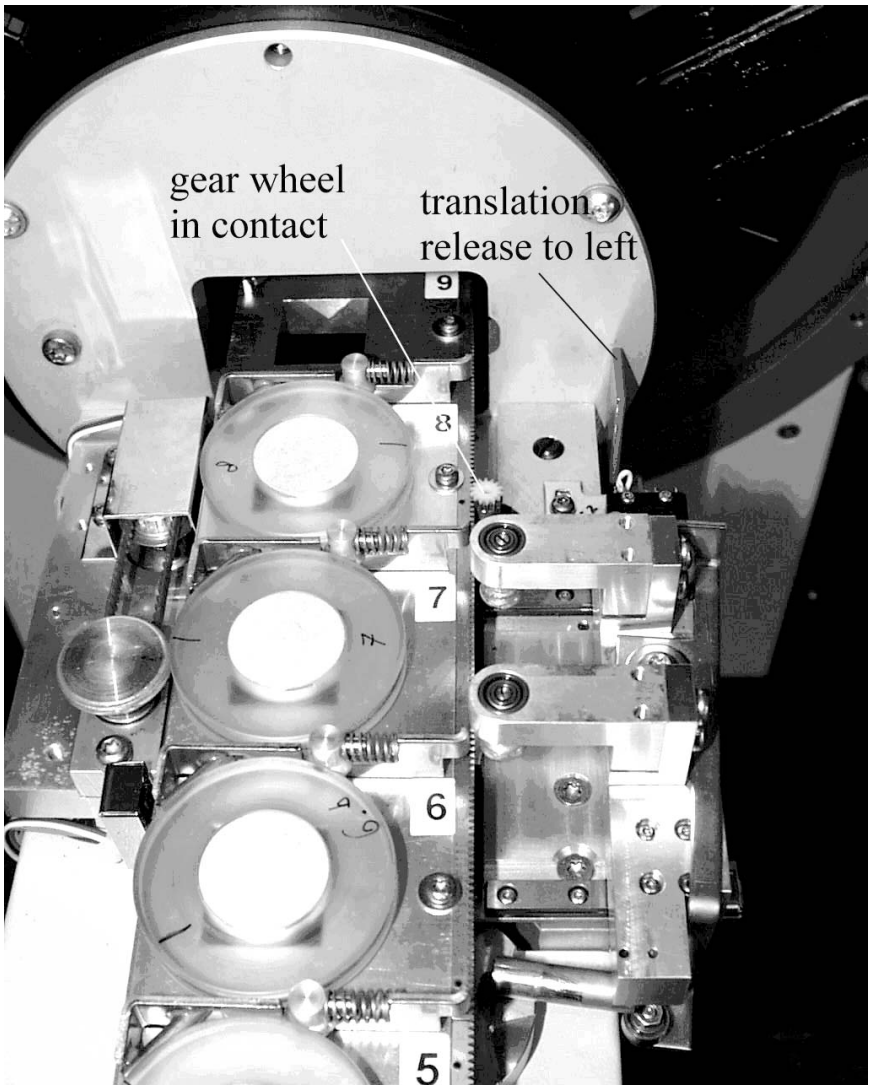


Fig. 1-19: 9 Position Sample Stage with magazine translation in working condition

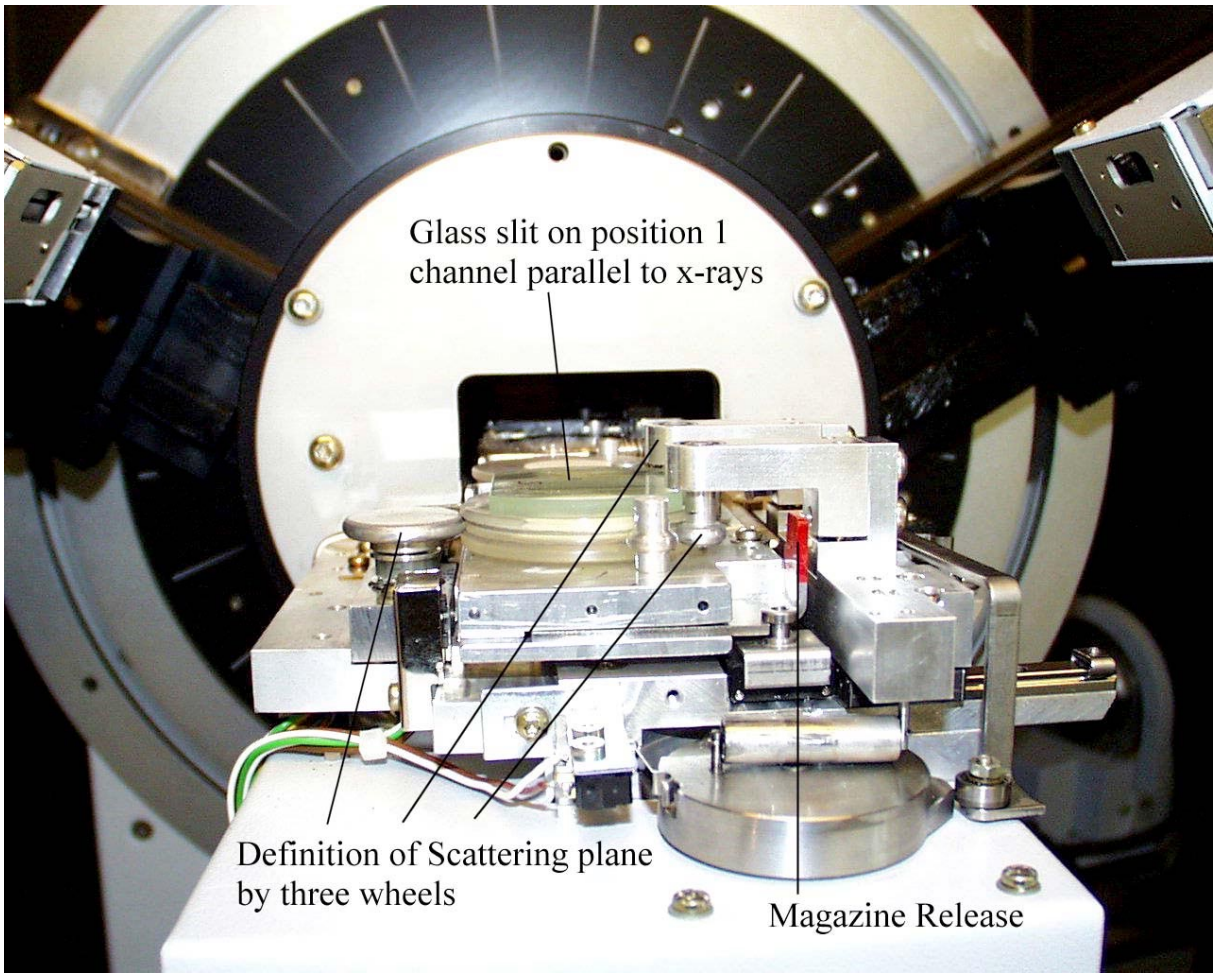


Fig. 1-20: Glass slit in measuring position

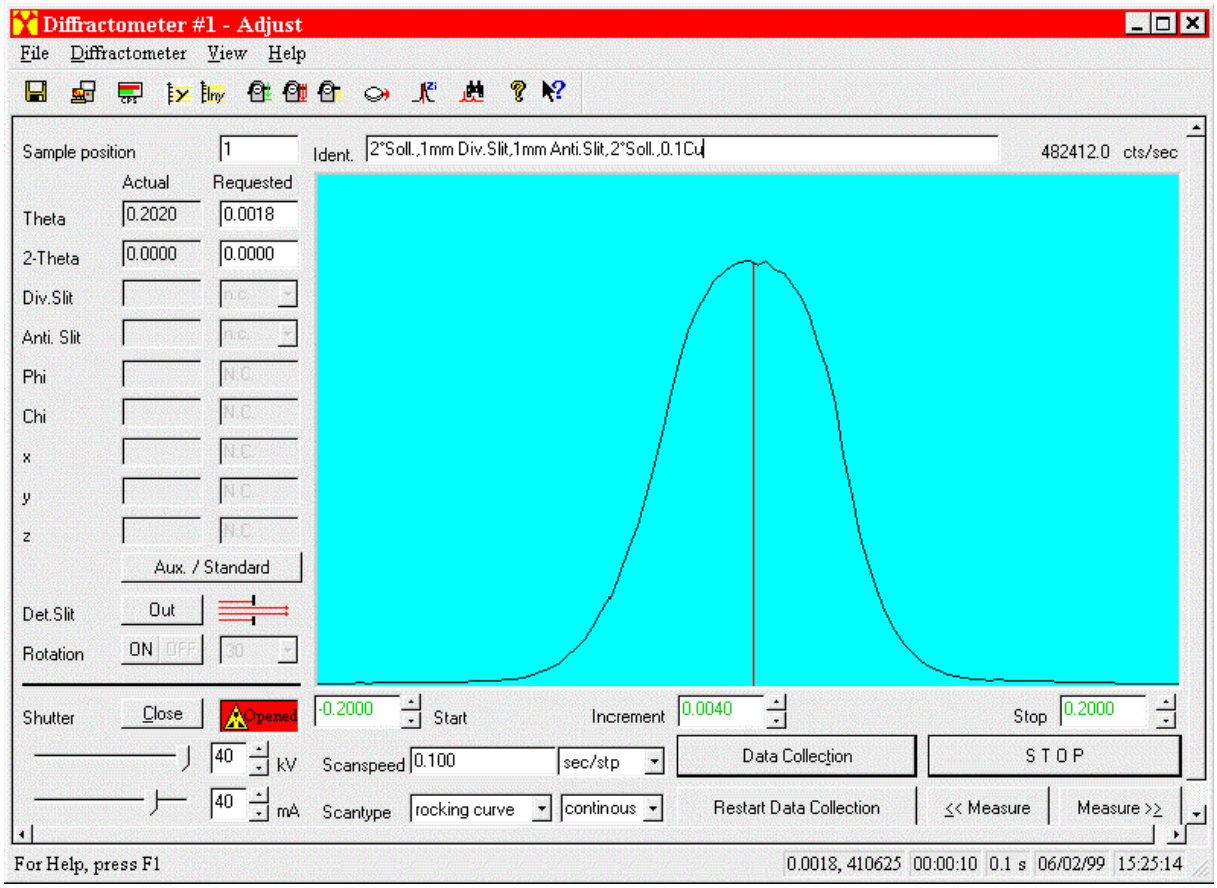


Fig. 1-21: Alignment of θ circle: Rocking curve with glass slit

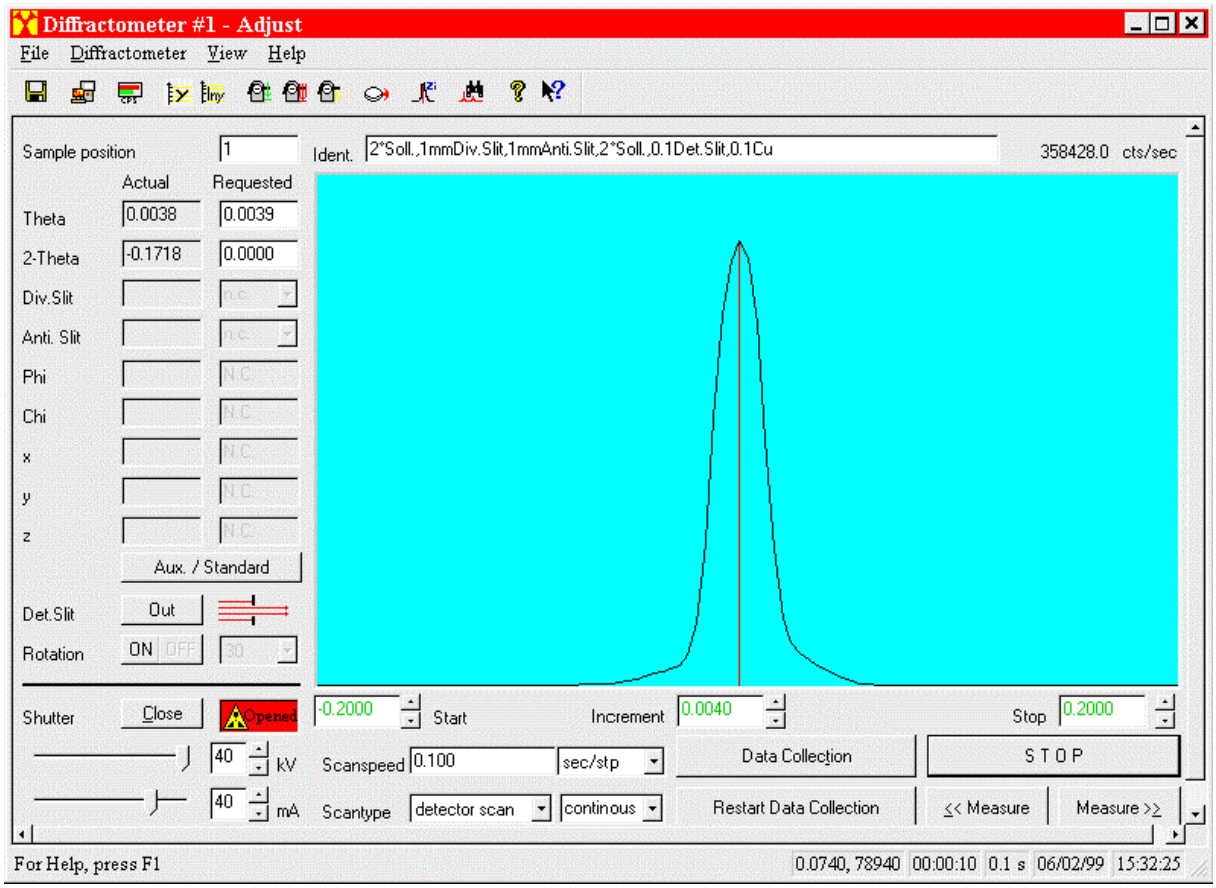


Fig. 1-22: Alignment of 2θ circle: Detector scan with glass slit

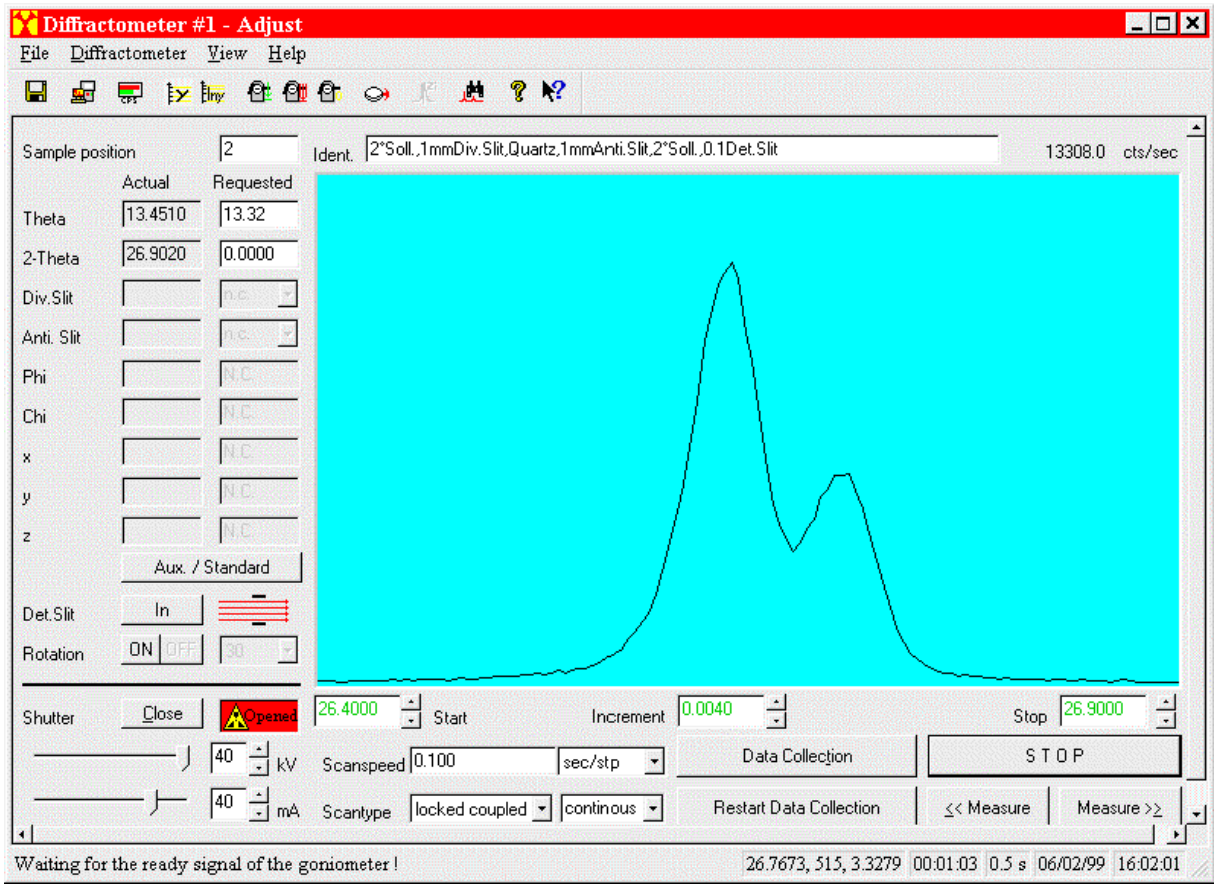


Fig. 1-23: Check of alignment: Coupled scan of 100% quartz reflection

Spare parts list

Tab. 1-1: Spare parts list of the 9 Position Sample Stage

Pos No.	Part number	Description
1	C79298-A3244-B240	Motor, rotation
2	C79298-A3244-B241	Motor, sample change
3	C79298-A3244-B242	Gear, complete
4	C79298-A3244-B243	Drive wheel, complete
5	C79298-A3244-B244	Bearing, complete
6	C79298-A3198-C29	Tooth belt
7	C79298-A3200-C2	Micro switch
8	C79298-A3220-B226	Light barrier module
9	C79298-A3220-B227	Sample stage control
10	K110-C1	Spring
11	K150-C1	Gear motor
12	K170-C1	Miniature guidance
13	K170-C2	Miniature guidance
14	623-2Z DIN 625	Standard bearing

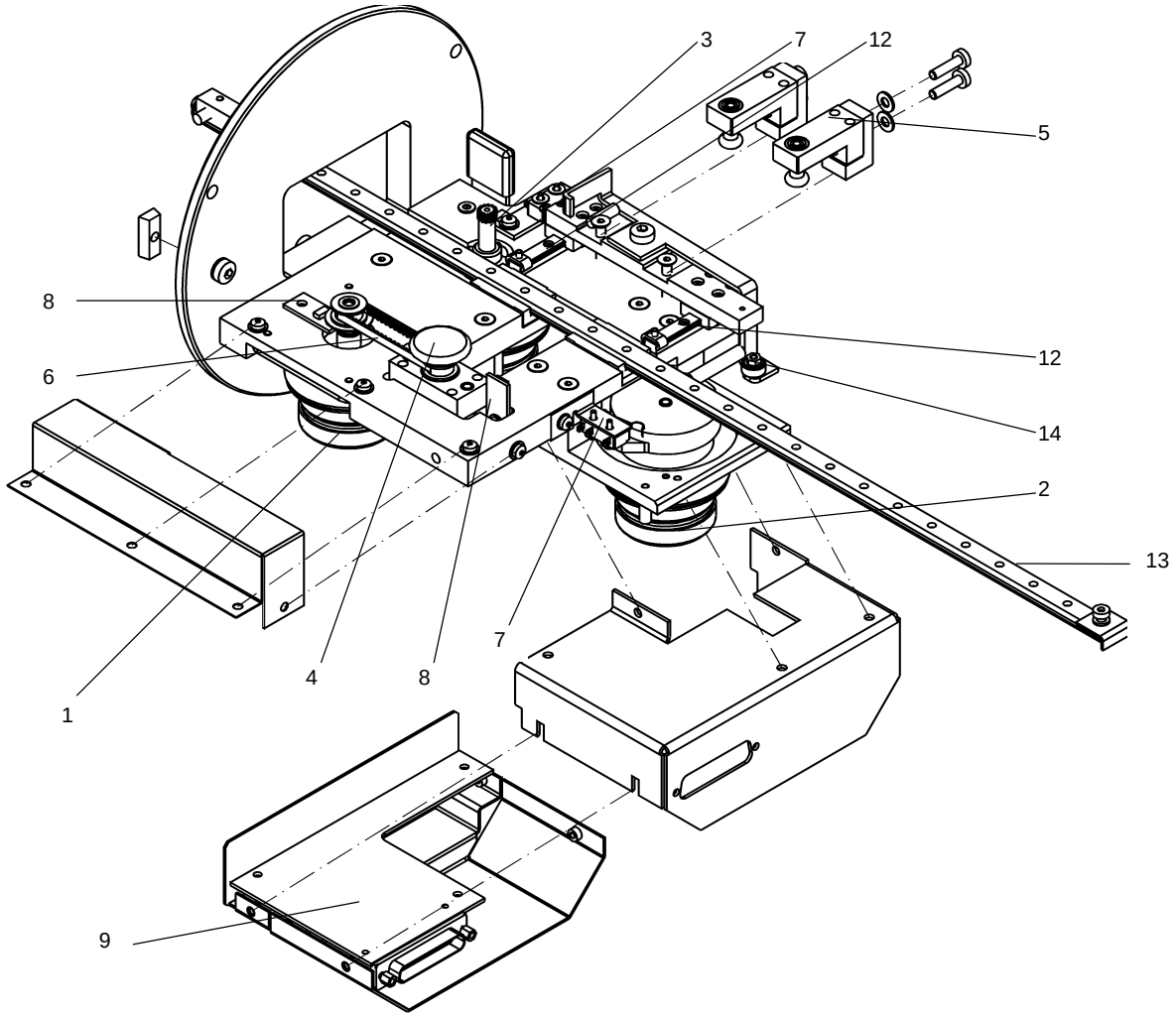


Fig. 1-24: 9 Position sample changer spare parts (I)

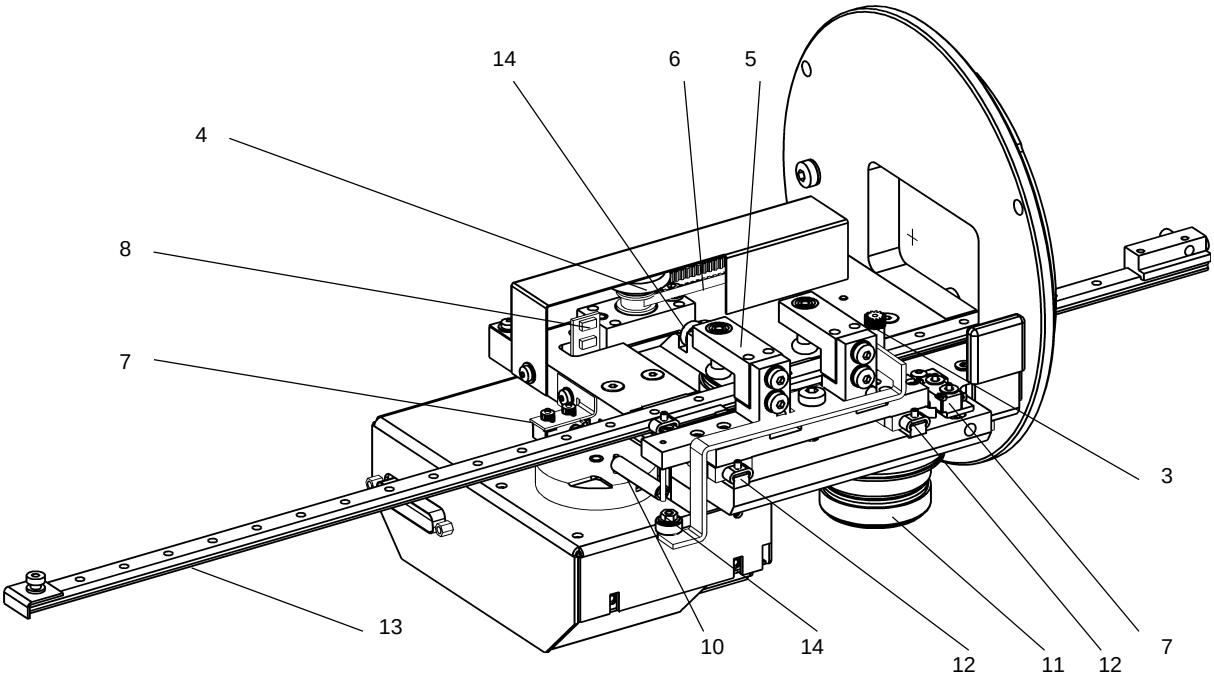


Fig. 1-25: 9 Position sample changer spare parts (II)

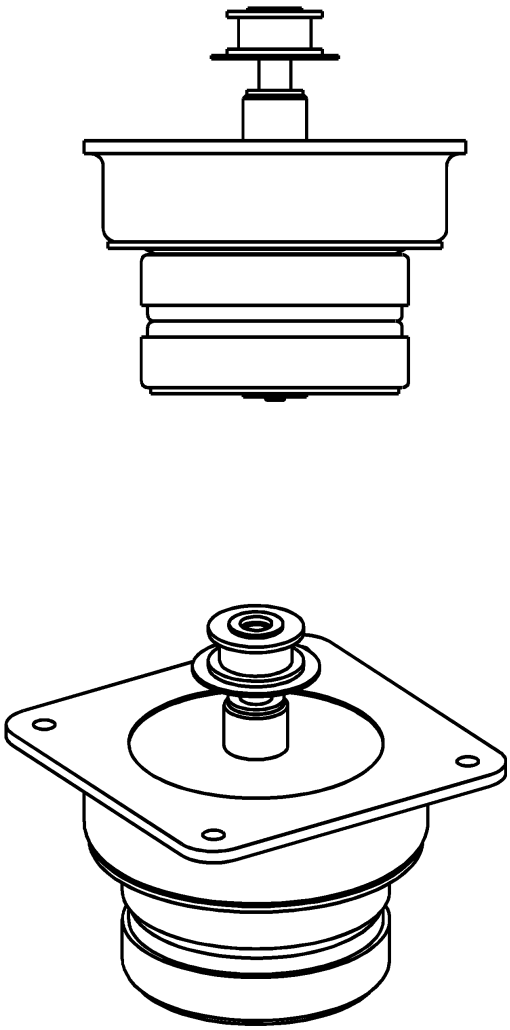


Fig. 1-26: Motor, rotation C79298-A3244-B240

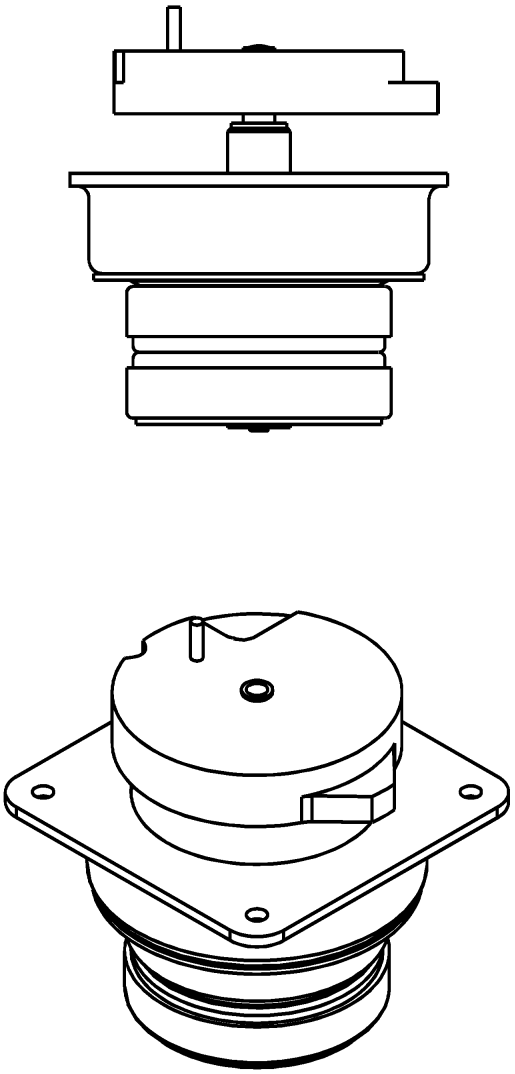


Fig. 1-27: Motor, sample change C79298-A3244-B241

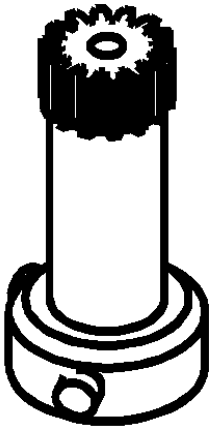


Fig. 1-28: Gear, complete C79298-A3244-B242

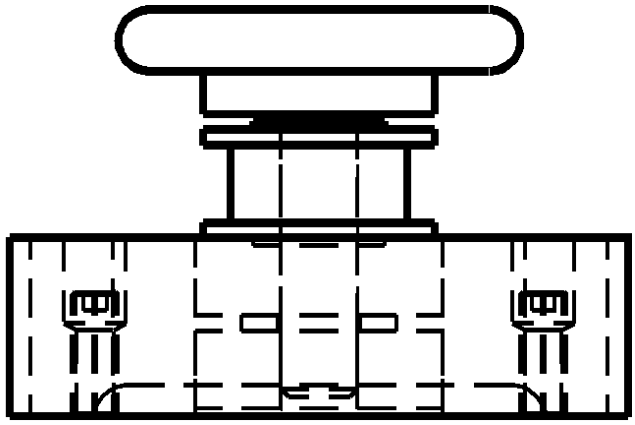


Fig. 1-29: Drive wheel, complete C79298-A3244-B243

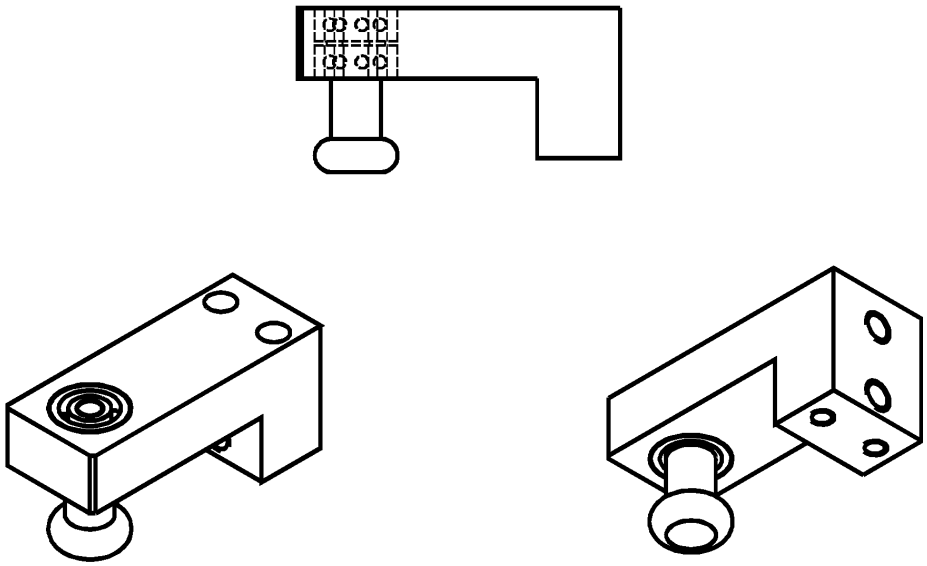


Fig. 1-30: Bearing, complete C79298-A3244-B244

2 Monochromators

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2 Monochromators

Focusing Primary Monochromator

The focusing primary monochromator of Johansson Typ generates $K\alpha_1$-radiation with high intensity and makes it possible to do X-ray diffraction with high resolution. These advantageous properties enable measurements from samples with low scattering ability or with very small amounts of samples. The powder samples can be fixed between two special foils or in thin glass capillaries. Measurements can be also performed with transmission sample changers as well as with capillary sample changers.

Primary Monochromator in Transmission Setup

Geometrical setup

The X-ray tube is fixed by mount 1 (fig. 2-2) with several degrees of freedom on primary track 3 so that the distance from tube 2 and monochromator to sample 10 can be changed along track 3. The primary monochromator (fig. 2-2, 2-5, 2-6, 2-7, 2-8, 2-9) is directly fixed at tube 2. Fig. 2-1 illustrates the beam path and fig. 2-2 pos. 3 the transmission setup (see also fig. 2-7).

Technical Data

Radiation	**$Cu_{K\alpha 1}$**
Crystal	focusing Ge-crystal (Johansson type)
Wavelength	1.540619 Å
a	approx. 120 mm
b	approx. 365 mm
α	6.639°
β	20.643°

Setup

X-long primary track

The X-long primary track 3 (fig. 2-2) enables the use of the goniometer not only in reflection (pos. 1) and transmission (pos. 3) but also in capillary geometry (pos. 2) by simply sliding the tube monochromator unit along the track. For each setup only one monochromator is necessary. For the reflection technique (pos. 1) the secondary focus has to be set to the primary part of the measurement circle. For the transmission setup (pos. 3) the focus is on the sec-

ondary part of the measurement circle and for the capillary technique (pos. 2) the focus is located at the sample P.

Parts of the monochromator

The monochromator (fig. 2-3) consists of the housing 4 with knife edge 2, the wedged exit window 5, holder for aperture 9 and soller holder 10. The crystal 7 (fig. 2-4 and 2-5) is mounted on a holder within the housing.

Preadjustment of the monochromator

Three screws 6a, 6b and 6c in the housing of the monochromator (fig. 2-3) enable all important adjustment steps to bring the crystal into the ideal position. The screws can be positioned very precisely by fine screw threads. Furthermore it is possible to set the knife edge 2 by adjusting screw 15. By using an aperture 8 the reflecting area of the crystal can be restricted and/or optimized. The primary focal distance cannot be adjusted but is optimized by adapter 3.

The preadjustments which make the search for the reflection easier have to be set by the screws 6a, 6b and 6c so that d3 = 24.3 mm (fig. 2-4) and d4 = 20.4 mm (fig. 2-5). The knife edge distance (fig. 2-5) has to be set to d5 = 10.2 mm. After finishing these preadjustments the knife edge, the wedged exit window, the slit holder and eventually the soller holder have to be mounted. The slit holder 9 (fig. 2-3) has to be centered with srew 7.

With these preadjustments the crystal is close to the diffraction position. The search for the Kα-reflection of the Ge-crystal with Cu-radiation is simpler than without preadjustment.

Mounting the monochromator

Monochromator 6, 7, 8 with adapter 5 (fig. 2-2) has to be mounted with four screws 9 flush to the tube housing (cf. fig. 2-7). The upper edges of knife edge housing, adapter and tube housing must be flush (cf. fig 2-7). Slit 8 and soller slit 11 have to be removed if they are mounted (fig. 2-3).

Preadjustment for the transmission setup

The distance between monochromator and detector focus slit must be set to the known B-value (secondary focal length) with a length scale. Tube and monochromator can be moved by loosening screws 3 (fig. 2-6) and by lifting the tube slightly to slide down or lifting strongly to slide up.

Adjustment under radiation

The licence issued for a fully protected device is rendered void when a configuration with primary monochromator is carried out. The safety functions can be bridged by means of a key switch when adjustment work is being carried out on an open radiation path. On no account may the safety functions be bridged by any other means than the key switch. The bridging key for the safety functions should only be issued to the person responsible for radiation protection and should be kept by him or another person in a safe place in order to prevent any improper use. To adjust srews 6a, 6b, 6c, 7 and 15 (fig. 2-3) a long adjustment

tool must be used, however, the housing is very radiation tight. After prealignment (fig. 2-4, fig. 2-5) of the monochromator, adjustment under radiation is usually not necessary.

⚠ **Warning:**
Work carried out on an unsecured diffractometer is dangerous! Adjustment work under radiation may not be executed by any other persons than those professionally exposed to rays and who are kept under medical observation and undergo dosage measurement. During adjustment work the parameters of the tube have to be at the lowest possible values (voltage and current). Long adjustment tools have to be used. Personnel should never put their hands into the primary beam! After the preadjustment, a final alignment under radiation is not necessary. The tube can be set to higher values when the adjustment is not performed under radiation.

Coarse adjustment

A fluorescent screen is to be positioned in the beam path. Move the detector to 90° and set the X-ray generator to 40 kV / 40 mA while safety functions are active. If safety functions are switched off set the generator to 20 kV / 5 mA. Darken the room and give your eyes time to adjust to even low intensities on the screen. If the security functions are active, the beam (at 40 kV/40 mA) can be seen even in normal day light (no direct sunlight).

Diffraction angle of the monochromator

After the preadjustment of screws 6a, 6b and 6c (fig. 2-3) the Bragg angle is approximately correct with the wedged crystal holder. Move the sample holder to 90° and position the fluorescent screen. The exact Bragg angle has to be set by small changes to screw 6a. Turn the screw gradually in 1/8 turns clockwise as well as counterclockwise. Normally the Kα-reflection can be found within +/- ½ turn. The low intensity Kβ-reflection is more than ½ turn from the preadjusted position. Nevertheless it is useful to proof the beam with a Ni-filter. Since the fluorescent screen is not positioned in the focal spot you will see a more or less bright reflection (depends on voltage and current). If the security functions are not active now, by all means switch them on! It is not necessary to optimize for maximum intensity or $K\alpha_1$-$K\alpha_2$-separation with the fluorescent screen. The final alignment with the detector is more precise at maximum radiation security.

Set the relative angle of the tube to the track with the degree of freedom of the tube mount 1 (fig. 2-2) so that the beam hits the goniometer centre. This adjustment can be performed gradually with fluorescent screen control after each step.

The final position is very sensitive to power changes of the tube (resolution, intensity). This is the reason why all adjustments have to be done with the voltage and current settings you will use for later measurements. We recommend a setting of 40 kV and 50 mA, but you can set the generator to 40 kV / 40 mA or other settings too (with possible intensity decrease or

increase). It is important that a new final adjustment is done after tube power changes. Set voltage and current and then wait for approx. 30 minutes until thermal stability of tube and monochromator have been reached. This is also valid before beginning a measurement.

In the next step insert suitable detector slits and absorber: 2 x 0.1 mm Cu absorber, eventually a Ni-filter, 0.1 mm detector slit. Measure the monochromator reflection by means of the program ADJUST (setting 'detector scan'). If you see a real maximum, set the detector to this maximum. If you don't see any maximum, set the detector approx. to the middle of the measured profile.

Now change the aperture to 6 mm to measure the maximum of the integrated intensity. This optimization has to be done with the ratemeter from ADJUST. Firstly optimize the Bragg angle of the monochromator with screw 6a to maximum intensity (open shutter (fig. 2-3) – close door – measure – open door – turn screw – close door – control intensity – etc.). Although the screws have fine threads the monochromator is very sensitive to changes of screw 6a. Note the maximum intensity (pos. B). Now optimize the knife edge for the primary beam path (screw 15) so that the maximum intensity is detected and the primary beam path is completely faded out.

Setting of the secondary focal length

Change the 0.1 mm slit to a 0.05 mm one. Measure the monochromator reflection with a detector scan. Determine the peak intensity I_p (maximum of the reflection profile) and FWHM in position B. Since it is easier to move the aperture holder with the detector on the secondary track than to move the heavy tube on the primary track, move the detector to determine the secondary focal length. Detector and aperture holder have to be moved simultaneously. For this purpose loosen the screws 1a, 1b, 1c and 1d from the holders (fig. 2-7) and move both units. For a reproducible control use a vernier caliper gauge.

The next step is the reduction of the distance between detector and focal aperture to the tube by 2 mm (pos. A). In this position the reflection profile must be scanned and I_p and FWHM has to be determined. The same procedure is to be done in position C (2 mm away from position B). Compare the determined values for I_p and FWHM in the positions A, B and C. The closer the detector aperture is to the focus, the greater are the values for I_p and the smaller for FWHM. If the position is found for the maximum I_p and minimum FWHM, the same procedure can be done with displacements of +/- 1mm. The secondary focal length can be determined with an accuracy of +/- 1 mm under favorable conditions even better. This procedure is worthwile if you think that resolution is very important. The FWHM of the monochromator reflection can be further reduced by means of screw 6c (fig. 2-3). Follow these steps:

1. Tilt crystal with screw 6c by a small amount (approx. ¼ turn or less).
2. Insert 6 mm detector slit
3. Set maximum intensity with screw 6a
4. Scanning detector with 0.05 mm slit
5. Determine peak intensity I_p of profile and FWHM
6. The best setting is reached with lowest FWHM and highest I_p

The focus of a well aligned monochromator is shown in fig. 2-12. The parameters are 0.05 mm detector aperture, 0.2 mm Cu-absorber, 50 kV, 40 mA.

Setting the distances for transmission geometry

Before you make any changes, measure the distance $\tilde{B}$ between monochromator (middle of the monochromator in fig. 2-4 -> d1, d2) and detector aperture (note the place where the slit aperture is within the aperture unit). For transmission geometry set slit holder and detector to the measuring circle (D = 435 mm). For a measuring circle diameter of 435 mm set the detector aperture to a distance of 217.5 mm from goniometer centre. The detector must be moved so that it is close to the aperture holder. Furthermore move the tube monochromator unit so that the distance from the goniometer centre is exactly $\tilde{B}$ -217.5 mm.

Adjustment of the primary beam to the goniometer centre

Replace the sample holder and mount a transmission holder. Use the glass slit for the adjustment of the beam to the goniometer centre. Firstly determine the correct zero position of the detector by a detector scan (measurement of the focal spot profile) and by setting the ZI-value for the 2ϑ-circle. Then set the glass slit into the sample holder and determine the position with maximum intensity with ADJUST (rocking curve setting). For this purpose use a 6 mm detector slit to determine the integrated intensity. Check whether the maximum intensity can be improved by tilting the tube with respect to the primary track (screw 2 in fig. 2-6). Maximize the intensity value with an alternating setting of screw 2 and rocking curve measurement. Determine the ZI-value for rocking curve and detector scan as well.

Primary monochromator in capillary setup

Geometrical setup

Move the tube with monochromator for changeing from transmission to capillary geometry along the primary track 217.5 mm away from the sample (distance from the middle of the monochromator crystal to the goniometer centre is the secondary focal length $\tilde{B}$). Fig. 2-8 illustrates the beam path for a capillary setup (also fig. 2-9, fig. 2-2 pos. 2). Since the focus is at the goniometer centre, you have to build a capillary sample holder onto the goniometer.

Adjustments

All adjustments of the monochromator must be performed as described in the subchapter of the transmission geometry. After that the distance from monochromator to the sample must be enlarged. To reduce the adjustment work after changing the setups, the monochromator beam should be parallel to the primary track and should go through the goniometer centre. This procedure will be described later in the subsection about reflection geometry. After the movement of the tube monochromator unit on the primary track, check with the glass slit whether the beam goes through the goniometer centre – otherwise small corrections have to be done, i.e. the relative angle of the tube with respect to the primary track must be changed. Use screw 2 (fig. 2-6) for this kind of adjustment.

System setup

Fig. 2-9 demonstrates the system setup for capillary measurements. Since the focus is at the goniometer centre, you have to build a capillary sample holder onto the goniometer. Furthermore you have to mount the antiscatter tube with additional capillary tube (with slit exit window) directly in front of the capillary sample. With this configuration you get maximum intensity with minimal noise.

Primary monochromator in reflection setup

Geometrical setup

Move the tube with monochromator after changing from transmission to reflection geometry along the primary track 435 mm away from the sample (distance from the middle of the monochromator crystal to goniometer centre is the secondary focal length $\tilde{B}$ + 217.5 mm). Fig. 2-10 illustrates the beam path for a reflection setup (fig. 2-2 pos. 1 and fig. 2-11). The focal spot of the crystal is on the primary side of the mesuring circle. Use a sample holder for reflection geometry.

Adjustments

All adjustments of the monochromator are to be done as presented in the subchapter about transmission geometry. After these adjustments, enlarge the distance of the monochromator as described above. To reduce the adjustment work during the changing between the different setups, the beam path should be set parallel to the primary track and should go through the goniometer centre simultaneously. This adjustment will be described in this subchapter. Check with the glass slit, whether the monochromator reflection hits the goniometer centre after movement of the tube monochromator unit along the primary track. Otherwise small corrections are necessary, i.e. the relative angle of the tube with respect to the primary track has to be changed. This adjustment must be done with screw 2 (fig. 2-6) and maximum intensity through the glass slit must

be set (focal spot, glass slit, detector slit have to be on a line).

Adjustment of the focus slit

Fig. 2-11 illustrates where the primary focus aperture must be. Firstly the cover, soller and slit must be removed from holder 2. Check with a fluorescent screen, which you have to put into the slit holder, whether the focal spot is in the middle of the slit holder. If this condition is not fulfilled, the slit holder can be moved up or down with screws 3a, 3b.

After that focus slits with decreasing slits widths (e.g. 2 mm, 1 mm, 0.6 mm, 0.2 mm, 0.1 mm) must be put into the holder and they have to be adjusted with screws 3a, 3b for maximum integrated intensity. If the monochromator is adjusted very well, 100% beam intensity will go through the 0.2 mm slit. If required the soller can be inserted. After the adjustment, mount the cover.

Adjustment of the antiscatter tube

After the adjustment of the focus aperture, the antiscatter tube 4 (fig. 2-11) can be installed. The antiscatter tube reduces the part of air scattering, which reaches the detector. This leads to an improvement of the signal/background ratio. The tube can be aligned with screw 5 so that the beam can pass the tube without intensity loss.

Adjustment of the beam path parallel to the primary track

In the ideal case two conditions must be fulfilled:

a) the monochromator reflection must be parallel to the primary track

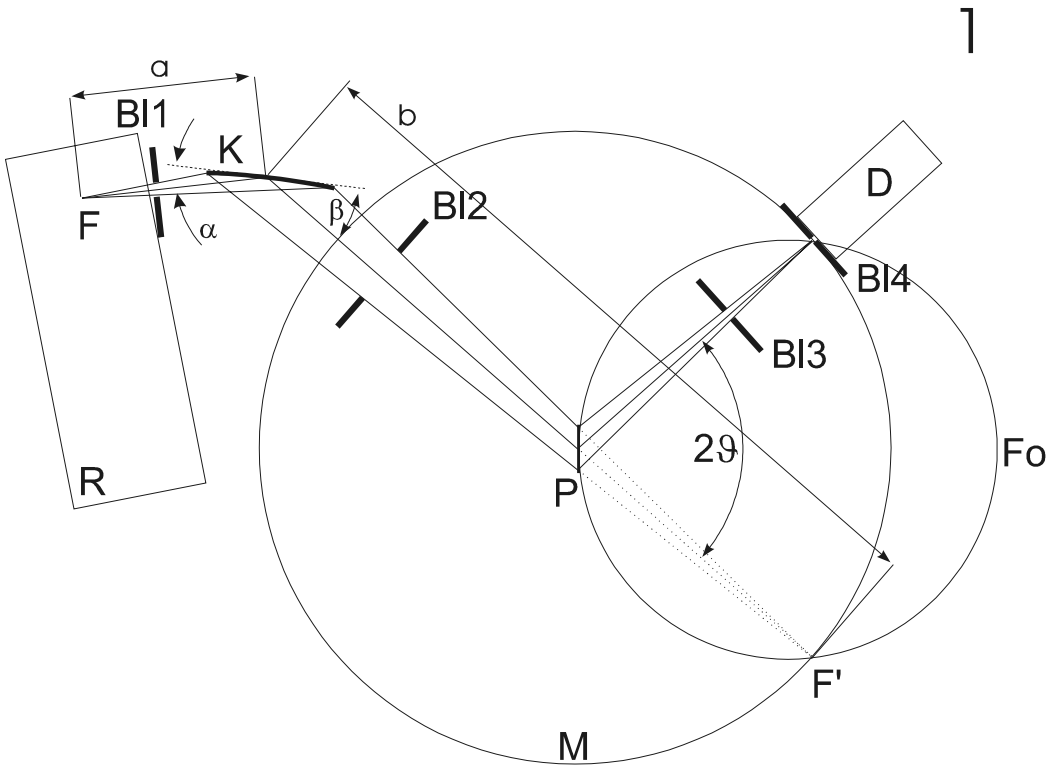
b) the beam path hits the goniometer centre

In this ideal case an adjustment is not necessary after movements of the tube monochromator unit. Production tolerances, high torques and forces are the reasons that these ideal case conditions cannot be reached. After movements along the primary track, small adjustments are always necessary.

Check firstly the difference of the actual setup from the ideal position. For this purpose move the tube with monochromator near to the sample holder (small distance) with a fluorescent screen. Remove the slit holder for the focus slit (fig. 2-12). Mark the middle position of the beam on the screen. After that, move the tube with monochromator as far as possible to the end of the primary track (great distance) and mark the middle of the beam on the screen as well. Determine the inclination of the tube from the value a of both marks. Incline the tube until the beam does not change any more. During this procedure the beam can be moved away from the goniometer centre. This difference can be corrected by translating the tube along the tube mount. By means of the fluorescent screen you can reach a rough adjustment which is good enough to make a fine adjustment with the detector.

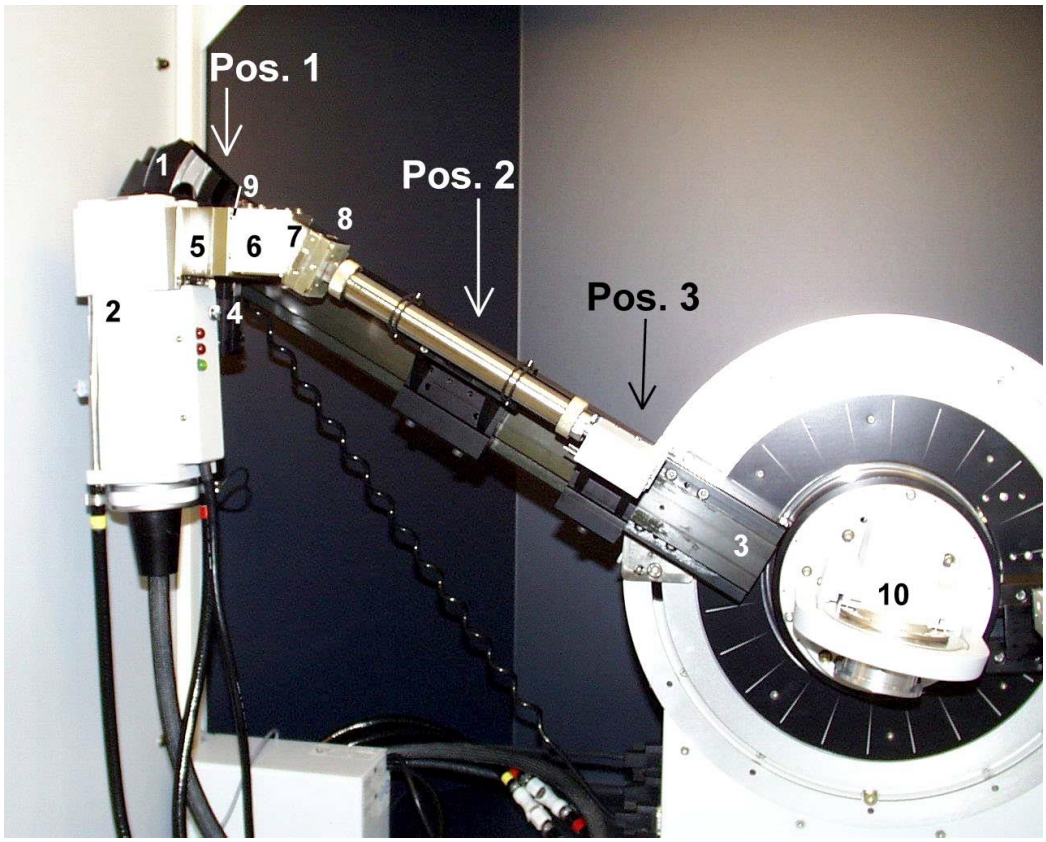
The procedure for the fine adjustment is the same as described before. The alignment by means of a detector is much more precise than with a fluorescent screen. Use the glass slit procedure for setting the beam path to the goniometer centre. If your adjustment is good enough, the value of α during the movement along the track is approx. 0.05° - 0.1°. The adjustment of the focal aperture for reflection measurements must be performed after this adjustment or must be repeated. Small deviations after the change of the modi (transmission, capillary, reflection) can be corrected by tilting the tube (screw 2 fig. 2-6).

Figures



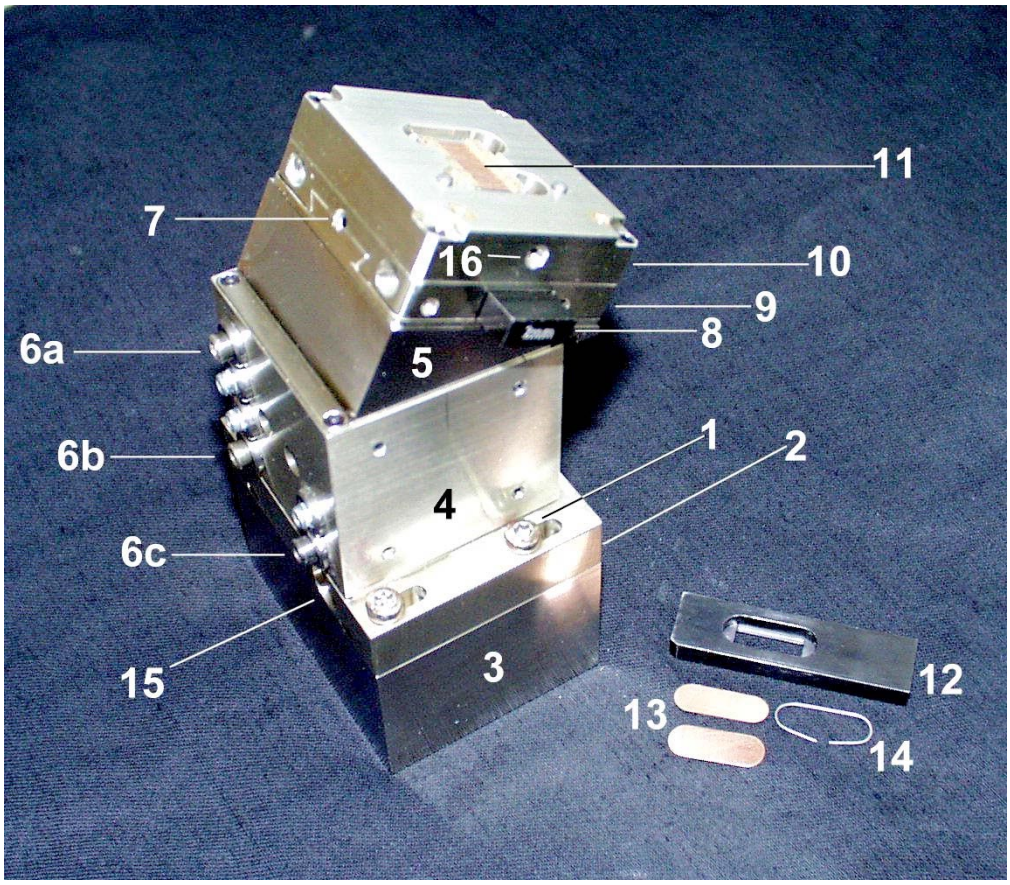
Bl1) Entrance aperture
Bl2) Exit slit
Bl3) Antiscatter slit
Bl4) Focus-detector slit
a) Primary focal length
b) Secondary focal length
D) Detector
F) Tube focus
F') Focus
P) Sample
R) X-ray tube
K) Monochromator crystal
M) Measuring circle
Fo) Focusing circle
α) Entrance angle
β) Exit angle
2ϑ) Diffraction angle

Fig. 2-1: Beam path for transmission



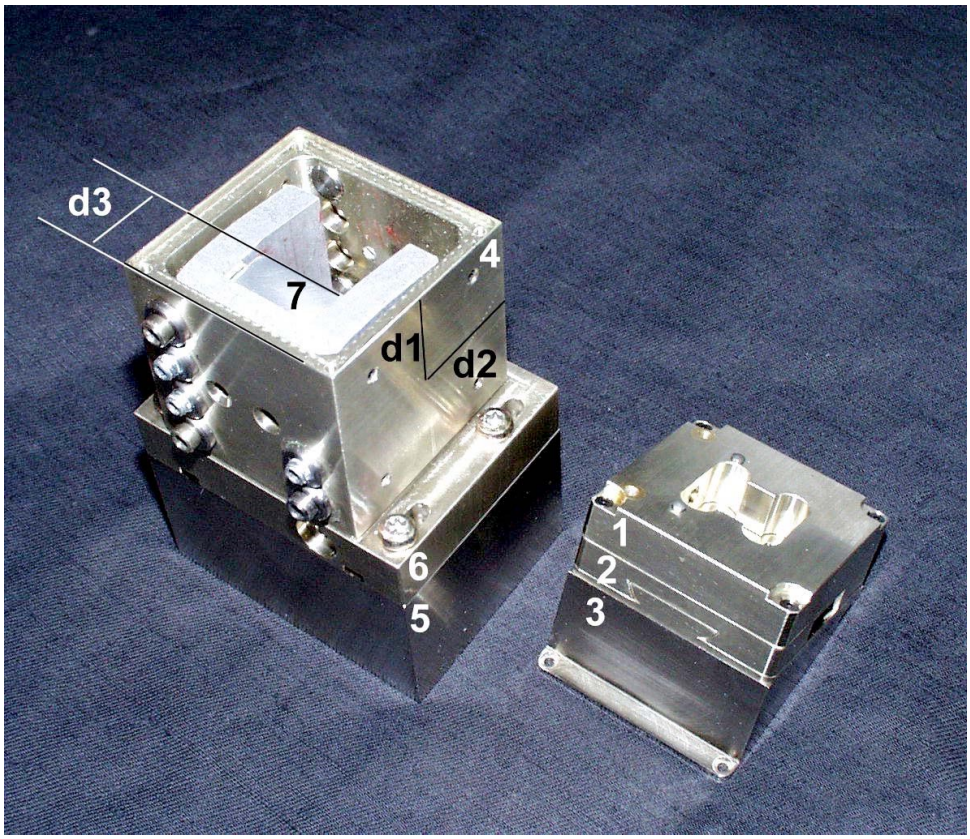
1) Holder for X-ray tube
2) X-ray tube
3) Primary track
4) Interlock
5) Adapter for monochromator
6) Monochromator housing
7) Wedged exit window
8) Aperture and soller holder
9) Fixing screws
10) Sample

Fig. 2-2: Setup for reflection



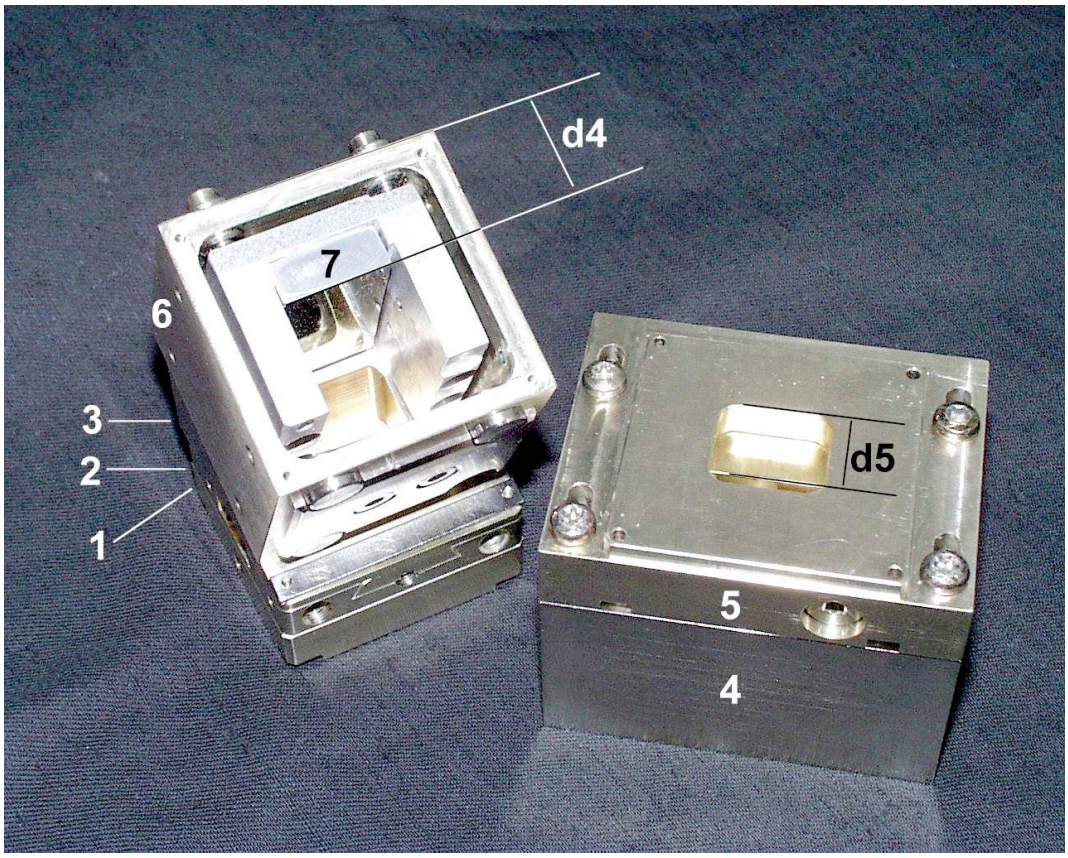
1) Fixing screws
2) Knife edge holder
3) Adapter
4) Monochromator housing
5) Wedged exit window
6) Fine screw threads for positioning the crystal
7) Screw for positioning of the divergence slit
8) Divergence aperture
9) Divergence slit holder
10) Soller holder
11) Soller slit
12) Slit
13) Absorber
14) Clamp for absorber
15) Screw for positioning the knife edge
16) Fixing screw for the soller

Fig. 2-3: Parts of the primary monochromator



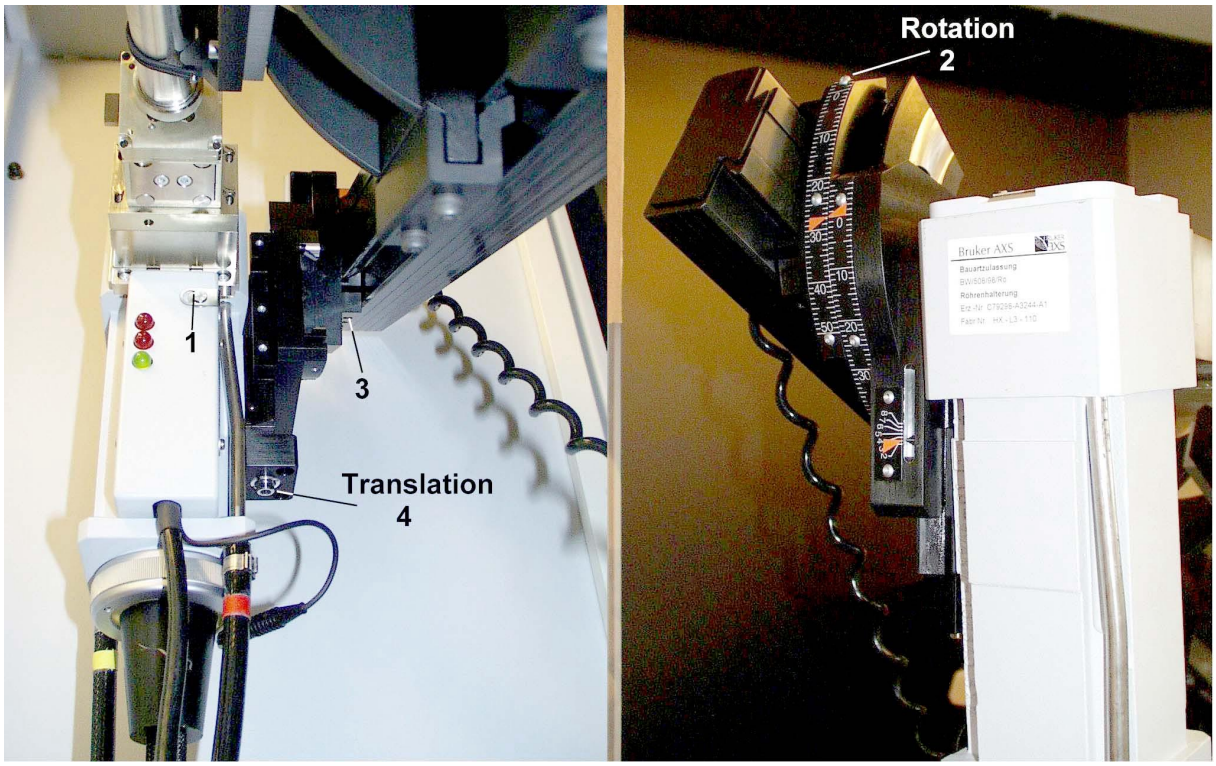
1) Soller holder
2) Slit holder
3) Wedged exit window
4) Monochromator housing
5) Adapter
6) Knife edge
7) Crystal mounted on wedged holder
d1 = 23.2 mm
d2 = 27.8 mm
d3 = 24.3 mm

Fig. 2-4: Crystal position inside the housing



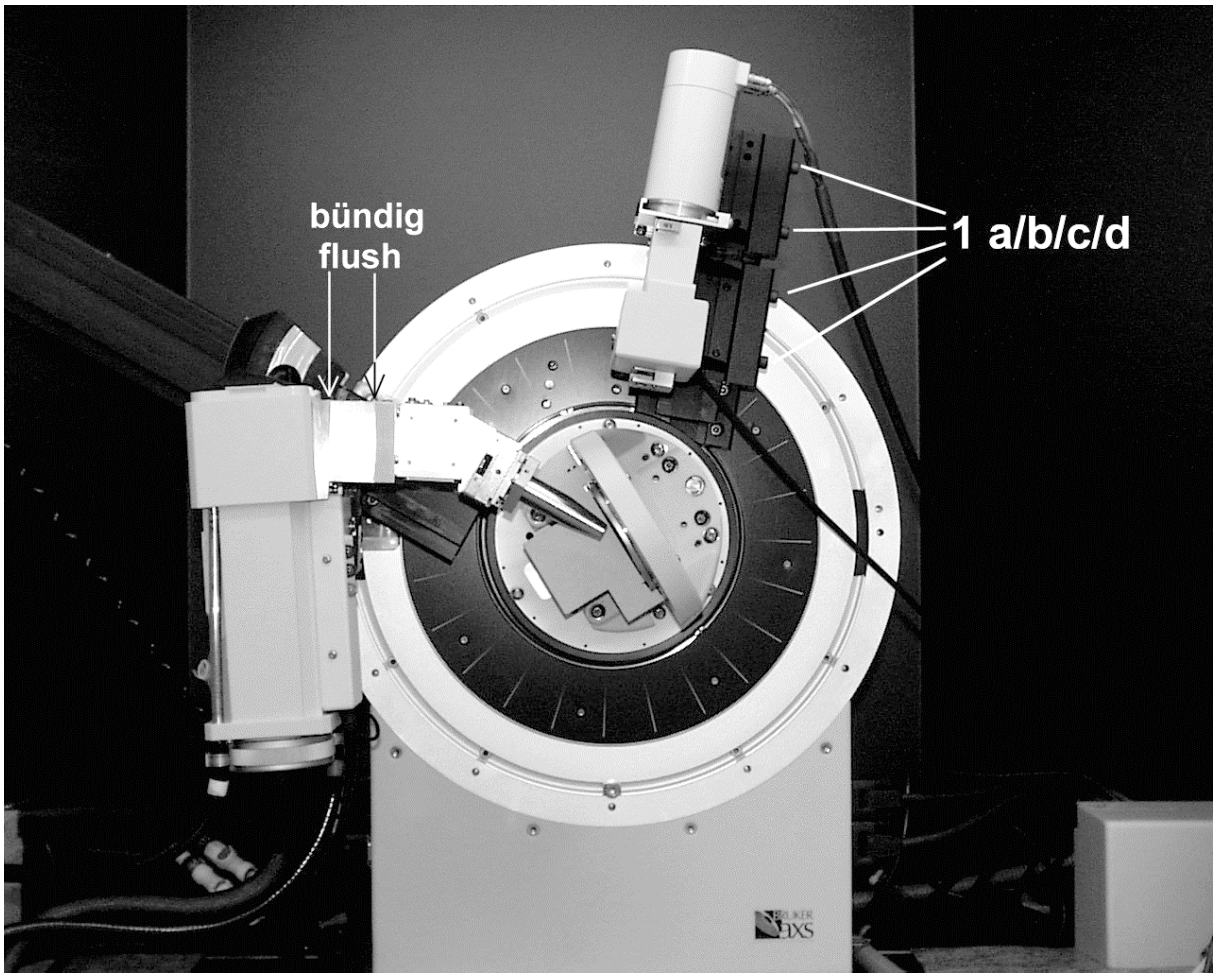
1) Soller holder
2) Slit holder
3) Wedged exit window
4) Adapter
5) Knife edge
6) Monochromator housing
7) Crystal mounted on wedged holder
d4 = 20.4 mm
d5 = 10.2 mm

Fig. 2-5: Crystal position and alignment of knife edge



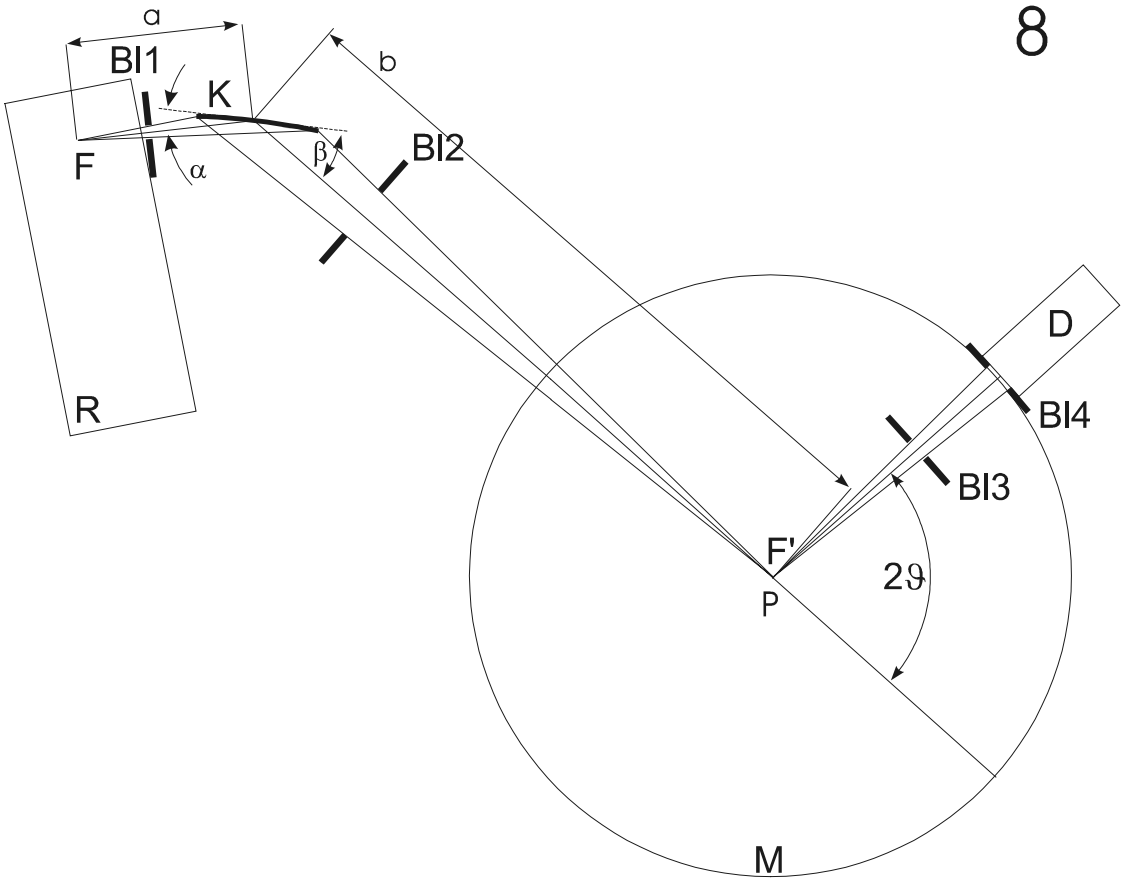
1) Interlock
2) Screw for tube inclination
3) Fixing screw for tube mount at the primary track
4) Screw for tube translation in tube axis direction

Fig. 2-6: Alignment screws for the X-ray tube



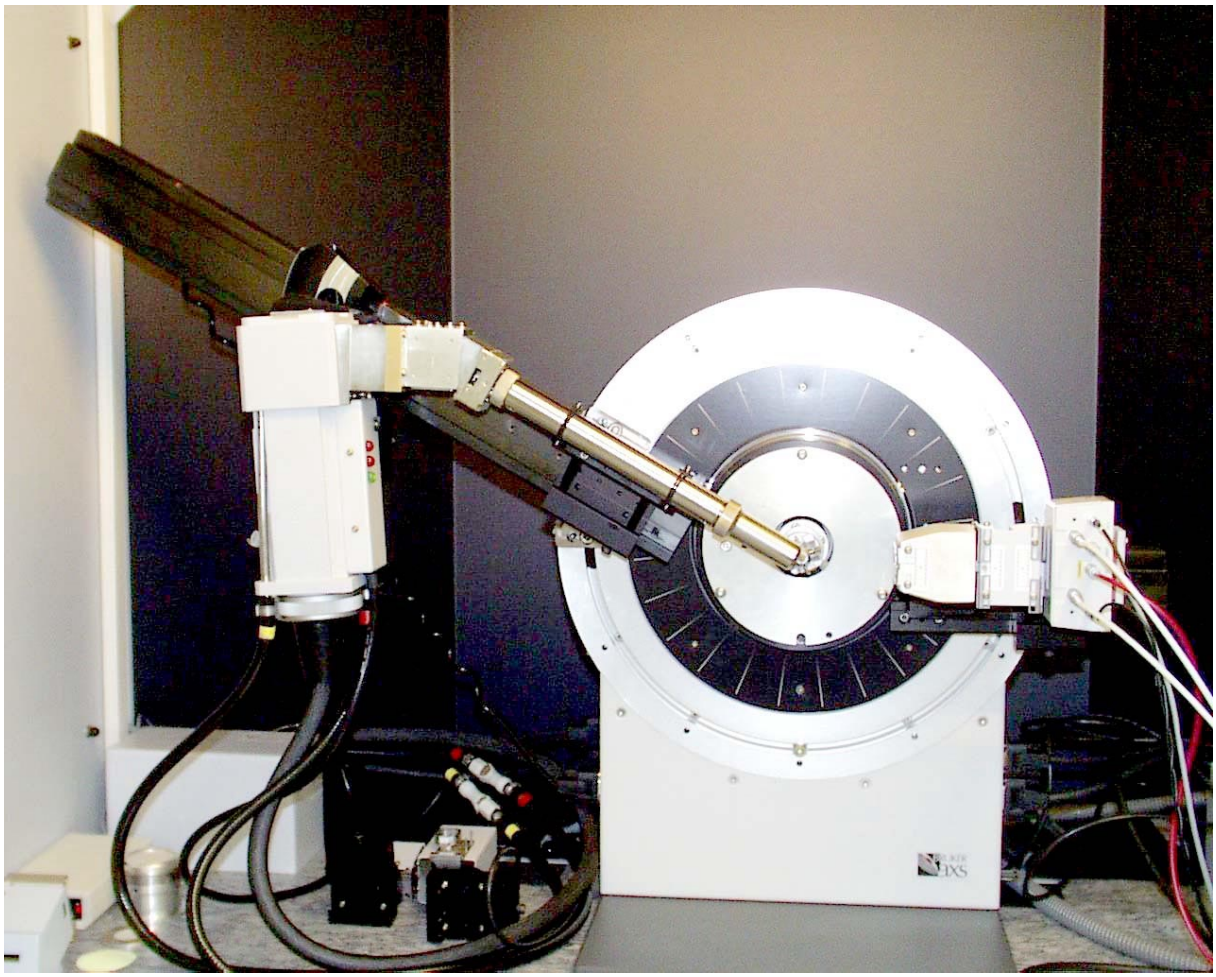
1) Fixing screw for aperture holder and detector at the secondary track

Fig. 2-7: Transmission setup



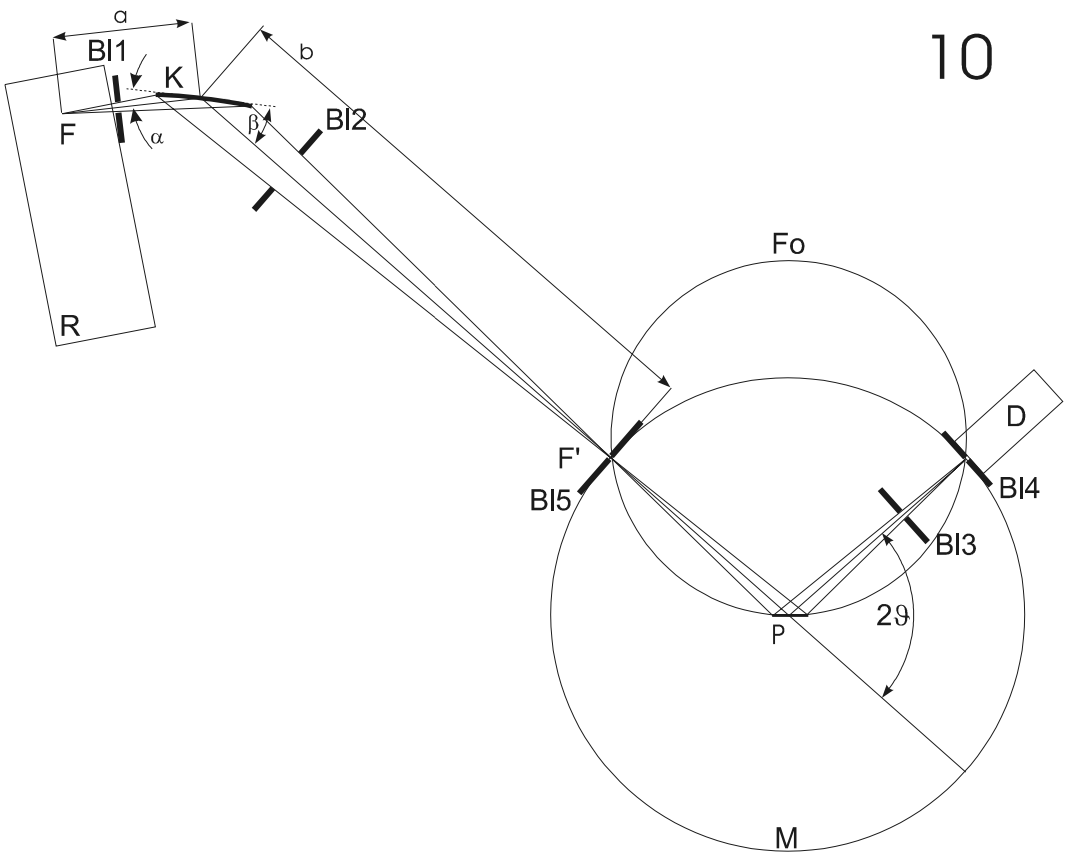
Bl1) Entrance slit
Bl2) Exit slit
Bl3) Antiscatter slit
Bl4) Detector slit
a) Primary slit
b) Secondary focal length
D) Detector
F) Tube focus
F') Focus
P) Sample
R) X-ray tube
K) Monochromator crystal
M) Measuring circle
α) Entrance angle
β) Exit angle
2ϑ) Diffraction angle

Fig. 2-8: Beam path for capillary measurements



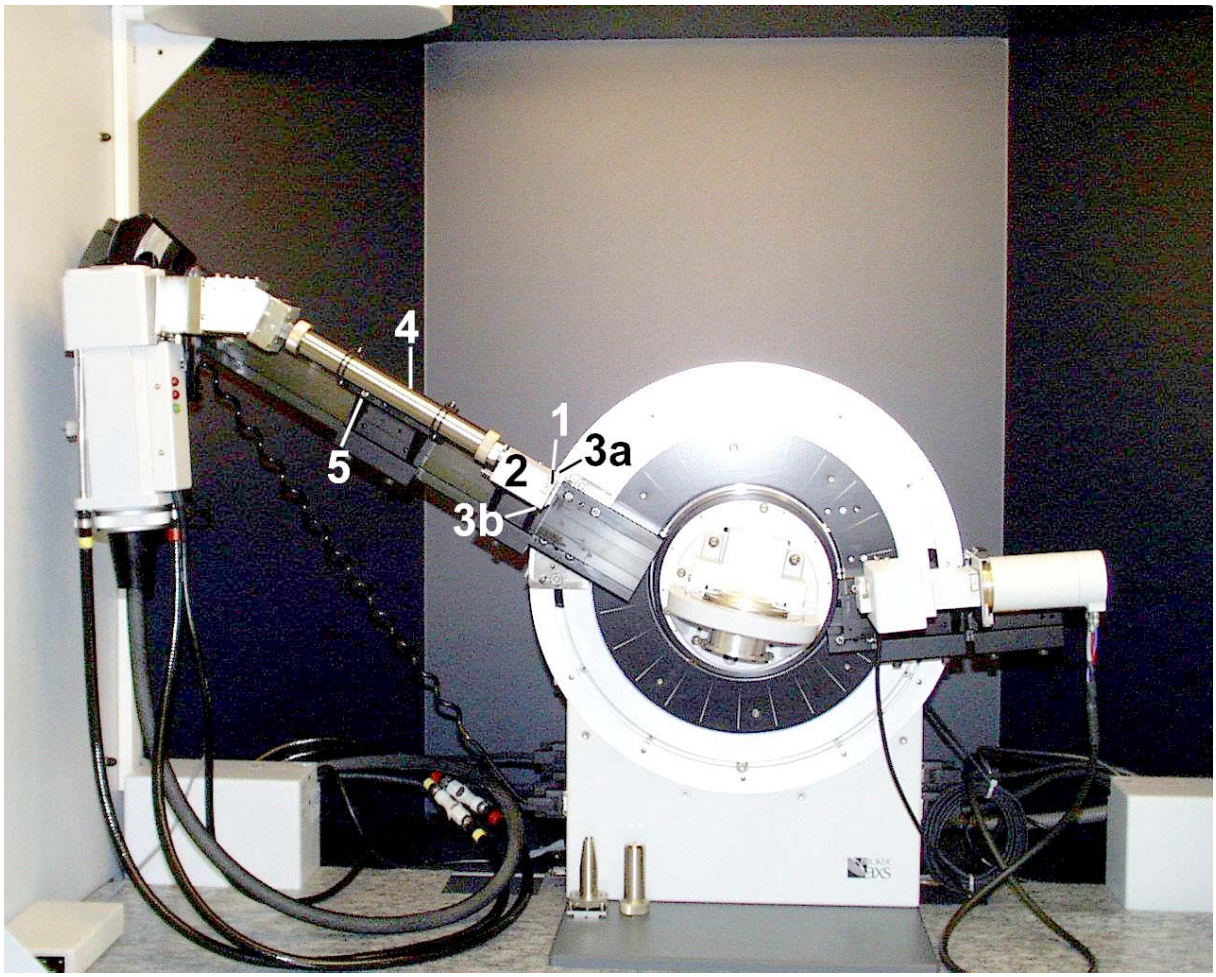
1) Capillary setup with antiscatter tube

Fig. 2-9: Setup for capillary measurements



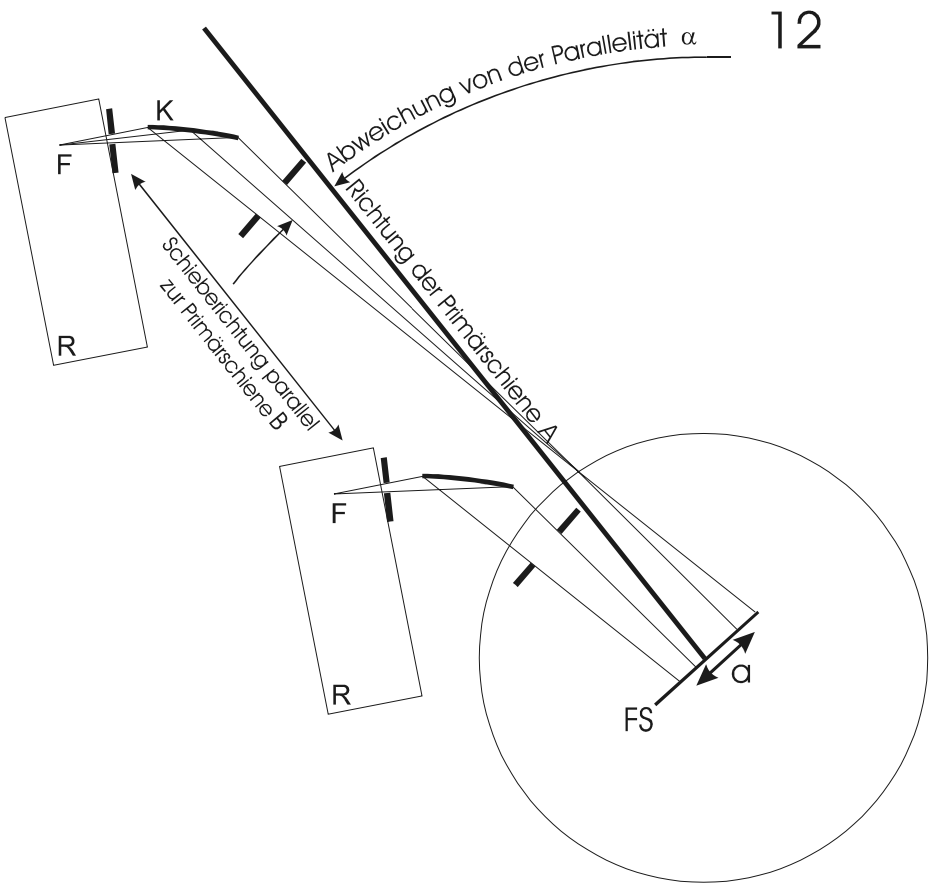
Bl1) Extrance slit
Bl2) Exit slit
Bl5) Primary focal slit
Bl3) Antiscatter slit
Bl4) Detector slit
a) Primary focal length
b) Secondary focal length
D) Detector
F) X-ray focus
F') Focus
P) Sample
R) X-ray tube
K) Monochromator crystal
M) Measuring circle
α) Entrance angle
β) Exit angle
2ϑ) Diffraction angle

Fig. 2-10: Beam path for reflection measurements



1) Focal aperture
2) Slit holder
3) Screws for adjustment of the primary focal aperture
4) Antiscatter tube
5) Screw for adjustment of the antiscatter tube

Fig. 2-11: Setup for reflection measurements



A) Direction of primary track
B) Movement of direction parallel to the primary track
F) Tube focus
K) Monochromator crystal
R) X-ray tube
a) Distance resulting from misalignment
α) Divergency angle (beam and track are not parallel)
FS) Fluorescent screen

Fig. 2-12: Alignment of X-ray source

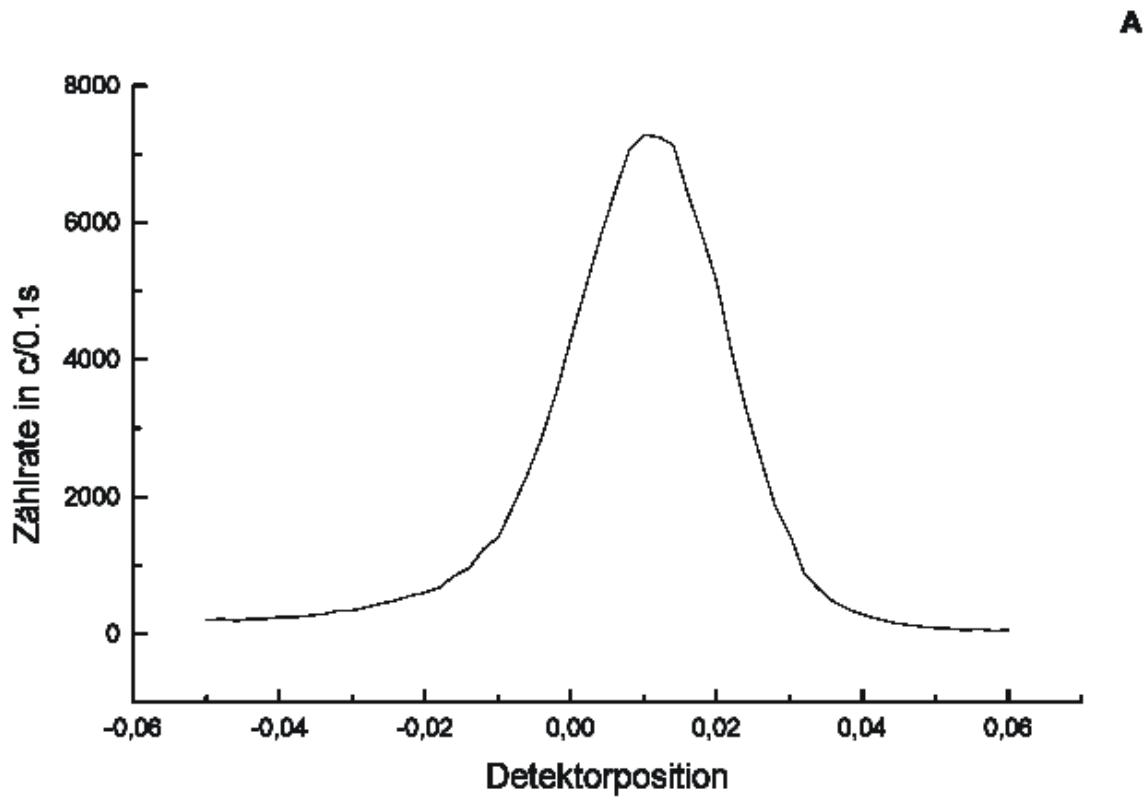


Fig. 2-13: Measurement of the profile of a monochromator reflection in the focus at optimal setting, 50 kV/40 mA, 2 x 0.1 mm Cu absorber, 0.05 mm detector slit

Focusing Primary Monochromator (Huber)

The focusing primary monochromator of Johansson Typ generates $K\alpha_1$-radiation with high intensity and makes it possible to do X-ray diffraction with high resolution. These advantageous properties enable measurements from samples with low scattering ability or with very small amounts of samples. The powder samples can be fixed between two special foils or in thin glass capillaries. Measurements can be also performed with transmission sample changers as well as with capillary sample changers.

Primary Monochromator in Transmission Setup

Geometrical setup

The X-ray tube is fixed by mount 11 (fig. 2-15) with several degrees of freedom on the primary track 16 so that the distance from tube and monochromator to sample P (fig. 2-16) can be changed along the track XP. The Primary monochromator M is directly fixed at tube R. Fig. 2-14 illustrates the beam path and fig. 2-16 pos. 3 the transmission position.

Technical data

Radiation	$Cu_{K\alpha1}$	$Co_{K\alpha1}$
Crystal	focusing Ge-crystal from Johansson type	focusing SiO_2-crystal, Johansson type
Wavelength	1.540619 Å	1.788965 Å
a	approx. 120 mm	approx. 120 mm
b	approx. 365 mm	approx. 355 mm
α	6.639°	7.697°
β	20.643°	23.341°

Setup

X-long primary track

The X-long primary track XP (fig. 2-16) enables the use of the goniometer not only in reflection (pos. 1) and transmission (pos. 3) but also in capillary geometry (pos. 2) by simply sliding the tube monochromator unit R+M along the track. For each setup only one monochromator M with fix secondary focal length is necessary. For the reflection technique (pos. 1) the secondary focus has to be set to the primary part of the measurement circle. For the transmission setup (pos. 3) the focus is on the secondary part of the measurement circle and for the capillary technique (pos. 2) the focus is located at the sample position P.

Mounting the monochromator

Mount the monochromator and adapter unit 1 (fig. 2-15) with the screws to the tube housing. First mount the adapter unit 1 with 4 screws (M4) to the tube housing then the dove tail part of the monochromator housing with entrance aperture slit (4 x M5). Then set the monochromator housing from the top of the tube onto the dove tail and move it down with screw 5.

Prealignment of the monochromator

Turn screw 5 (fig. 2-15) of the monochromator until $\gamma = 5°$; d1 and d2 mark the position of the tube focus 15. Pin 2 marks the mid point of the crystal. Turn screws 6/6' and 7/7' so that the crystal surface is parallel to the line focus of the tube and the long crystal edges are perpendicular to the line focus. Open the slides 9 completely. Set the distance from tube focus 15 and mid of crystal (pin 2) with screw 4. This distance has to be set to the given A value (see sticker on monochromator housing). Move entrance slit slide 13 to position open. Remove the antiscattering tube 10 for the adjustment procedure. Using screw 3 set d3 and d4 so that d3 = 8.3 mm and d4 = 11.1 mm (valid for Cu radiation and Ge crystal and Co radiation and SiO_2 crystal). In this position the crystal is approx. in diffraction position.

Preadjustment for transmission setup

Set the distances tube focus to monochromator (pin 2) and monochromator (pin 2) to detector slit to the values given on the sticker on the monochromator housing. The tube with monochromator can be moved along the track after loosening the screws 4 (fig. 2-17) and slightly lifting the tube at the lower end (HV cabel).

Adjustment under radiation

The licence issued for a fully protected device is rendered void when a configuration with primary monochromator is carried out. The safety functions can be bridged by means of a key switch when adjustment work is being carried out on an open radiation path. On no account may the safety functions be bridged by any other means than the key switch. The bridging key for the safety functions should only be issued to the person responsible for radiation protection and should be kept by him or another person in a safe place so to prevent any improper use. To adjust srews 3 and 4 (fig. 2-15) a long adjustment tool must be used. After prealignment of the monochromator, adjustment under radiation is not necessary.

⚠ **Warning:**
Work carried out on an unsecured diffractometer is dangerous! Adjustment work under radiation may not be executed by any other persons than those professionally exposed to rays and who are kept under medical observation and undergo dosage measurement. During adjustment works, the parameters of the tube have to be at the lowest possible values (voltage and current). Long adjustment tools have to be used. Personnel should never put their hands into the primary beam! After the preadjustment, a final alignment under radiation is not necessary. The tube can be set to higher values when the adjustment is performed with activated safety functions.

Rough alignment

Bring a fluorescent screen into the expected beam path. Move the detector to 90°. Set tube parameters to 40 kV and 40 mA or 50 mA while the safety functions are active. Darken the room and give your eyes time to see even low intensities on the screen. If the security functions are active (use 40 kV / 50 mA), the beam can be seen even in normal daylight (no direct sunlight). Open the entrance aperture slide to 100%.

Diffraction angle of the monochromator

After setting d3 and d4 with screw 3 (fig. 2-15) the Bragg angle is approx. in the right position. Move the sample holder to 90° and bring a fluorescent screen into the expected beam path. The exact diffraction angle can be aligned by small changes to screw 3. Do not set the monochromator to the low intensity Kβ-peak. Since the fluorescent screen is not in focus, you will see a broad reflection with more or less intensity (Kα is broader than Kβ). After this rough alignment, the security functions have to be activated again. The fluorescent screen is not necessary for an optimization of the reflection of maximum intensity and Kα1/Kα2 separation. The fine adjustment can be performed with the detector more precisely and at maximum security.

Use the degree of freedom 1 (fig. 2-17) to change the relative angle from tube to track so that the beam hits the goniometer center (fluorescent screen).

This setup can be performed by succesively changing the tube position with the following screen control (closed radiation protection door). To see the monochromator reflection, increase the tube power (e.g. 40 kV / 40 mA or 40 kV / 50 mA)!

Adjustment of the primary focal length

Since the final monochromator setup is very sensitive to power changes of the tube (resolution, intensity), the complete fine adjustment has to be made with these values of current and voltage which you will use later for measurements. We recommend 40 kV / 40 mA or 40 kV / 50 mA. Other values are also possible but you may loose intensity. The next step is to use proper detector apertures and absorber: 2 x 0.1 mm Cu absorber, Ni filter and 0.1 mm

detector slit. Measure the monochromator reflection with the program ADJUST with a detector scan (remove the fluorescent screen). If you see a maximum in your plot, move the detector to this maximum. If you dont't see any maximum move the detector to the middle of the profile.

Now change the 0.1 mm slit to a 6 mm one to adjust the monochromator to maximum integrated intensity. Use the ratemeter of ADJUST for the procedure. First set the monochromator with screw 3 (fig. 2-15) to maximum integrated intensity (open shutter – close door – measure – open door – turn screw – close door – control intensity – open door – turn screw – etc.). The monochromator is very sensitive to screw 3 because the screws have a relatively big slope. Make a note of the maximum integrated intensity (pos. B).

In the next step change the distance between pin 2 and tube focus 15 to determine the primary focal length of the monochromator. Turn screw 4 clockwise two times (pos. A) and maximize the integrated intensity with screw 3. Do the same procedure counterclockwise two times (pos. C) away from pos. B. Compare the values of the integrated intensity in positions A, B and C. If the max. intensity is at B you can do the same procedure for +/- 1 turn. If the optimal position is at A or C, proof whether the distance has to be increased or decreased further.

In most cases the setup can be done with a tolerance of +/-1 mm. If maximum resolution and intensity is important, we recommend doing this time consuming procedure. Check with a detector scan between several alignment steps whether the middle of the reflection is still within the detector aperture. After the determination and setting of the primary focal length, push in the entrance slit 13 and adjust with screw 3 (fig. 2-15) for maximum integrated intensity.

Adjustment of the secondary focal length

Remove one Cu-Absorber and exchange the 0.1 mm detector slit with a 0.05 mm one. Measure the monochromator reflection with a detector scan. Determine the peak intensity I_p (maximum of reflection profile) and FWHM in this position (pos. B). Since it is easier to change the detector's position than the tube's position the determination of the secondary focal length has to be done by varying the detector distance on the secondary track. The position of the detector and the slit holder have to be changed simultaneously. Loosen the screws of the holder (fig. 2-18, 4) and shift both units. We recommend controlling the position with a caliper gauge referring to a reference point.

In the next step reduce the distance of the aperture detector unit to the tube by 2mm (pos. A). Measure the reflection profile with a detector scan in this position and determine I_p and FWHM. The same procedure has to be done in pos. C (2 mm far away from pos. B). Compare the measured values for I_p and FWHM in pos. A, B and C. If you are near the focal position the best value for I_p is max. and for FWHM is min.. If max. I_p and min. FWHM is

determined, the same procedure with a variation of +/- 1 mm has to be done. It is recommended to determine the focus with an accuracy of +/- 1 mm if resolution and intensity are considered very important. The FWHM can be further reduced by aligning the crystal with the screws 6/6' and 7/7' (fig. 2-15). To do this follow these steps:

1. Tilt crystal with screws 6/6' (1/4 turn) by a small angle
2. Use 6 mm detector slit and 0.2 mm Cu absorber
3. Set max. integrated intensity with screw 3
4. Scan detector with 0.05 mm and 0.1 mm Cu absorber
5. Determine I_p and FWHM
6. Best setting is with max. I_p and min. FWHM

Do the same procedure with screws 7/7'. The focus of a well aligned Johansson monochromator has to look like the plot of fig. 2-25. Used apertures and absorbers are: 0.05 mm detector aperture, 0.2 mm Cu absorber, 40 kV, 50 mA.

Setting the distances for transmission geometry

Set slit holder and detector to the measuring circle (435 mm diameter) for measurements in transmission geometry. The first step is to determine the distance $\tilde{B}$ between the monochromator (pin 2 in fig. 2-15) and the detector slit (note the position of the slit within the slit unit). For the measuring circle of 435 mm the distance of the detector slit to the goniometer center has to be 217,5 mm. Move the detector so that it is near the slit. Furthermore move the tube and monochromator unit so that the distance between pin 2 and goniometer center is the difference of $\tilde{B}$ - 217.5 mm.

Alignment of the primary beam to the goniometer center

Use the glass slit to set the convergent primary beam to the goniometer center. The first step is to determine the ZI value for the 2ϑ circle with a detector scan of the beam focus. Then mount the glass slit into the sample holder and determine the setting with max. intensity ('rocking curve' in ADJUST). For this procedure use the 6 mm detector slit to measure the integrated intensity. Proof whether the intensity value can be improved by inclining the tube with respect to the primary track (screw 1 in fig. 2-17).

Maximize the intensity value by alternating the setting of screw 1 and measuring (rocking curve) the profile. If you found the optimum position determine the ZI values for 'rocking curve' and 'detector scan'. Mount the sample holder for transmission geometry on the goniometer.

Primary monochromator in capillary geometry

Geometrical setup

Move the tube with monochromator by 217,5 mm from the goniometer center along the primary track in order to change from transmission to capillary geometry (distance from pin 2 in fig. 2-15 to goniometer center has to be the secondary focal length $\tilde{B}$). In fig. 2-19 you see the beam path in capillary geometry (fig. 2-16, pos. 2). Since the focus of the crystal is in the position of the sample, a capillary sample holder has to be mounted.

Alignment

All alignments of the monochromator have to be done as described in the subchapter of transmission geometry. After that, enlarge the distance of the monochromator to the sample. To reduce the alignment work between the different geometries, set the beam parallel to the primary track and to the center of the goniometer. This alignment is described later in the subchapter of reflection geometry. After moving the tube monochromator unit, proof with the glass slit whether the beam hits the goniometer center. Small corrections are always necessary by translation or rotation of the tube by a small amount. The alignment can be done with screw 1 (fig. 2-17). In order to reduce the air scattering, mount the antiscatter tube for capillary measurements.

Primary monochromator in reflection geometry

Geometrical setup

Move the tube with monochromator from transmission to reflection geometry along the primary track by 435 mm away from the sample (distance of pin 2 in fig. 2-15 to the goniometer center has to be $\tilde{B}$ + 217.5 mm). You see in fig. 2-20 the beam path for a reflection setup (fig. 2-16, pos. 1 and fig. 2-23). The position of the focus is on the primary side of the measuring circle. Mount a sample holder for reflection geometry.

Alignment

All alignment work of the monochromator has to be done as described in the subchapter of transmission geometry. After that enlarge the distance of the monochromator as described above. To minimize the alignment work, when you change between different geometries the primary beam has to be parallel to the primary track and has to hit the goniometer center. This alignment procedure is described in this chapter. After moving the monochromator tube unit along the primary track, proof with the glass slit whether the beam hits the goniometer center. Small corrections are always necessary. To do this, change the relative angle from tube to track with screw 1 (fig. 2-17). Maximize the integrated intensity through the glass slit.

Alignment of the focus aperture

You see in fig. 2-23 the position of the focus slit. First step is to remove the cover, soller and aperture from the holder 2. Proof with a fluorescent screen whether the focus of the beam is in the middle of the aperture holder, otherwise the aperture holder position has to be aligned with screws 2/2' (fig. 2-21).

Afterwards adjust the aperture position with decreasing slit width (2 mm, 1 mm, 0.6 mm, 0.2 mm, 0.1 mm) for maximum intensity with screws 2/2'. About 50 % to 80 % intensity pass through the 0.1 mm slit and 100% through the 0.2 mm slit. In general a 0.2 mm aperture is recommended.

Alignment of the antiscattering tube

After the alignment procedure, the anti scattering tube 1 (fig. 2-22 and 3 in fig. 2-21) can be mounted. The antiscattering tube reduces the air scattering and the background of the detector with an improvement of the signal/background ratio. The position of the antiscattering tube can be changed within small limits with screw 4 (fig. 2-21 and 2-22) so that the beam passes the tube.

Alignment of the beam parallel to the track

Two conditions have to be fulfilled in the ideal case:

a) The monochromator reflection is parallel to the primary track

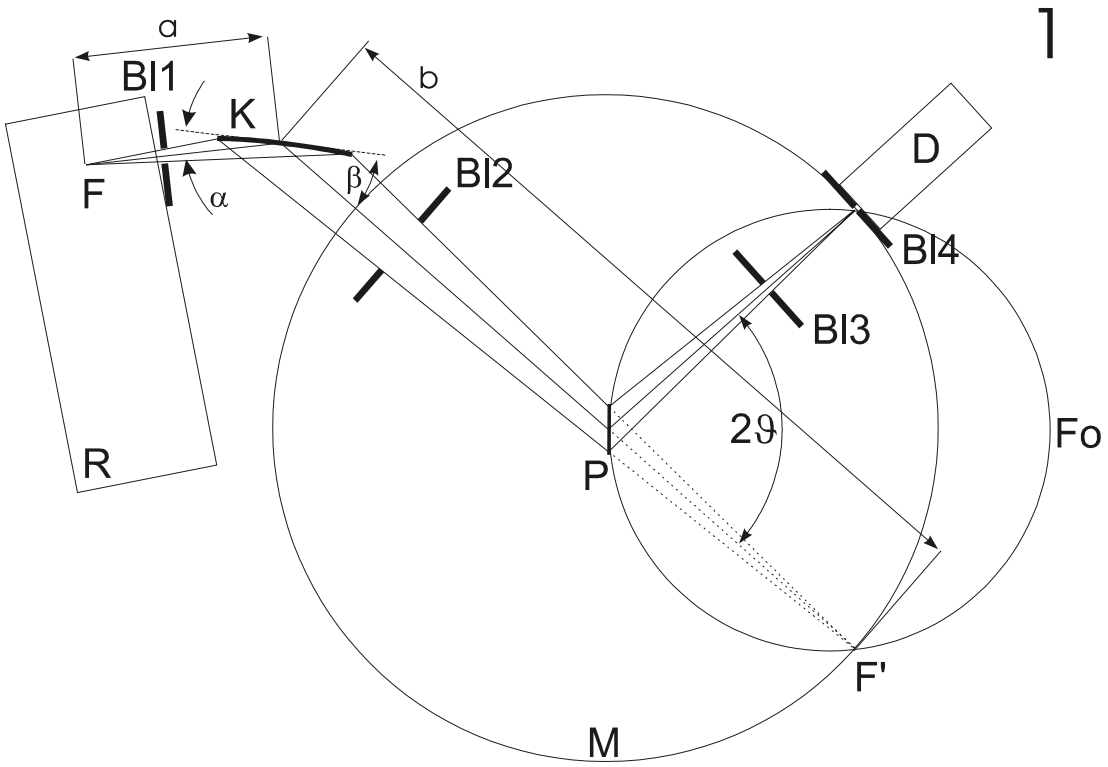
b) The reflection hits the goniometer center

If these conditions are fulfilled, adjustments are not necessary after moving the tube monochromator unit. Production tolerances and changes of forces make it necessary for small adjustments to be made after moving the tube.

In the first step, proof the deviation of the setting from the ideal case. Move the tube near the goniometer center (small distance) to the fluorescent screen in the sample holder. Remove the aperture holder for this adjustment. Mark the middle of the beam position on the screen. After that, move the tube far away from the screen (big distance) along the track and mark again the beam position on the screen. The misalignment can be determined from the distance a (cf. fig. 2-24). Incline the tube so that the beam is not moving on the screen when you move the tube along the track. In general the beam is not in the center of the goniometer. In this case translate the tube monochromator unit with the translation screw of the tube mount. This rough alignment with a fluorescent screen is good enough to make a fine adjustment with the detector and the glass slit.

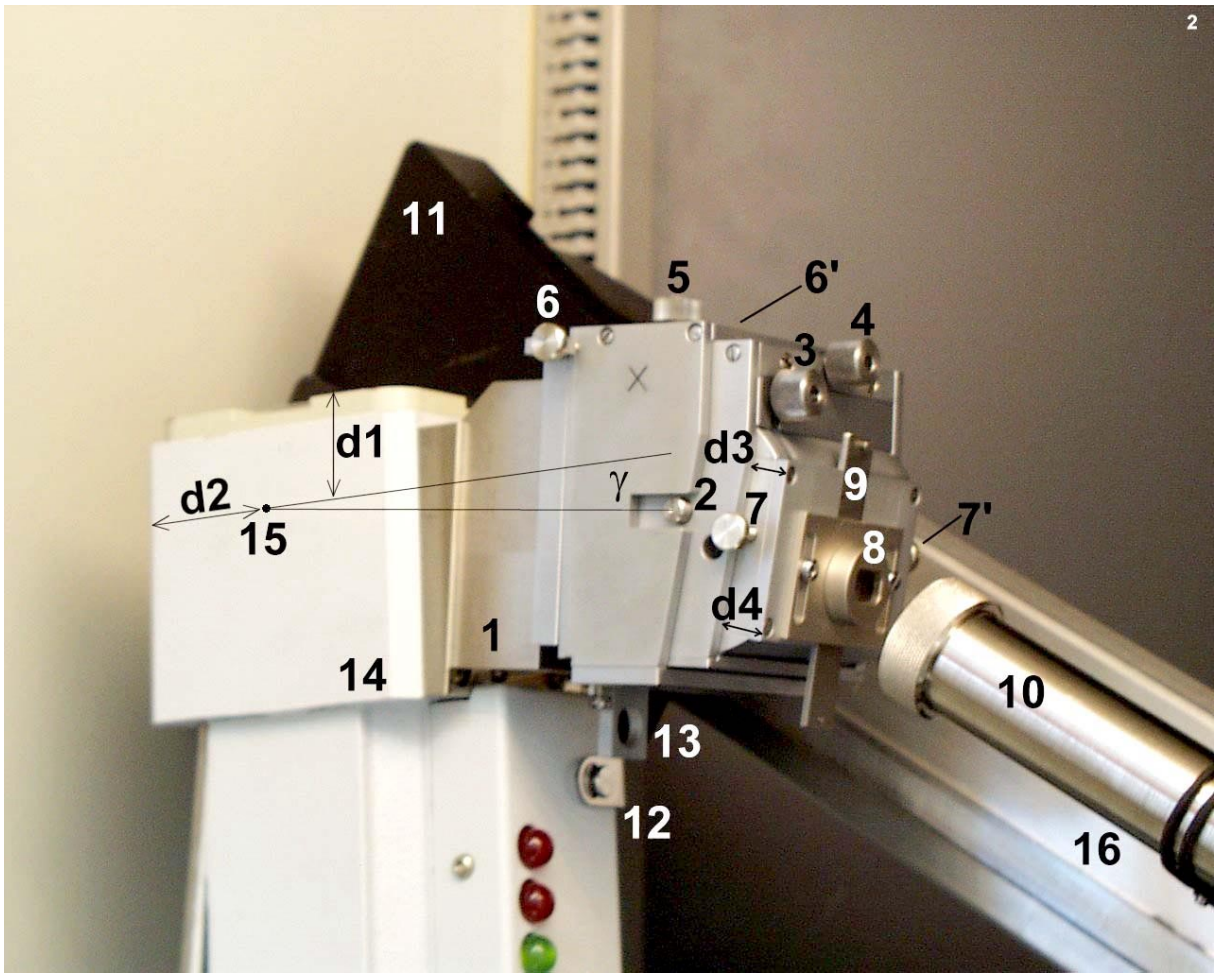
The principle of fine alignment procedure is the same one as described for the rough alignment. The alignment with the glass slit and the detector is much more precise. For a final adjustment, the value for α is about 0.05° - 0.1° or better. Repeat the alignment for the focus aperture for reflection measurements after this alignment. Small deviations after the changes from transmission, reflection and capillary can be corrected by small inclinations or translations of the tube (cf. fig. 2-17).

Figures



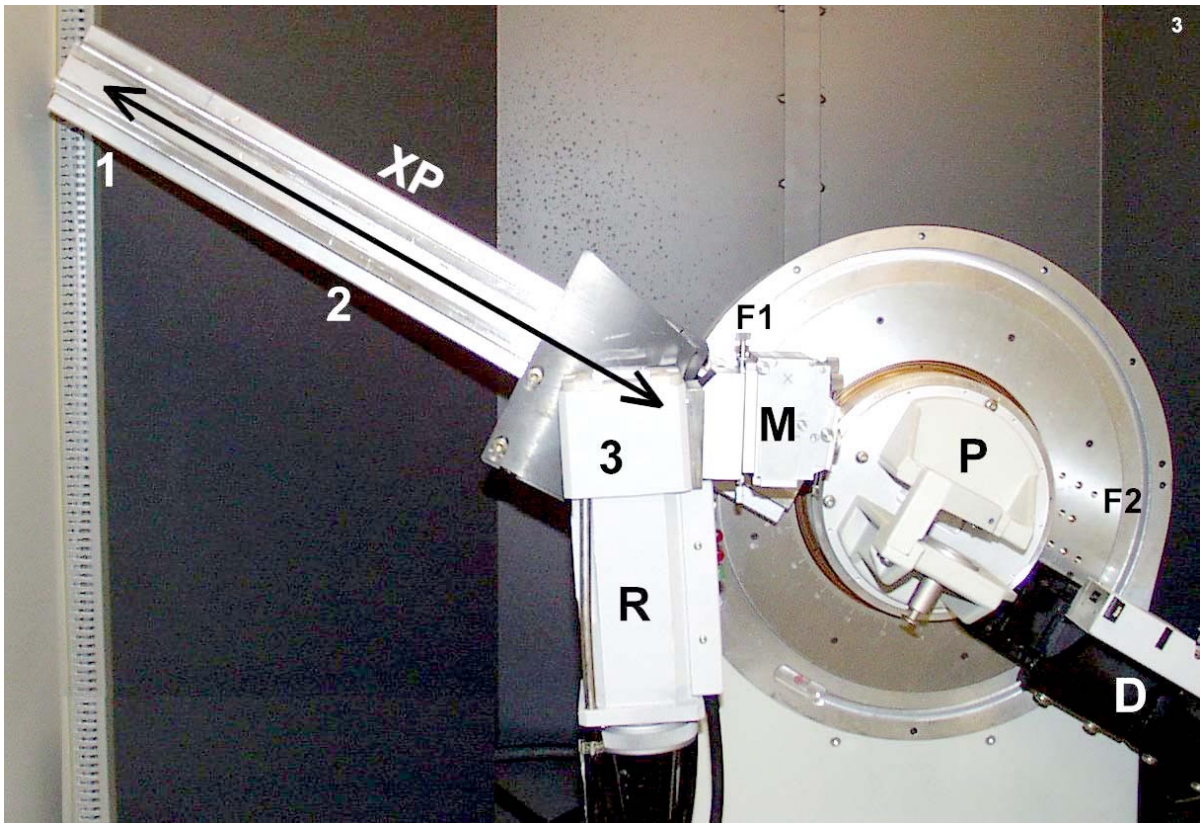
Bl1) Entrance slit
Bl2) Exit slit
Bl3) Antiscattering slit
Bl4) Focus detector slit
a) Primary focal length
c) Secondary focal length
D) Detector
E) Tube focus
F') Focus
P) Sample
R) X-ray tube
K) Monochromator crystal
M) Measuring circle
Fo) Focusing circle
α) Entrance angle
β) Exit angle
2ϑ) Diffraction angle

Fig. 2-14: Beam path for transmission


2
11
5
6'
6
4
3
d1
d3
d2
γ
2
7
9
7'
15
8
d4
1
14
10
13
12
16

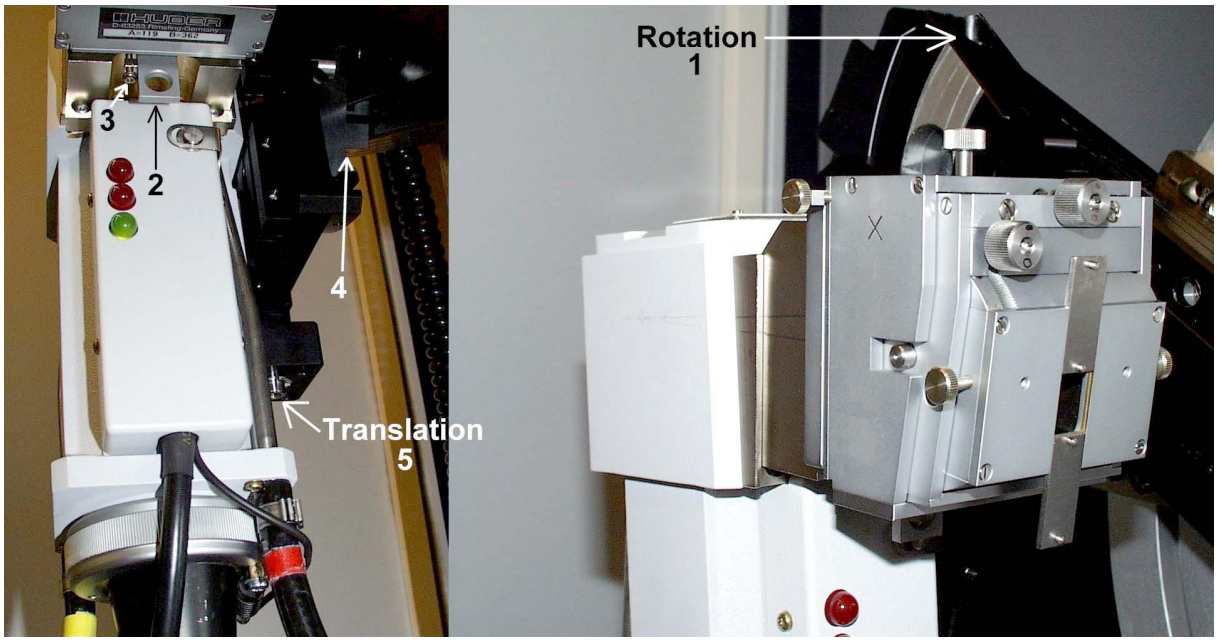
1) Adapter plate for monochromator mounting
2) Mid point pin of the monochromator
3) Alignment screw for the Bragg angle
4) Alignment screw for the primary focal length setting (distance of Pin 2 to tube focus)
5) Alignment screw for the take off angle γ (translation of monochromator parallel to the tube axis)
6) Inclination of the crystal perpendicular to the tube axis
7) Inclination of the crystal in direction of the tube axis
8) Slit
9) Variable exit slit
10) Antiscatter tube
11) Tube mount
12) Interlock
13) Entrance slit holder with alignment screw
14) Tube focus
15) Position of the tube focus
16) Xlong primary track
d1) 27.8 mm
d2) 42.6 mm
d3) 8.3 mm (Ge crystal, Cu radiation): 8.5 mm ($Si0_2$ crystal, Co radiation)
d4) 11.1 mm (Ge crystal, Cu radiation): 10.95 mm ($Si0_2$ crystal, Co radiation)
γ) 5°

Fig. 2-15: Monochromator from Huber with tube



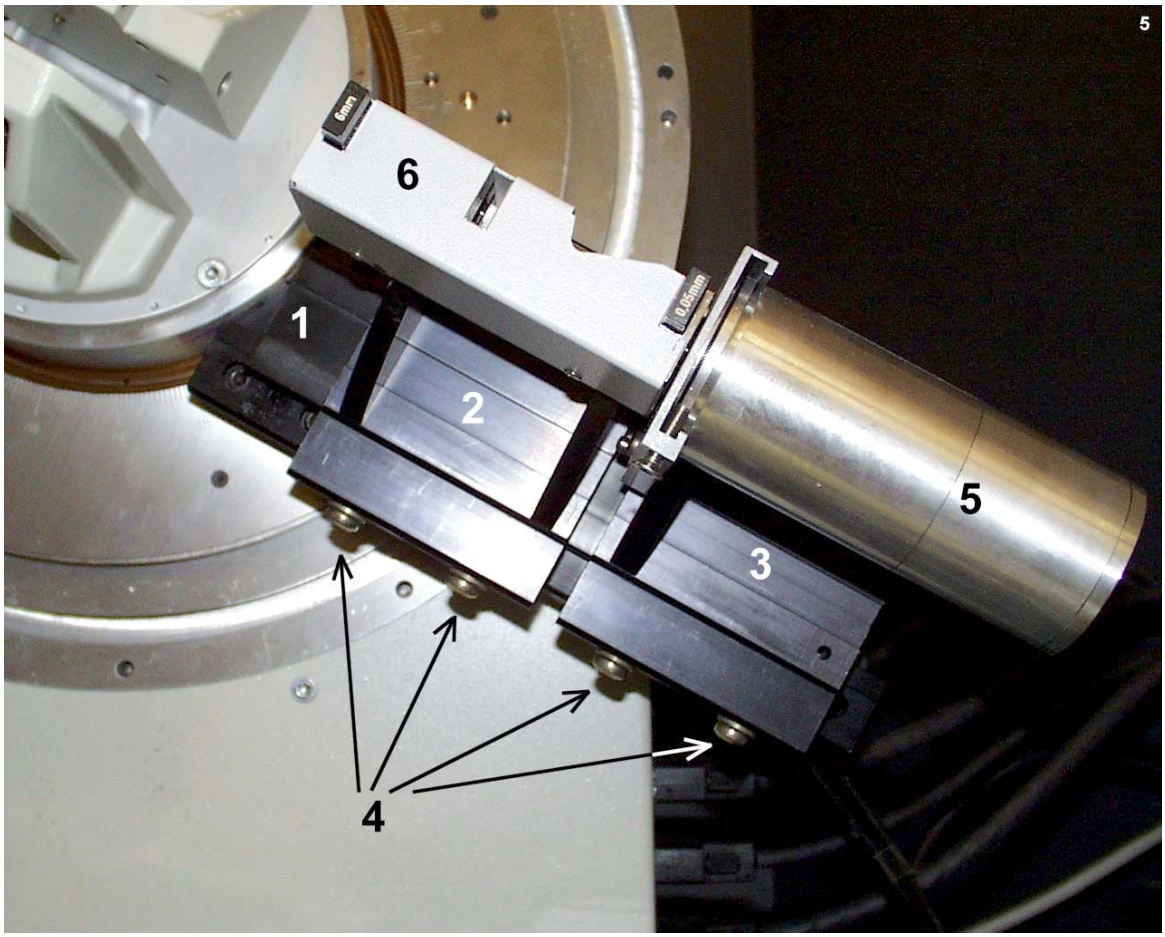
XP) Xlong primary track
M) Primary monochromator
R) X-ray tube
A) Adapter plate
P) Sample holder
D) Detector with slit holder
F1) Primary side of the measuring circle
F2) Secondary side of the measuring circle
1) Position of the tube for reflection geometry
2) Position of the tube for capillary geometry
3) Position of the tube for transmission geometry

Fig. 2-16: Movement of the X-ray source along the track



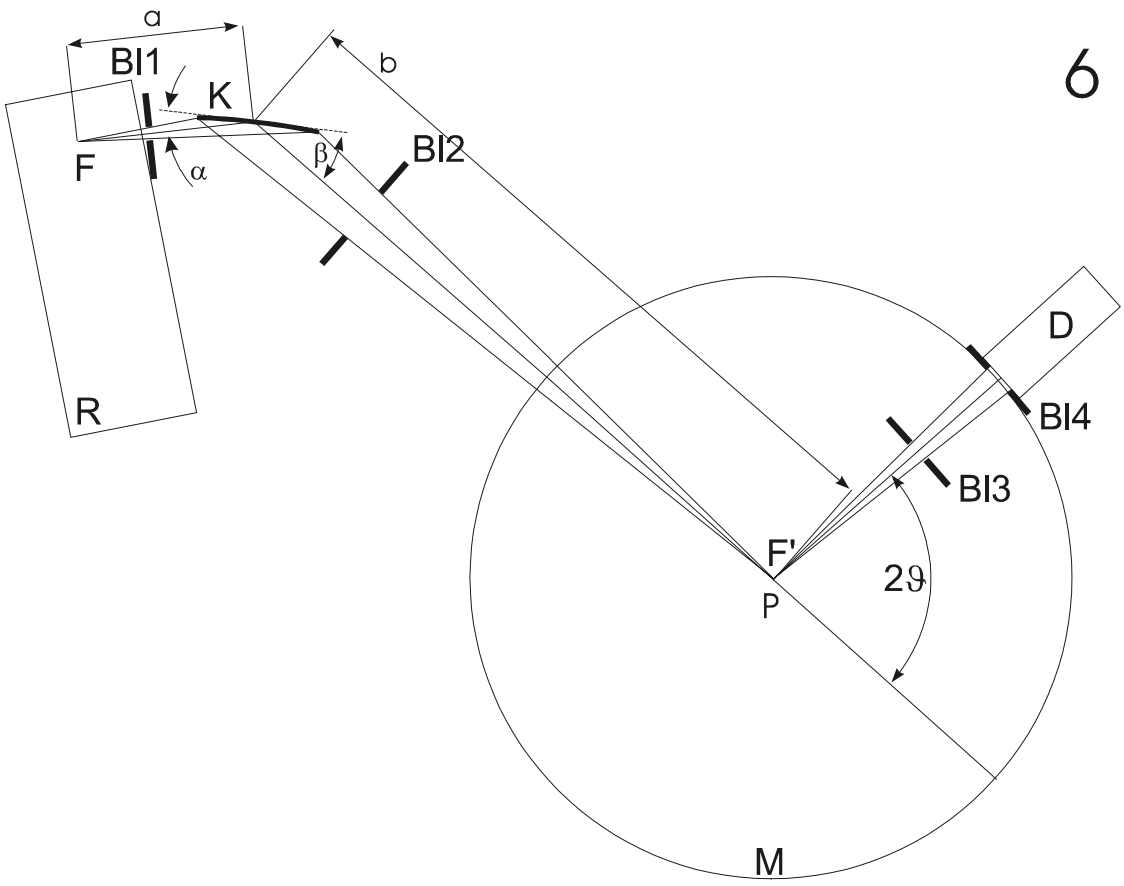
1) Alignment screw to incline the tube
2) Entrance slit
3) Alignment screw for entrance slit
4) Mounting screws for tube mount
5) Alignment screw to translate the tube in direction of the tube axis

Fig. 2-17: Alignment screws for the X-ray tube



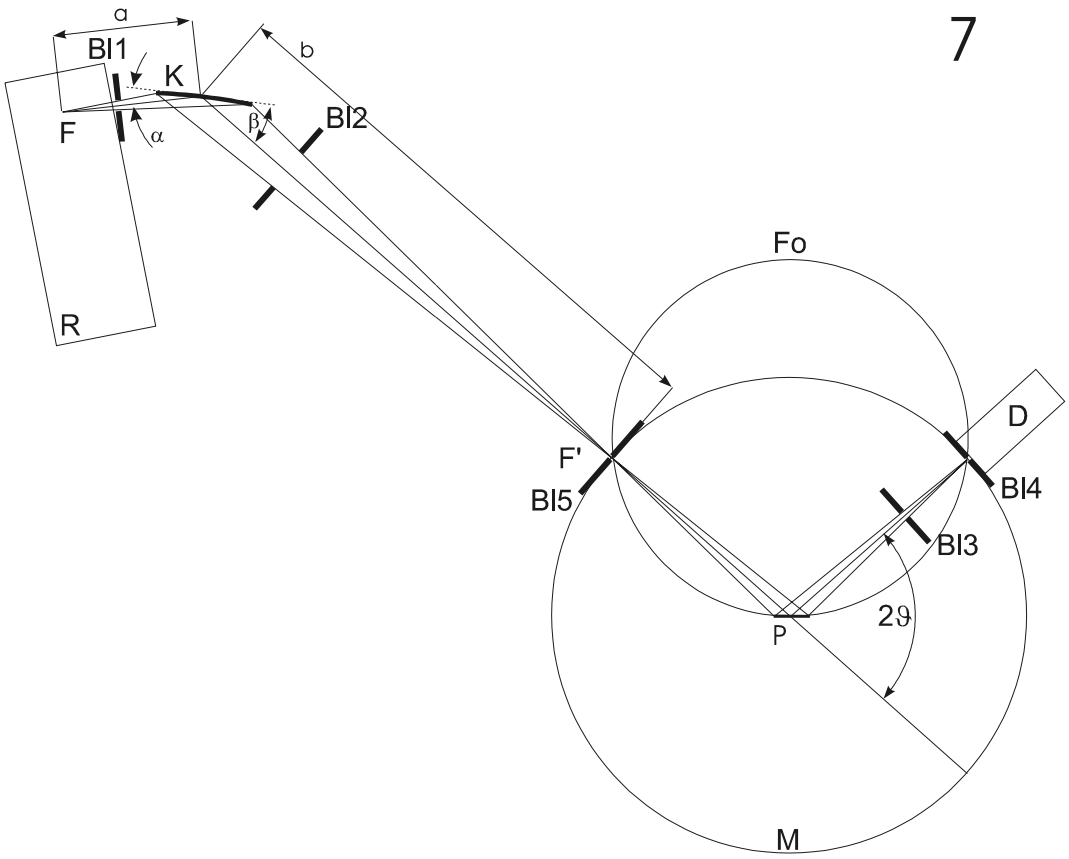
1) Short secondary track for the detector
2) Mount for aperture holder
3) Mount for detector
4) Mounting screws to fix the holder
5) Detector
6) Slit holder

Fig. 2-18: Detector with slit holder



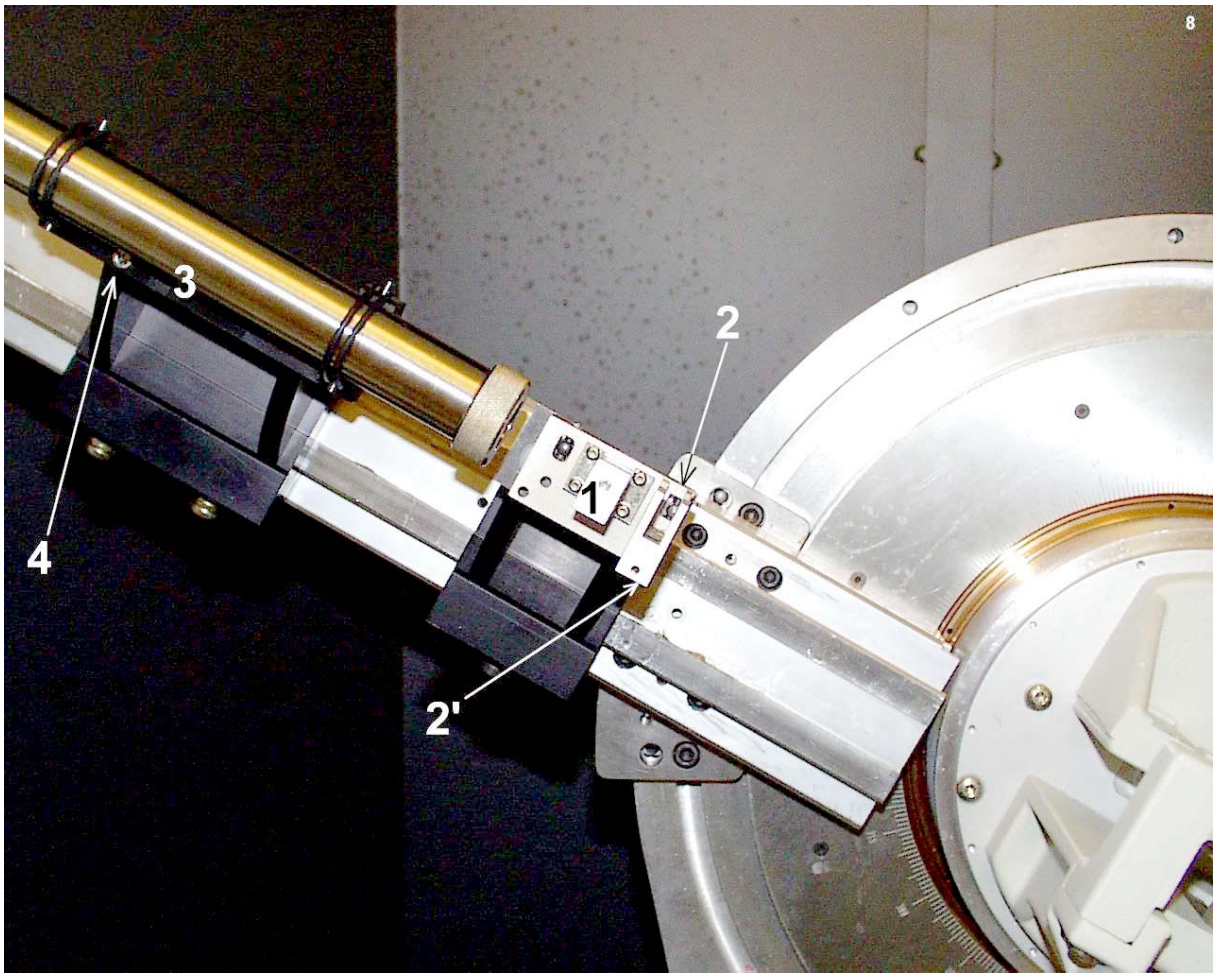
Bl1) Entrance slit
Bl2) Exit slit
Bl3) Antiscattering slit
Bl4) Detector slit
a) Primary focal length
b) Secondary focal length
D) Detector
F) Tube focus
F') Focus
P) Sample
R) X-ray tube
K) Monochromator crystal
M) Measuring circle
α) Entrance angle
β) Exit angle
2ϑ) Diffraction angle

Fig. 2-19: Beam path for capillary geometry



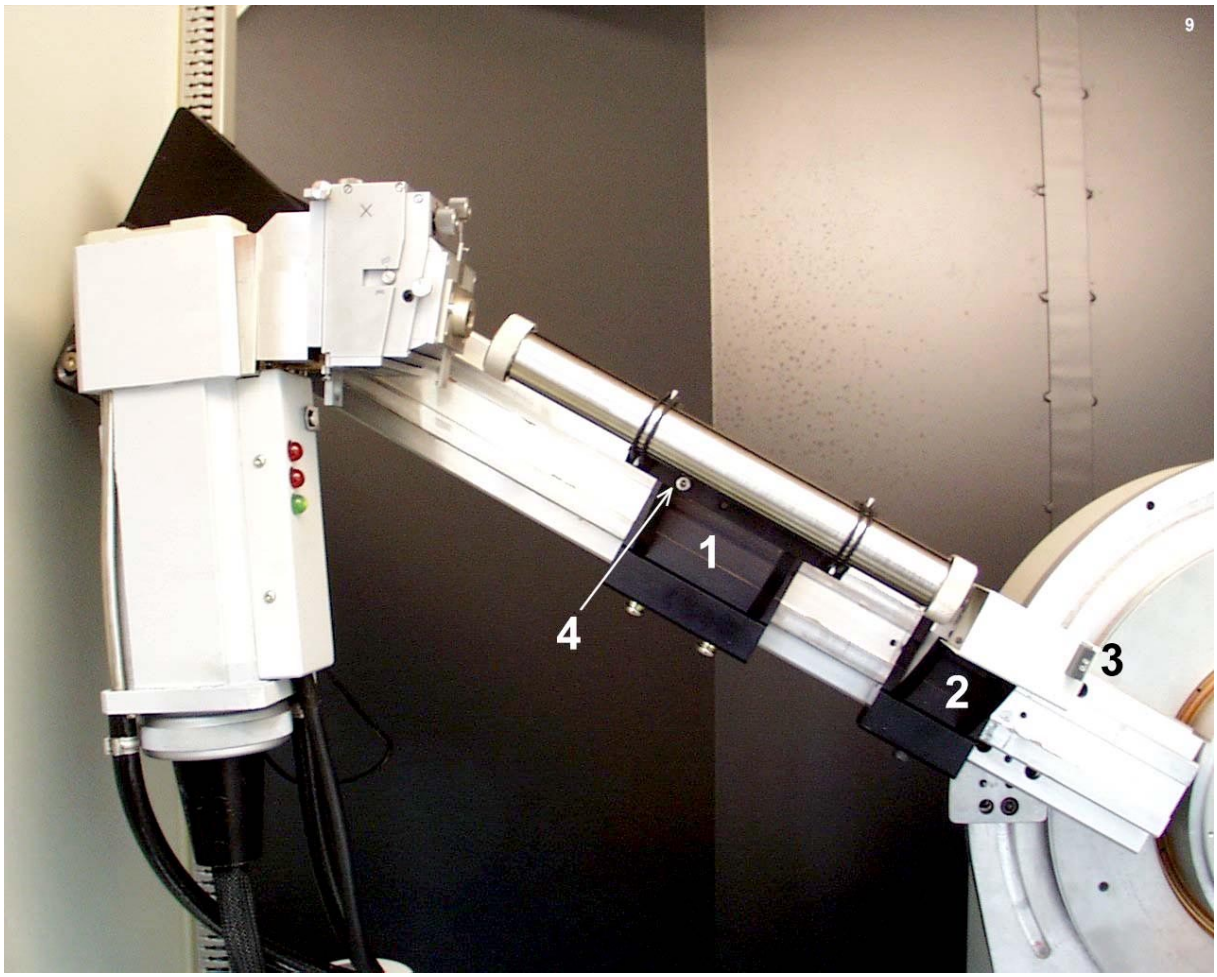
Bl1) Entrance slit
Bl2) Exit slit
Bl5) Primary focus slit
Bl3) Antiscattering slit
Bl4) Detector slit
a) Primary focal length
b) Secondary focal length
D) Detector
F) Tube focus
F') Focus
P) Sample
R) X-ray tube
K) Monochromator crystal
M) Measuring circle
α) Entrance angle
β) Exit angle
2ϑ) Diffraction angle

Fig. 2-20: Beam path for reflection



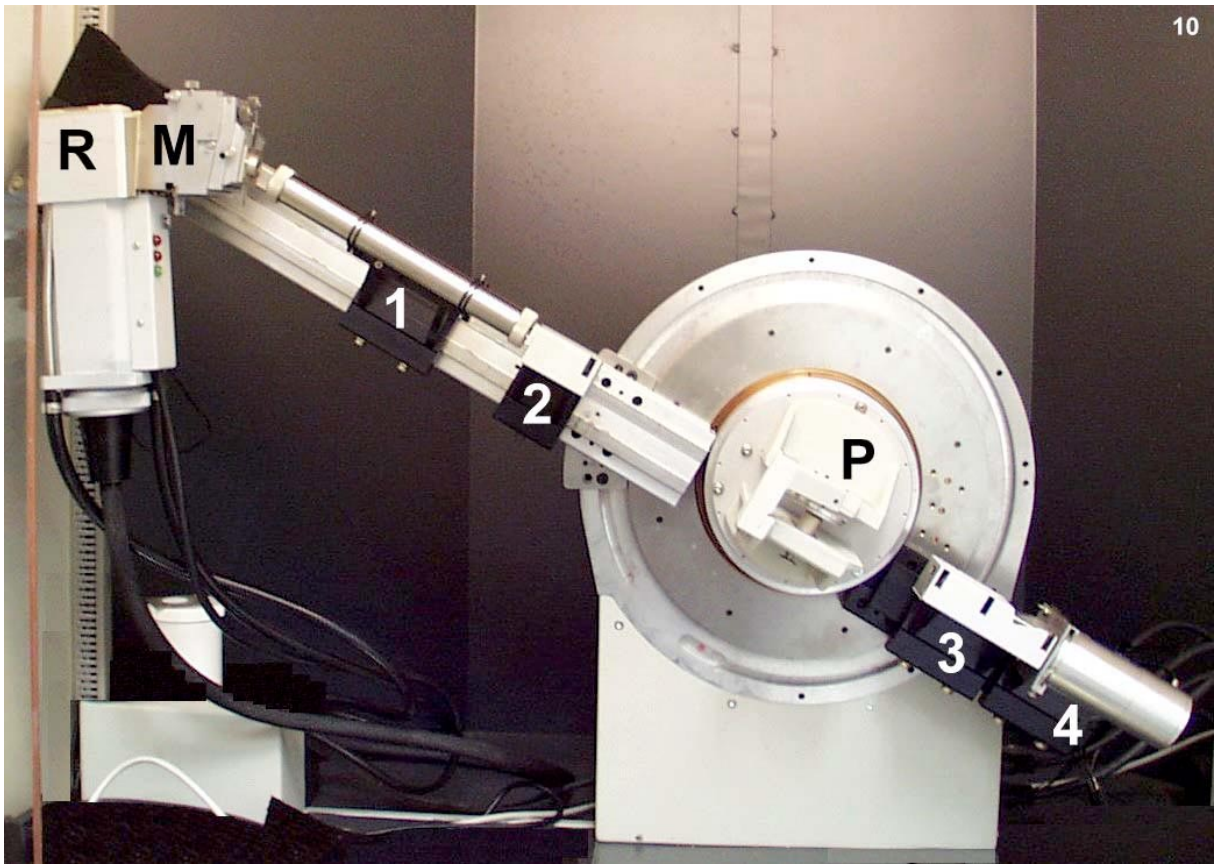
1) Holder for soller
2/2') Alignment screw for focus slit
3) Antiscattering tube with holder
4) Fixing screw for tube alignment

Fig. 2-21: Antiscattering tube and focus slit holder



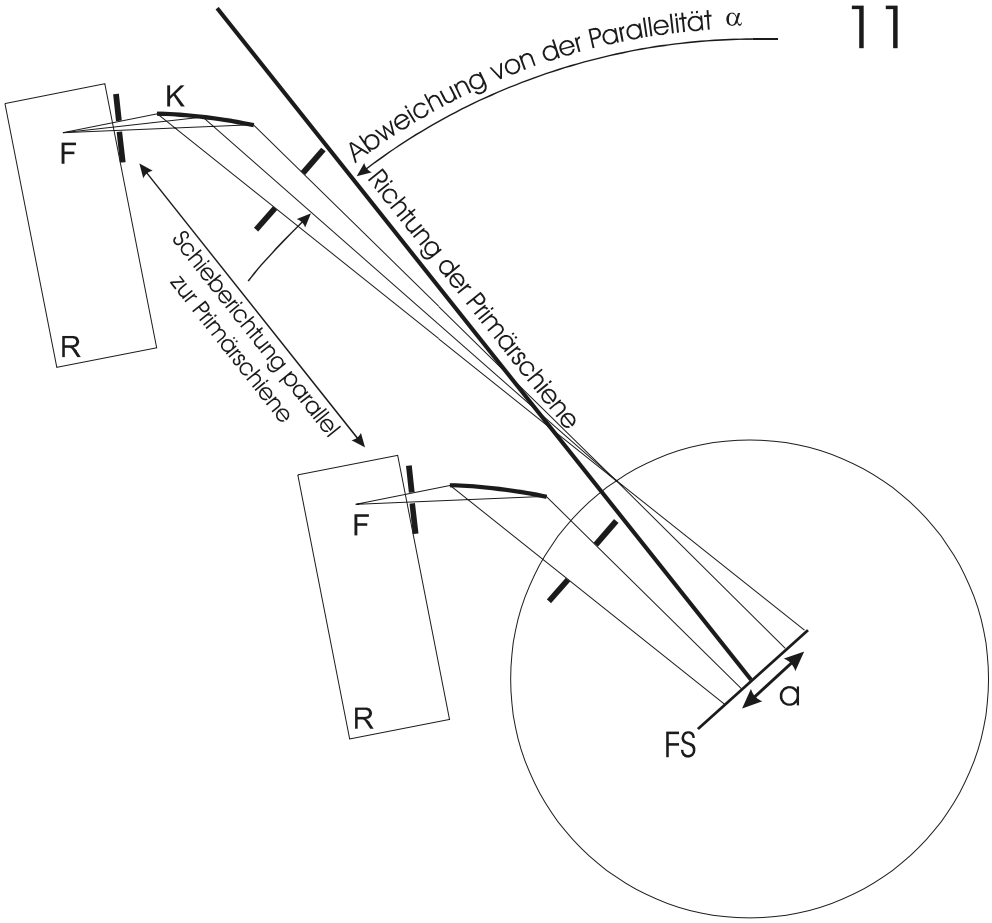
1) Antiscattering tube with holder
2) Holder for focus slit
3) Focus slit
4) Fixing screw for tube alignment

Fig. 2-22: Setup for capilllary measurements



R) X-ray tube
M) Monochromator
1) Antiscattering tube holder
2) Holder for focus slit
P) Sample holder
3) Holder for detector slit
4) Detector

Fig. 2-23: Setup for reflection measurements



F) Focus of tube
K) Monochromator crystal
R) X-ray tube
a) Distance from misalignment
α) Divercence angle (track and beam are not parallel)
FS) Fluorescence screen

Fig. 2-24: Alignment of X-ray source

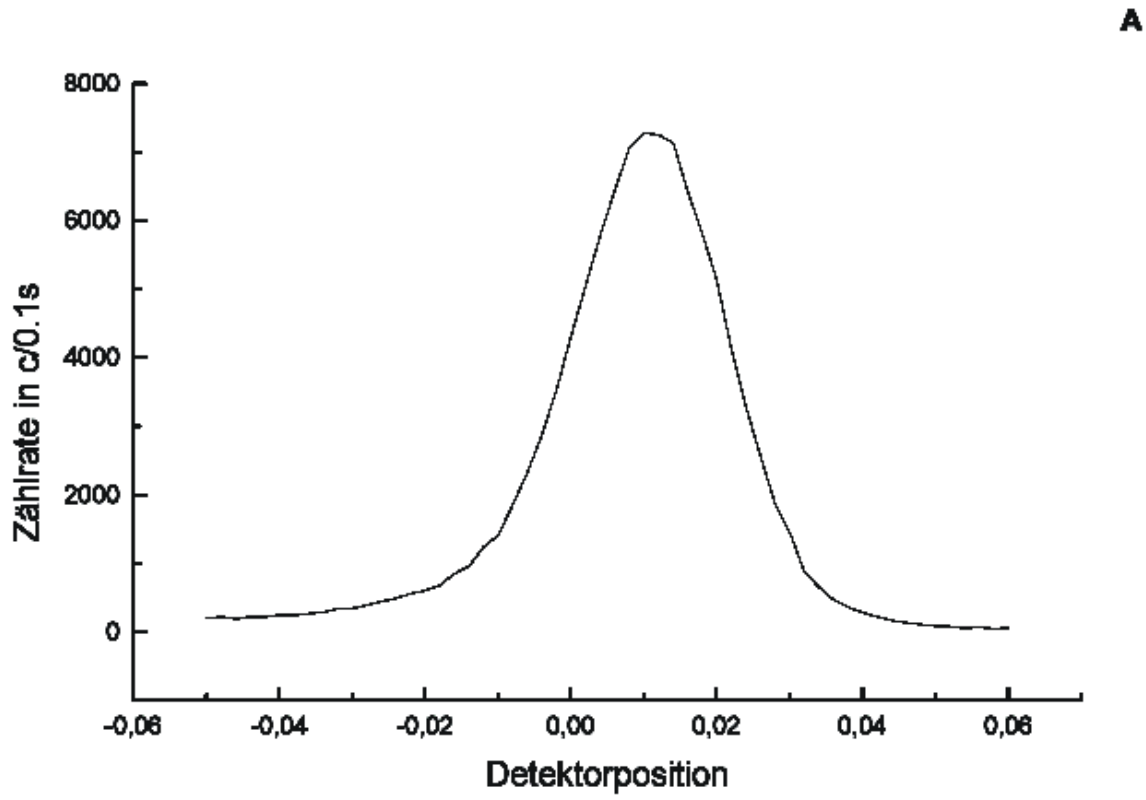


Fig. 2-25: Reflection profile of focus after perfect alignment.
50 kV/40 mA, 2 x 0.1 mm Cu absorber, 0.05 mm detector slit

3 Alignment of Focusing and Parallel Beam PolyCap

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3 Alignment of Focusing and Parallel Beam PolyCap

PolyCap parallel: **order number: C79298-A3244-B119 (D8)**

PolyCap focusing: **order number: C79298-A3244-B144 (D8)**

Introduction

The PolyCap is optimised for use on D8. There are additional mounting parts for D5000 and D5005 available. Alignment is more tedious for these systems and the focus position cannot be optimised for the application. Collision safety is not guaranteed for D5000 and D5005.

For standard set up a 0.4° grazing incidence Soller is recommended. Depending on application the standard slit system or the a single slit (C79298-A3244-B157) mounted on the detector holder might be more efficient.

The alignment is easiest with a 4DOF tube mount. It is recommended to use at least a 3DOF. The additional costs are more than compensated by the easier alignment.

Specification (parallel)

Energy of X-rays (starting at 10keV the guiding of the PolyCap is reduced)	5 ... 10 keV (20 keV)
Size of♣ tube focus (point)	0.3x0.3 ... 0.4x1.2
Input area diameter	5 +/- 0.2 mm
Opening angle (input)	4°
Focal length	70 +/- 2 mm
Length of lens	45 +/- 2 mm
Divergence with 5.0 mm output aperture	< 4 mrad = approx. 0.25°
Useful beam size at output	>4 mm
Transmission	>30%
Use with AXS instruments	D8, D5005, D5000

Specification (focusing)

Energy of X-rays (starting at 10keV the guiding of the PolyCap is reduced)	5 ... 10 keV (20 keV)
Size of tube focus (point)	0.3x0.3 ... 0.4x1.2
Input area diameter	5.5 +/- 0.4 mm
Opening angle (input)	4.5°
Input focal length	70 +/- 2 mm
Length of lens	100 +/- 2 mm
Divergence with 4.0 mm output aperture	< 8 mrad = approx. 0.5°
Useful beam size at output	>5.5 mm
Transmission	>10%
Output focal length	200 +/- 4 mm
Beam size at output focus	<0.85 mm
Use with AXS instruments	D8, (D5005, D5000)
(For D5000 and D5005 the output focus is not adjustable with respect of the goniometer)	

Advantages for point focus-applications

- Horizontal and vertical parallel beam
- Intensity gain approx. factor 10 to 100 (comparison with equal divergences provided by set up with pinholes)
- Circular spot with about 4.5 mm diameter (FWHM) resp. < approx. 0.85 mm for focusing PolyCap
- Local divergence less than 0.25°(parallel) resp. 0.5°(focusing)
- One optic for various wavelengths Cr, Cu, Co, Fe, (Mo)
  (For Molybdenum the amplification by the PolyCap is less effective.)
- Averaging without oscillation (parallel)
- Reduced defocusing effects (Higher Reduction for focusing PolyCap)

Applications

- Texture
- Residual stress
- Fast Phase analysis
- Micro diffractometry (focusing PolyCap resp. parallel PolyCap with near sample pinhole)
- Single crystal (focusing PolyCap and detector with moderate Energy resolution - better than $\Delta E/E=50\%$)

Prealignment

In case of D8 the distance between outside of mounting plate (part 3), i.e. tube side, and the metal tube (part 15) the distance (fin - 53 mm) must be aligned (D5000 / D5005: fin – 61 mm). fin is the input focal length of the PolyCap (Distance between tube focus and glass of PolyCap). The value of the focal length is written on the metal tube (part 15). The value is 68 mm < fin < 72 mm. The distance can be adjusted with the big ring (fig. 3-1 resp. 3-2 resp. 3-3 resp. 3-5) on the optics holder (part 14). If this is not sufficient the position can be shifted with the help of two fixing rings within the optic holder. After this preparing work the PolyCap can be fixed to the tube of the D8 with four screws (part 6).

⚠ **Caution:**
If the mounting screws are too long the contact switches of the tube will not be activated and the PolyCap will not be rigidly fixed to the tube.

The PolyCap does not change the direction of the X-rays as do e.g. monochromators. Therefore, the tube must be rotated to 0° with the first rotation of the DOF if e.g. a Göebel mirror was mounted before.

In case of a focusing PolyCap the output focal length must be adjusted. For most applications we recommend to put the focus into the goniometer centre. This can be achieved by sliding the tube stand parallel to the track.

Now, put a fluorescent screen (if possible with crosshair) into the sample position. For systems with cradle take the fluorescent screen from the accessories of the cradle and move the instrument to x=z=0, y=8.2, Phi=0°, theta=0°, Chi=90°, 2theta=0°. Y=8.2 mm compensates for the thickness of the screen.

Adjust the horizontal and vertical rotation of the optics holder (part 14) parallel to the base (part 1) of the housing. The translations of the optics holder should be moved to following start positions: Vertical translation screw (translation parallel to High Voltage cable of the tube) must be leveled with the holder. The horizontal translation screw (translation parallel to goniometer program must be approx. 1.5 mm higher than the holder (approx. 5 turns counter clockwise from the leveled position).

Operate generator at standard measurement power (e.g. Cu 40 KV, 40 mA, Cr 35 KV, 40 KV, Mo 50 KV, 40 KV for tubes with long fine focus). All alignments can be done with closed radiation safety box. All changes due to alignment can be judged by looking through the window on a fluorescent screen or by using a detector. The tube should be moved orthogonal to the fluorescent screen.

Warning: Radiation danger: ⚠
There is a high radiation level to the neighbourhood of the opened PolyCap housing if the tube shutter is open. Therefore, we strictly warn opening the safety circuit of the instrument with the service key for hot tuning the PolyCap!

Maybe, you can already see a bright spot inside the weak circle of the white radiation. In this case optimise the intensity by alternate tuning the rotations of optics holder until you have a homogenously illuminated spot

If you do not see a bright spot, tune the rotation along the direction of the smaller tube focus dimension (in case of a long fine focus tube use the back rotation for a vertical goniometer and the lower screw for a horizontal goniometer) +/- 1 rotation in 1/8 turns, until you can see a brighter spot inside the weak circle of the white radiation. Then optimise as described above until the circle is homogenously illuminated.

Horizontal and vertical position and direction of the beam: Move the X-ray spot into the middle of the fluorescent screen, i.e. the centre of rotation, by combining translation and rotation of the PolyCap. Check with the fluorescent screen that the beam hits the middle of the detector slit (especially vertical to the scattering plane). This should be tested for two positions of the slit: close to the goniometer and far away. By this you can find out if the beam has a slope with respect to the scattering plane.

It is helpful to watch the beam spot at the sample position for grazing incident angles (5° ... 10°). The beam ellipse should be parallel to the sample stage. For instruments with translation stages (e.g. systems with a cradle) the fluorescent screen can be moved by motors to find about the slope of the beam.

Alignment

The optimisation of the intensity is performed in two main steps:

1. Optimise intensity
2. Guide beam through centre of rotation

The alignment of the beam direction is somewhat different for theta-theta and theta-2theta systems. Also it depends on the sample stage. Therefore, the alignment will be described for two main configurations:

1. Horizontal theta-2theta instrument with Eulerian cradle (parallel PolyCap with Cu radiation and focusing PolyCap with Cu and Mo radiation)
2. Vertical theta-theta instrument with standard sample stage (parallel PolyCap with Cr radiation).

For other configurations follow the description closest to your system.

Optimising the intensity

For secondary optics use the standard slit system or the detector holder with slit holder. Put a 6 mm slit in front of the detector. The intensities given below are just meant for checking the function and alignment of the PolyCap. They cannot be used to compare the relative intensities at different wavelengths because the emission of the tubes changes a lot with High Voltage at the tube and the hardening resp. absorption of the X-rays is quite different for filters, absorbers, and air.

In addition it is recommended not to optimise the intensity with High Voltages different from the advised ones. Because of hardening or inefficient stimulation of the characteristic Kα radiation the PolyCap might be optimised for the white radiation instead of the characteristic.

Parallel PolyCap, 40 KV, 0.2 mm Cu, without Kβ-Filter, 300 mm between tube focus and goniometer axis, Detector at the end of the long track.

Anode	Generator Voltage	I(direct)/cps
Cr	20 kV	50000
Co	20 kV	30000
Cu	20 kV	90000
Mo	20 kV	75000

Focusing PolyCap, 40 KV, 0.2 mm Cu, 300 mm between tube focus and goniometer axis, Detector at the end of the long track.

Anode	Generator Voltage	I(direct)/cps
Cr	20 kV, 10 μm V	4000
Cu	20 kV, 12 μm Ni	65000
Mo	30 kV, 65 μm Zr	22000

Optimise both rotation of the optics holder until the intensity is at least 80% of the intensity given.

Guiding beam through centre of goniometer

The alignment of parallel and focusing PolyCap is quite similar. We just recommend to position the output focus into the sample i.e. the goniometer axis. All alignments should be done at 20 KV (30 KV for Mo) and 40 mA. Alignment at higher HV emphasize the white radiation and might lead to misinterpretation of the direct beam measurements. We recommend to use the detector holder with slit for alignment purposes if nothing different is written.

☞ **Hint:**
Never use empty slit holders directly in front of scintillation detectors! Magnetic fields of the slit holders may shift the working point of the detector. This is avoided by always putting a slit.

The back of the detector should be at level with the secondary track. This makes the measurements comparable to the examples given. At the same this avoids collisions. On the primary side the tube should be shifted to 600 mm diameter measurement circle (parallel PolyCap, focusing PolyCap must be adjusted with respect of the output focus). The other standard conditions are: 0.2 mm Cu absorber, 0.012 mm Ni filter for Cu radiation, (Pos. 7 in fig. 3-1 and 3-2). Here we give examples for Cu and Mo radiation (theta-2theta, horizontal) and Cr Strahlung (theta-theta, vertical).

Theta-2Theta

1. **Slope of beam:**
   Detector scan at 0° +/- 5°with 6 mm Detector slit (fig. 3-7) and 0.2 mm Cu. Afterwards perform a detector scan with diagonal slit C79298-A3158-B90 (fig. 3-8). If the maximum position of the two scans differs by more than 0.04° the slope of the beam must be corrected. This can be achieved easiest by the second rotation of the 4DOF. If this degree of freedom is not available the PolyCap must be translated vertical to the scattering plane. Of course the intensity must be tuned to maximum again by the corresponding rotation of the optics holder.

   These two scans and the tuning must be repeated until the error is smaller than 0.04°.
   Fig. 3-9 shows the danger of too high generator voltage: At 40 KV the 8 KV Cu Kα radiation is just a sharp reflection on a broad plateau. Both reflections do not have the same centre of mass! For different choices of slits or generator HV it might even happen that the sharp Cu reflection cannot be seen at all.

2. **Parallelism to Track:**
   This step is necessary only if the distance between tube and sample shall be changed quite often (several times a week). In most cases the optical alignment described above will be sufficient. This optimisation can also be performed after installation of the instrument.

As for the alignment of the Göbel mirrors in D8 Discover for high resolution (cf. D8 manual part 2) the beam position must be determined for two distances between tube and sample. In the first position the tube should be moved as far from the goniometer as possible and in the second as close as possible. The position can be measured via a x-scan with the pinhole for Chi=90°. The first rotation of the DOF must be used to make the difference in x less than 0.2 mm. Afterwards, the first translation of the DOF can be used to move the beam orthogonal to the track until the maximum in x is at 0 +/- 0.1 mm.

3. **Distance to Goniometer:**
   The tube must now be shifted to the desired distance from the goniometer, i.e. 200 mm between end of lens (part 17) and centre of goniometer for the focusing PolyCap. For the parallel PolyCap this distance should be minimised to reduce air scattering and absorption. Of course you must avoid possible collisions. For systems with cradle the 600 mm measurement circle diameter is recommended.

4. **Height with respect to scattering plane:**
   With the third degree of the DOF (vertical translation) the height of the beam above the goniometer must be adjusted. In a first step this can be done with the help of the crosshair on the fluorescent screen. For optimum precision you must perform a x-scan with pinhole (accessories of cradle: underneath the fluorescent screen) at Chi=Phi=theta=y=z=0 across the primary beam. The height must be corrected until x=0 +/- 0.05 mm (fig. 3-11), provided that the Eulerian cradle has been centred in advance.

5. **Centring to Goniometer:**
   Now a fine tuning of the beam through the goniometer centre must be done with the first rotation of the DOF. The optimisation must be repeated until the maximum of a x-scan for Chi=90° is at x = 0 +/- 0.05 mm (fig. 3-10).

6. **Zero position 2Θ:**
   Take out the pinhole. Perform a detector scan with 0.1 mm detector slit. The position of the maximum must be reset to zero (new ZI value of 2theta), 2theta = 0° +/- 0.04° (fig. 3-12)

7. **Zero position Θ:**
   Mount glass slit. Scan theta with 6 mm detector slit for 2theta=x=y=z=Chi=0 and Phi=90°. Use position of maximum for redefining theta=0 (+/- 0.04°) (fig. 3-13).

8. **Control of alignment:**
   Exchange glass slit with quartz sample. Measure 100% reflection with 1 mm detector slit. By this the alignment is checked. The nominal position of the 100% reflection (ICDD 46-1045) must be found within 0.05°: 26.640° for Cu Ka (fig. 3-14), 40.048° for Cr Ka, 31.035° for CoKa, and 12.178° for MoKa. If this is not the case the whole alignment must be repeated.
   A measurement with slits is of course not adequate for a parallel optic. Therefore, we recommend to use the 0.4° grazing incidence Soller as a standard secondary optic.

Fig. 3-15 shows such a measurement for Quartz.
We recommend to use just a wide detector slit - e.g. 6 mm – if intensity is more important than a good compromise between intensity and resolution.

Theta-Theta (vertical and without Eulerian cradle)

1. **Slope of beam:**
   Perform detector scan at 0° +/- 5° with 0.1° steps without sample, 0.2 mm Cu absorber and 0.1 mm detector slit. The position is correct if the maximum of the Kα reflection is at 0° +/- 0.04°. Otherwise the position must be adjusted with the first rotation of the tube stand (DOF). For D5000/D5005 the correspondent alignment screws (e.g. take off angle) can be used if available. If this is not the case the PolyCap must be translated in it's optics holder with subsequent optmisation of the intensity with the rotation of the PolyCap. Preferable, this tuning should be done with low HV (20 KV, resp. 30 KV for Mo) to get now alignment errors because of white radiation (cf. theta-2theta). In Fig. 3-32 the measurement has been done at 35 KV (Cr radiation). Due to this the Kα radiation is a weak reflection on a broad plateau of white radition. The centre of mass of the two types of radiation is not necessarily the same.
   Next repeat the detector scan at +/- 5° with 0.1° steps without sample, 0.2 Cu absorber, with diagonal slit C79298-A3158-B90. The position is allowed to differ by 0.04° from the first one (cf. Alignment theta-2theta). If the difference is larger the beam has a significant slope with respect to the scattering plane. In this case the PolyCap must be translated vetical to the scattering plane. The intensity must be optimised again by rotation. This changes the inclination of the beam because the PolyCap is now looking at a different angle towards the focus of the tube.
   The PolyCap must be shifted as long as the two detectors scans (standard and diagonal slit) differ by more than 0.04°.

2. **Height of beam with respect of scattering plane:**
   With the third degree of freedom of theDOF the height of the beam with respect to the scattering plane must be adjusted. Of course, for a vertical goniometer this is a horizontal movement. The precision gained during optical prealignment should be sufficient for a vertical theta-theta goniometer, i.e. +/- 0.5 mm.

3. **Centring to Goniometer, Zero Position Tube:**
   Perform tubescan with glass slit, 6 mm antiscatter slit, 6 mm detector slit, 0.1 mm Cu absorber. The area above and below the glass slit should be covered by something absorbing (e.g. lead tape). The beam of the parallel PolyCap is broader than the whole slit. Part of the beam might pass above or below and could lead to wrong results. Reset ZI-value of the tube to 0° +/- 0.04° (cf. fig. 3-5).

4. **Zero Position Detector:**
   Perform detector scan with glass slit, 6 mm anti scatter slit, 0.1 mm detektor slit. Define new detector ZI value to 0° +/- 0.04° (cf. fig. 3-3)

5. **Checking centre of rotation**:
   Repeat detector scan with 0.1 mm detector slit (step 1 theta-theta). The position of the maximum must be 0° +/- 0.04°. If this not fulfilled steps 1 to 5 must be done once again.

6. **Checking alignment:**
   At last the whole alignment must be checked with a measurement of the 100% reflection of quartz with 6 mm anti scatter and 6 mm detector slit: The nominal position (ICDD 46-1045) should differ by no more than 0.05° (26.640° for Cu Kα, 40.048° for Cr Kα, 31.035° for Co Kα und 12.178° for Mo Kα (fig. 3-35, example Cr radiation). If this precision is not achieved, step 1 – 5 must be repeated.

Figures

Technical Drawings

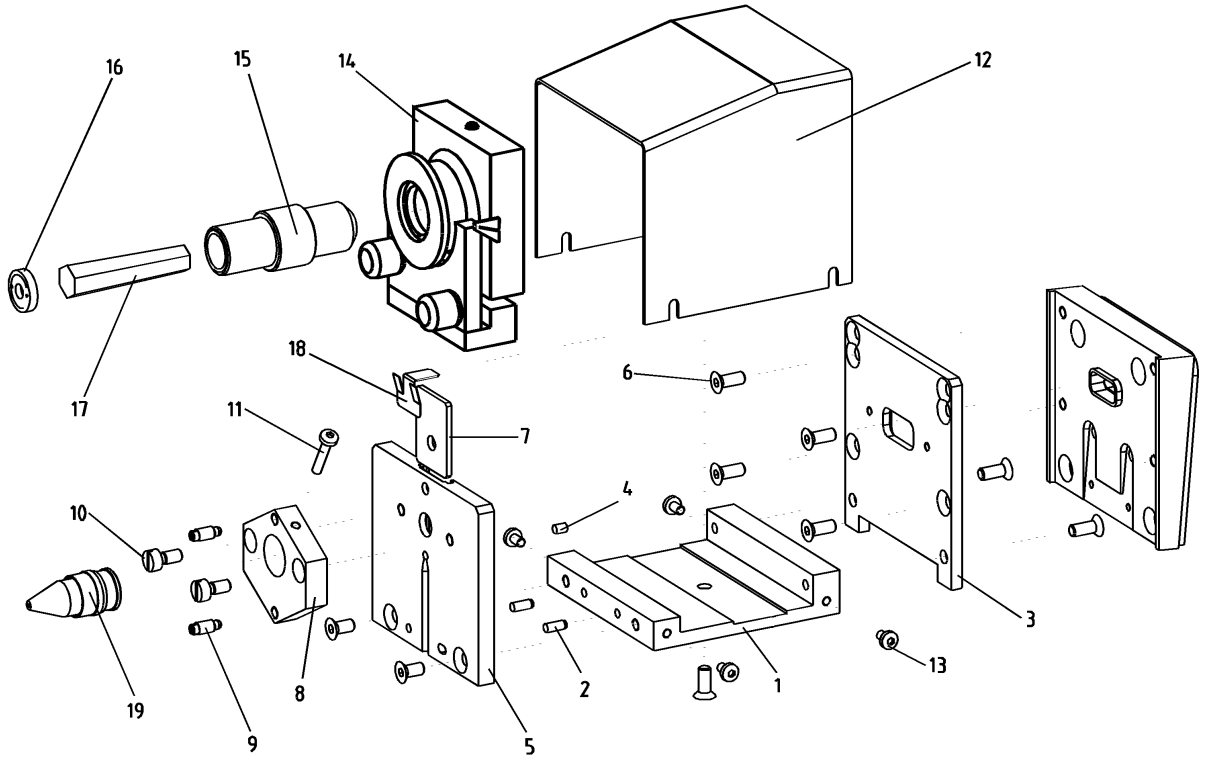


Fig. 3-1: Mounting the parallel PolyCap.

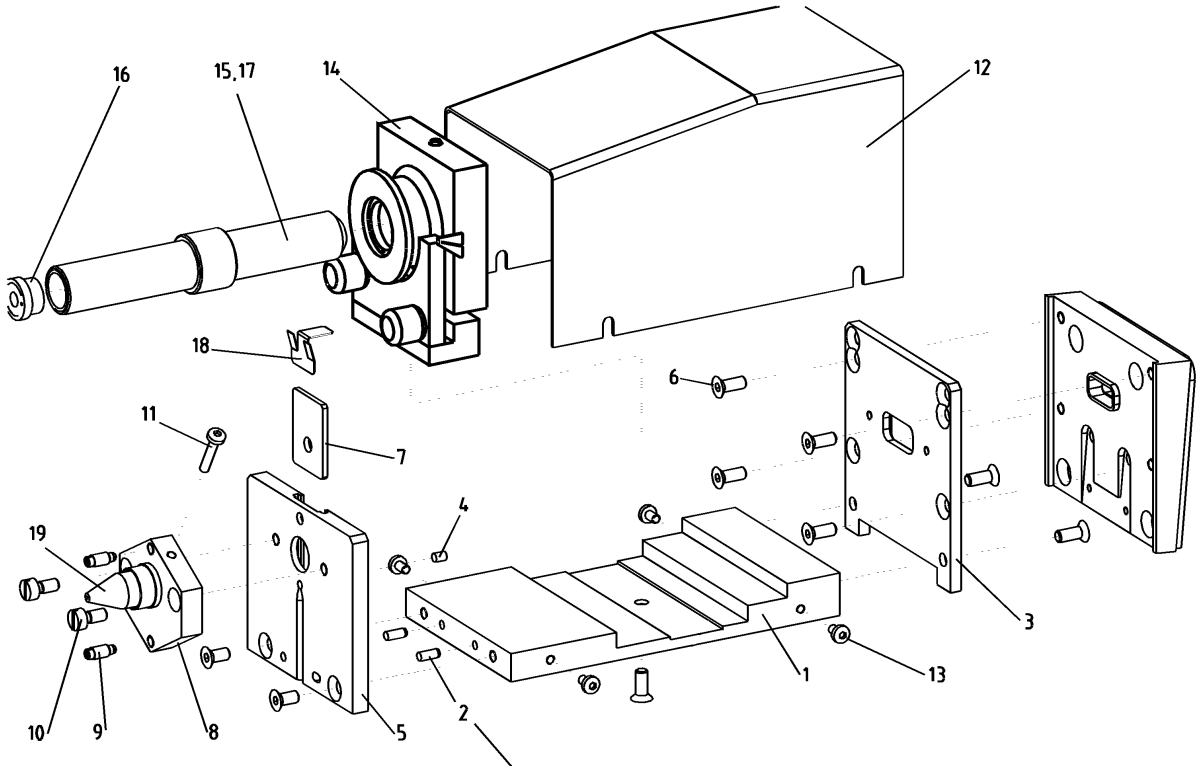


Fig. 3-2: Mounting the focusing PolyCap.

Views on the mechanics

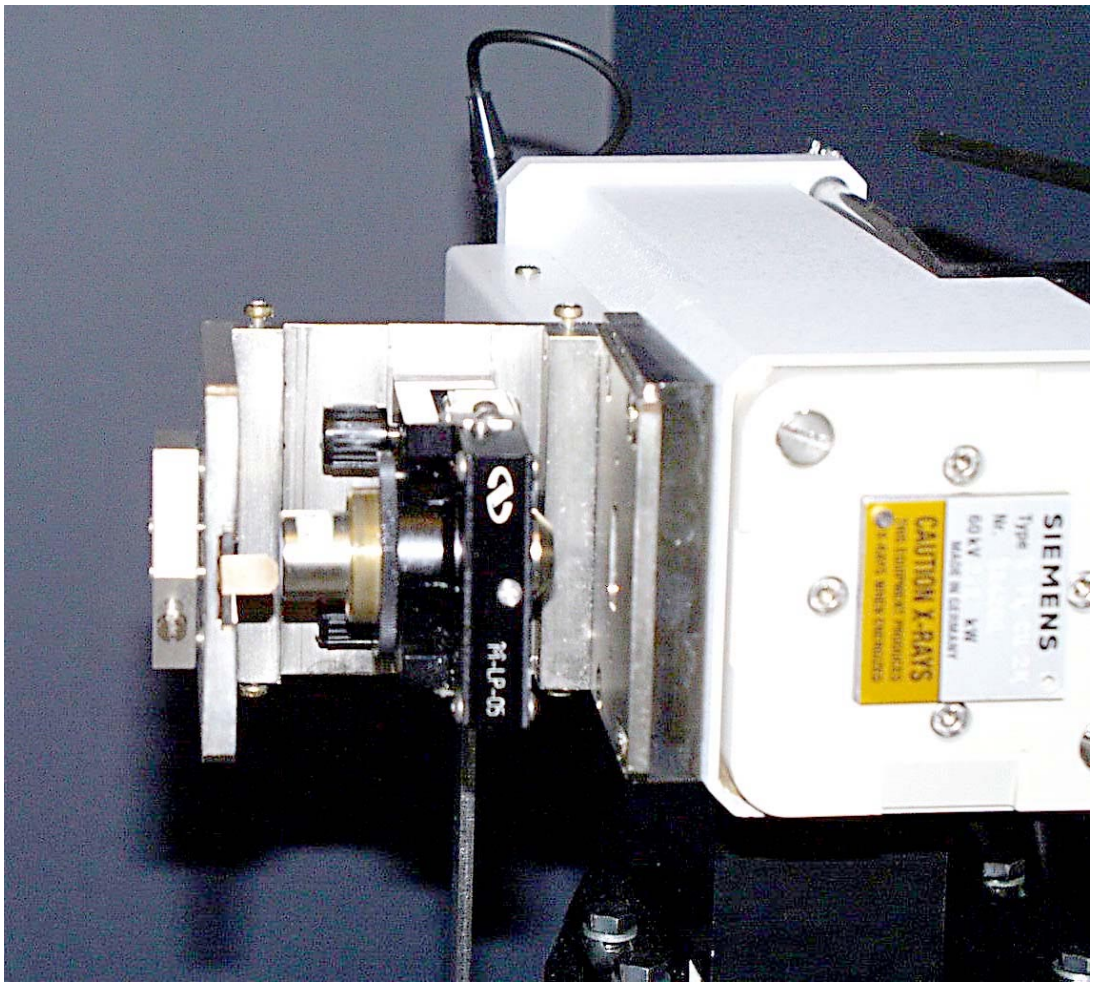


Fig. 3-3: View on the mechanics of the mounted parallel PolyCap.

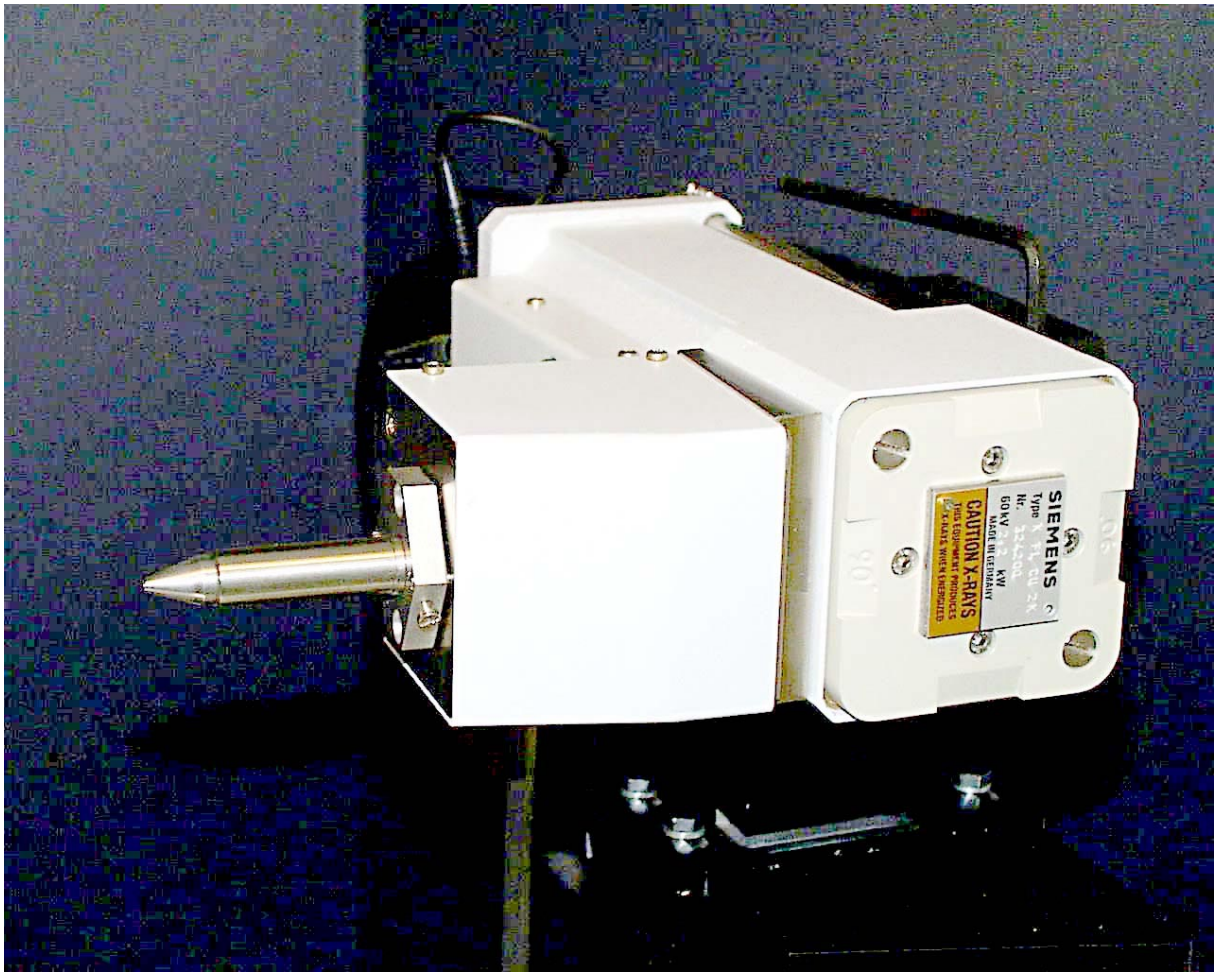


Fig. 3-4: View on complete parallel PolyCap with near sample pinhole.

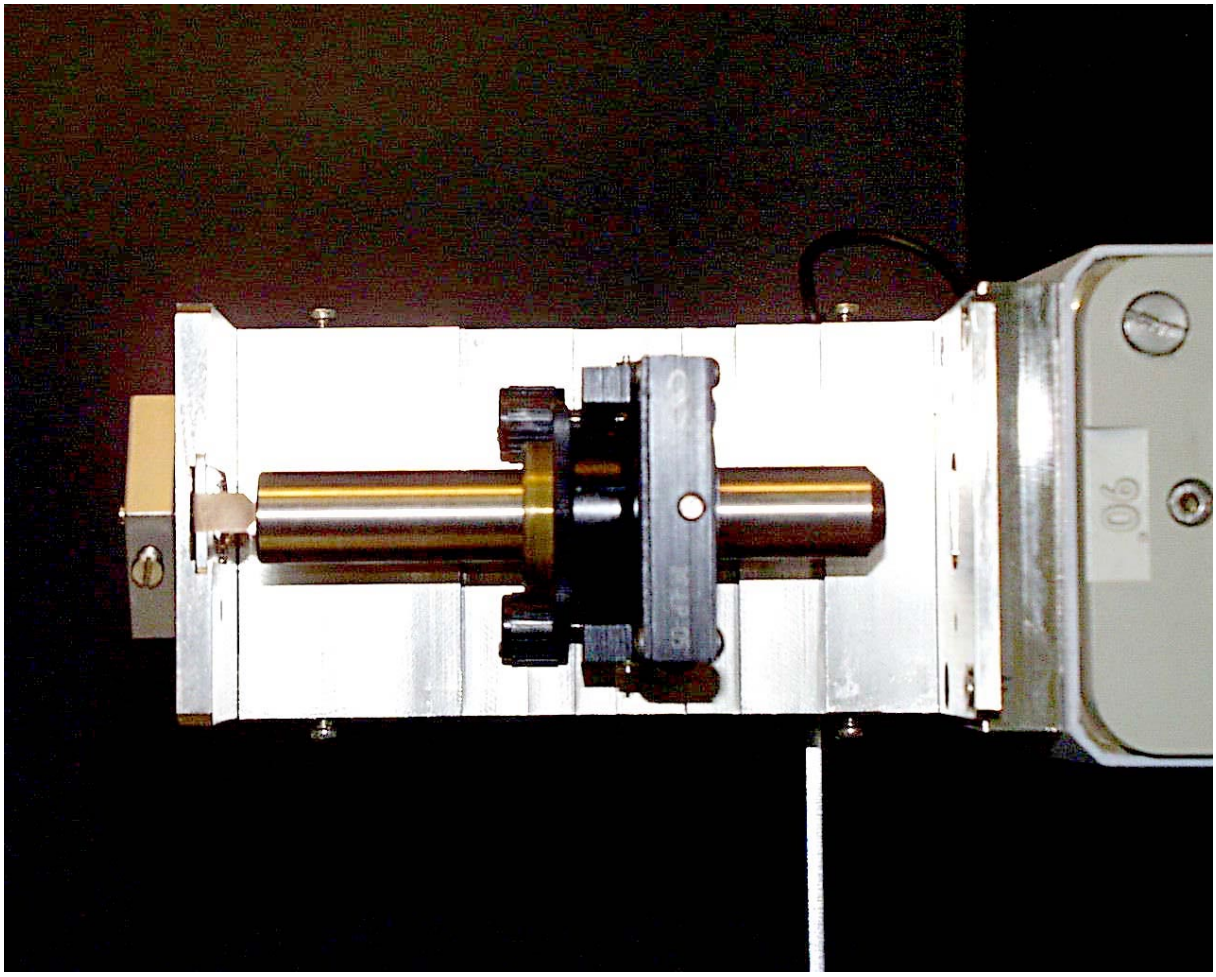


Fig. 3-5: View on the mechanics of the mounted focusing PolyCap.

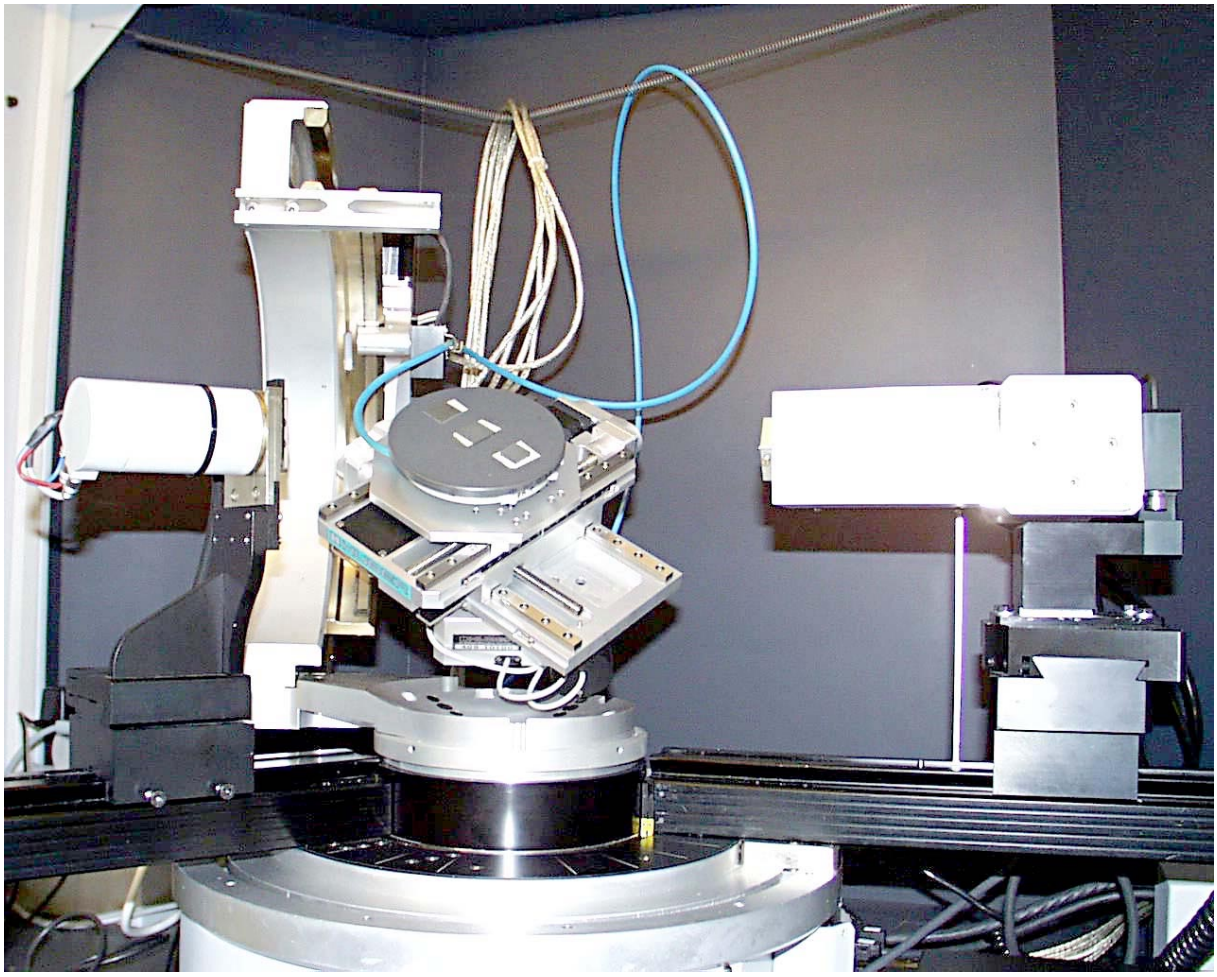


Fig. 3-6: View on complete focusing PolyCap mounted on a horizontal theta-2theta D8 Discover with ¼ circle Eulerian cradle.

Alignment of Parallel PolyCap with Cu radiation on horizontal theta-2theta with cradle

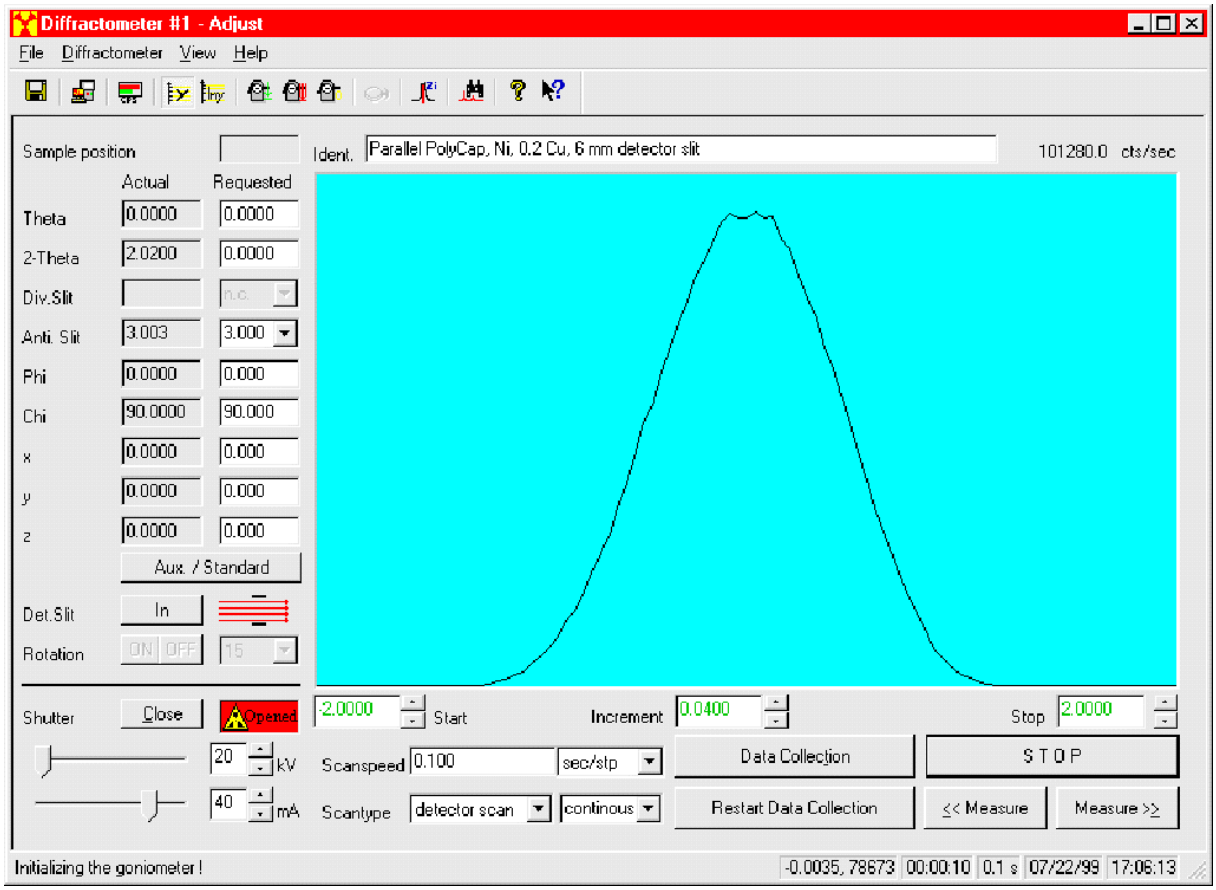


Fig. 3-7: Primary beam of parallel PolyCap with Cu tube, Ni filter, 0.2 mm Cu absorber, 6 mm detector slit, at 20 KV and 40mA.

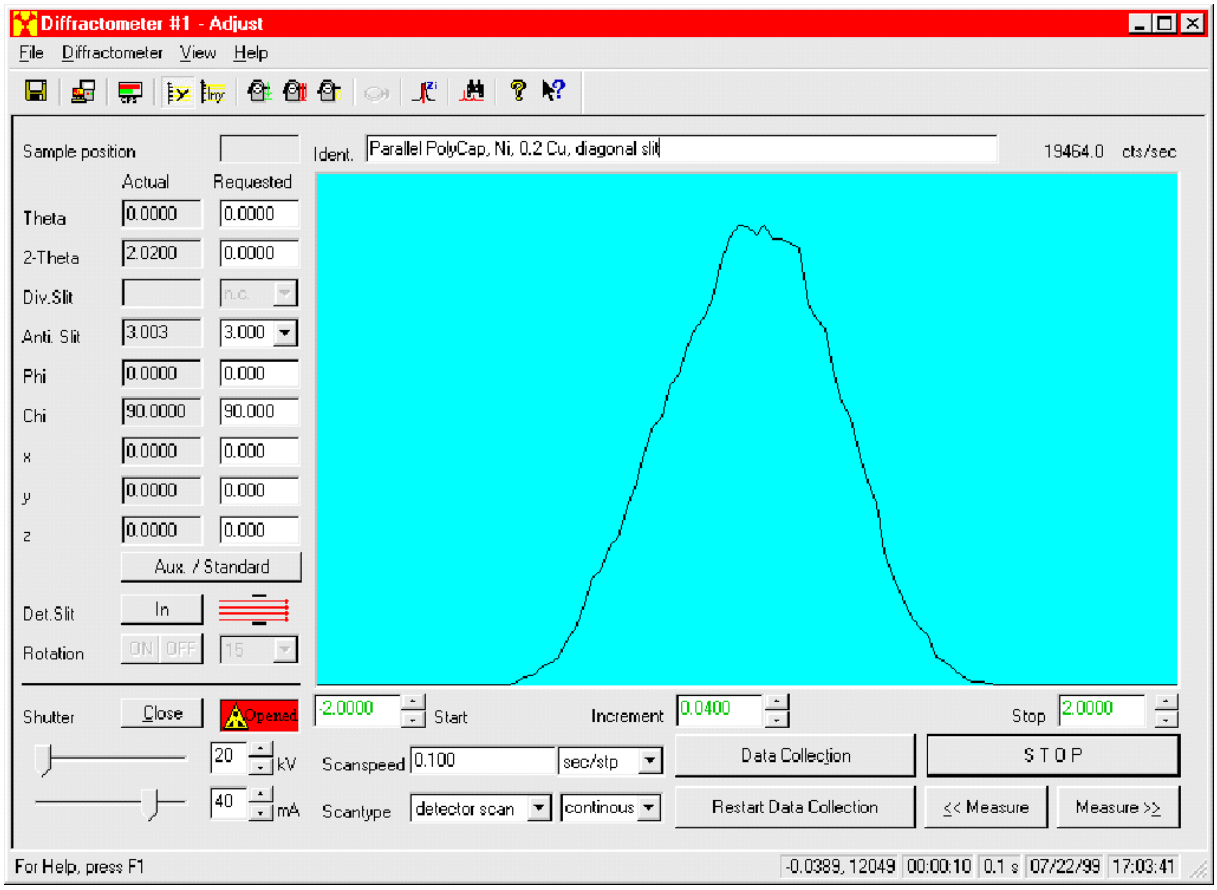


Fig. 3-8: Primary beam of parallel PolyCap measured with diagonal slit: Check of beam slope.

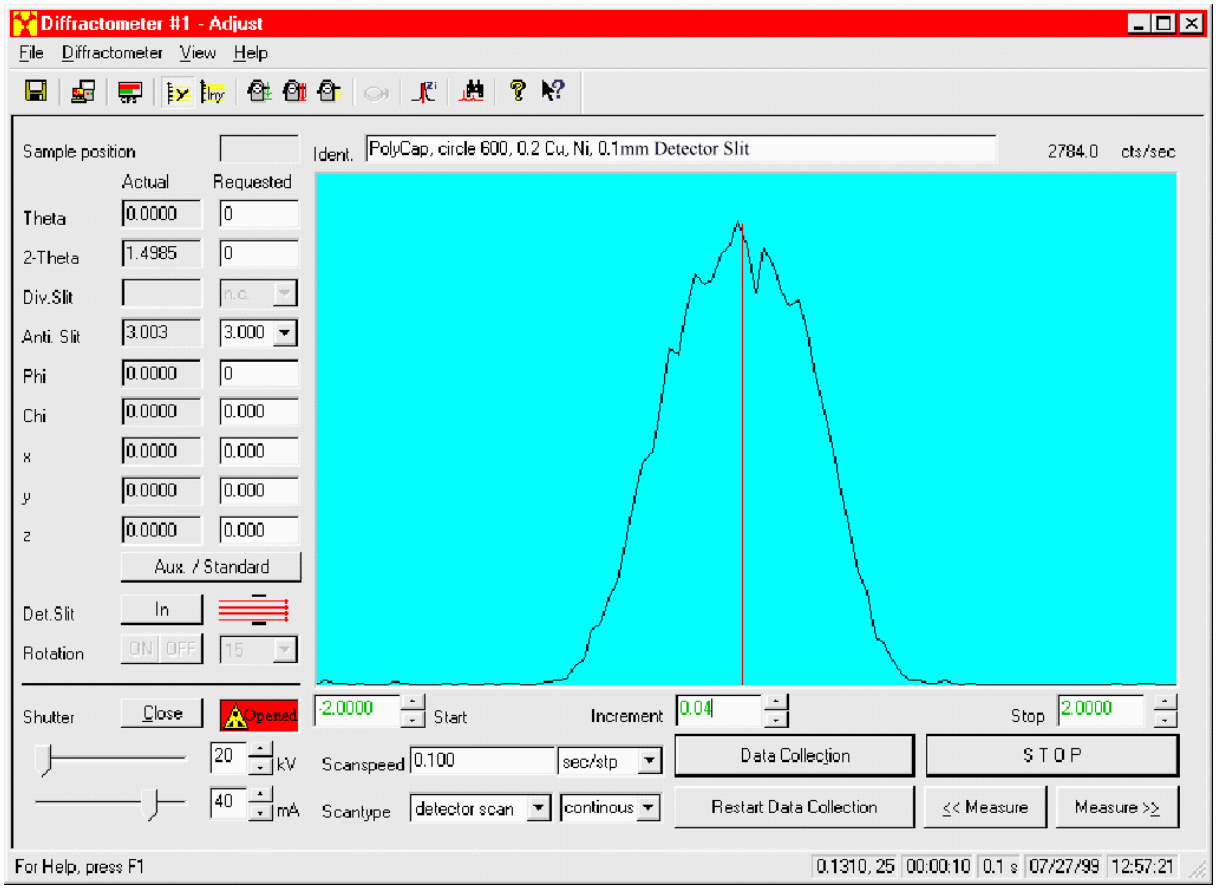


Fig. 3-9: Primary beam of parallel PolyCap (Cu tube) at 40 KV with 0.1 mm detector slit: Risks of high voltage during primary beam alignment – the sharp peak on top of the plateau is the interesting part.

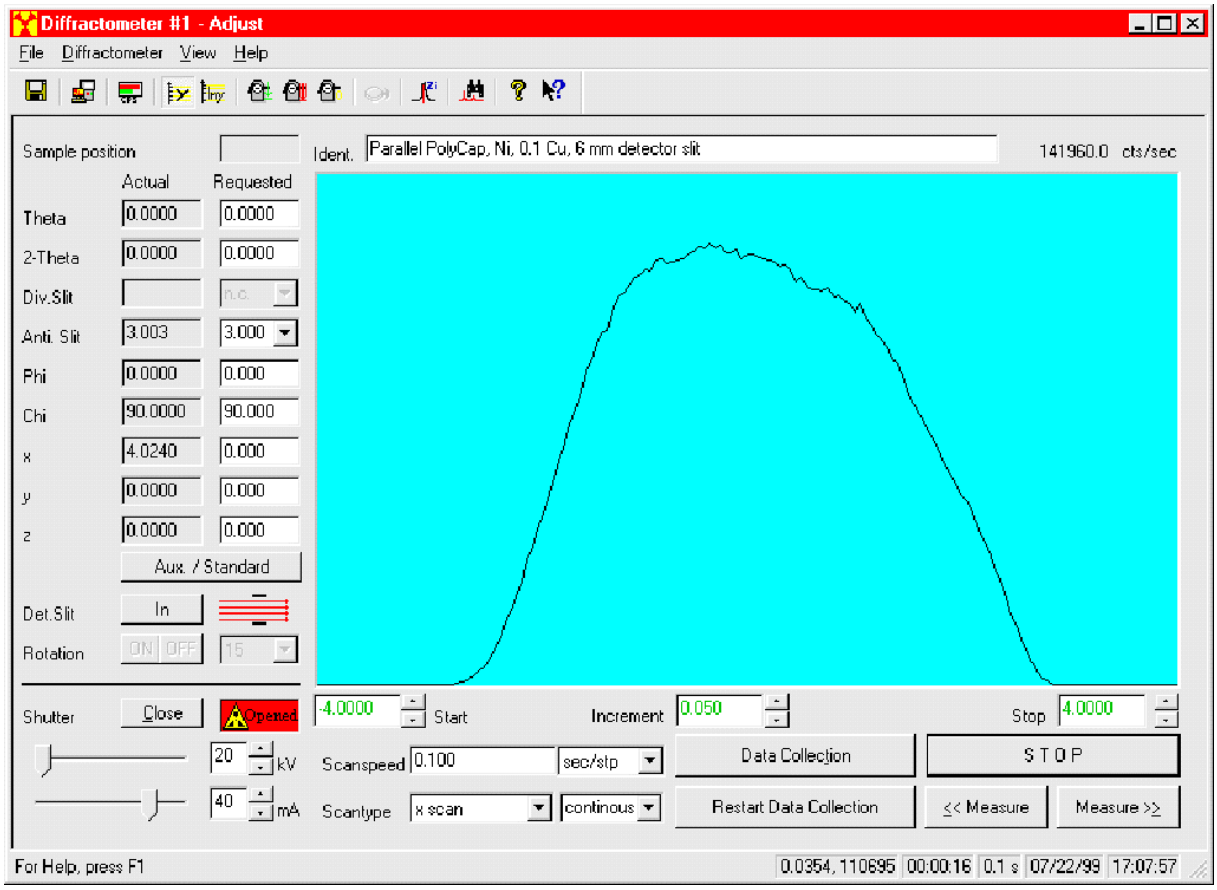


Fig. 3-10: Horizontal beam position for parallel PolyCap (Cu radiation) – X-Scan with pinhole in scattering plane.

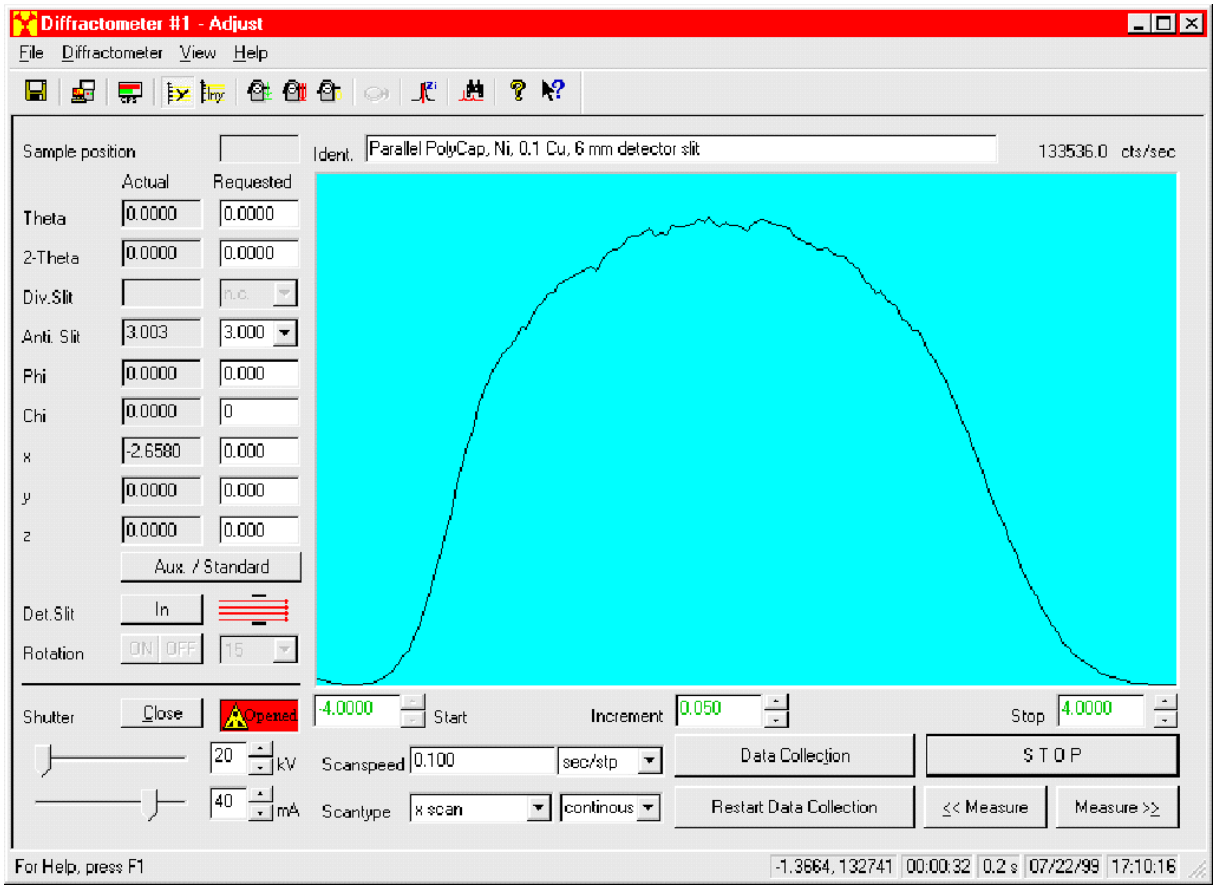


Fig. 3-11: Vertical beam position for parallel PolyCap (Cu radiation) – X-Scan with pinhole normal to scattering plane.

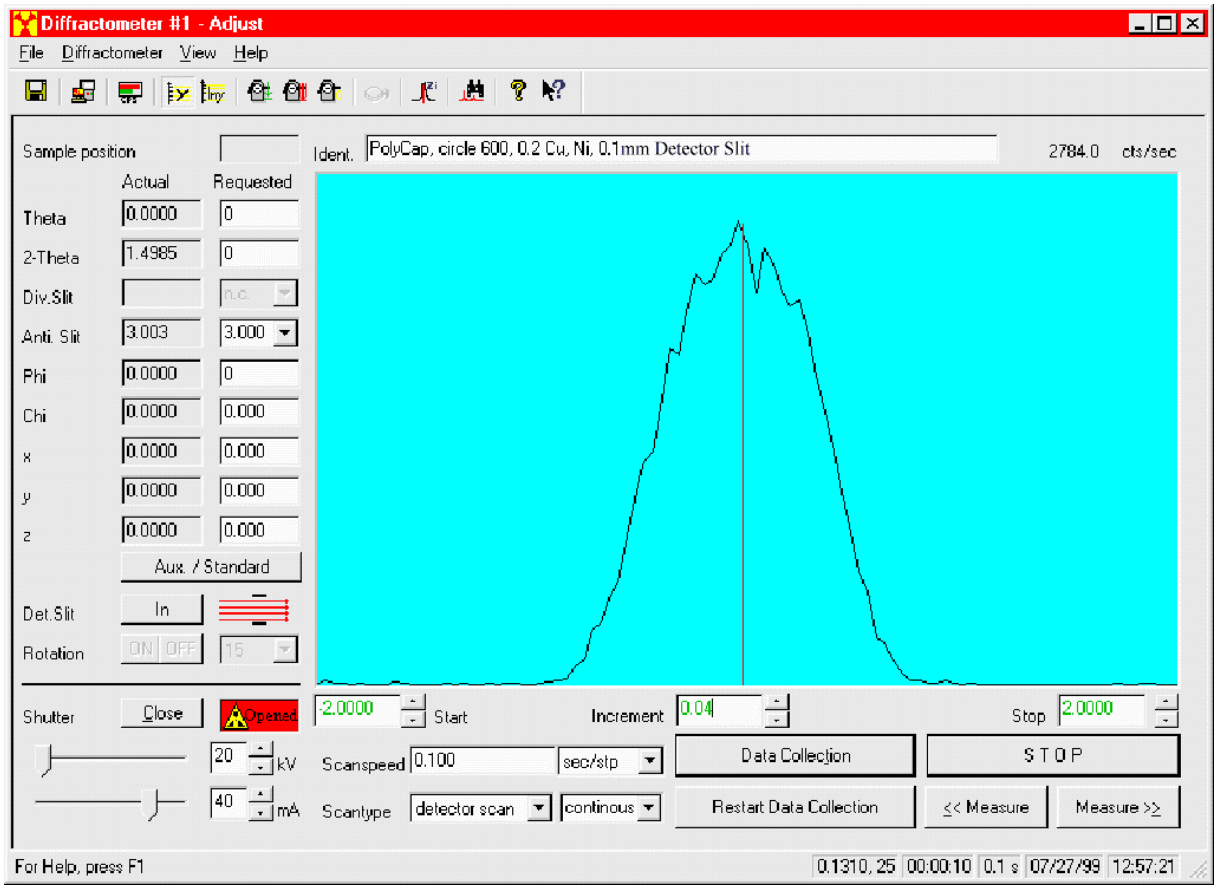


Fig. 3-12: Detector scan through primary beam for parallel PolyCap (Cu radiation): Zero Position of 2theta.

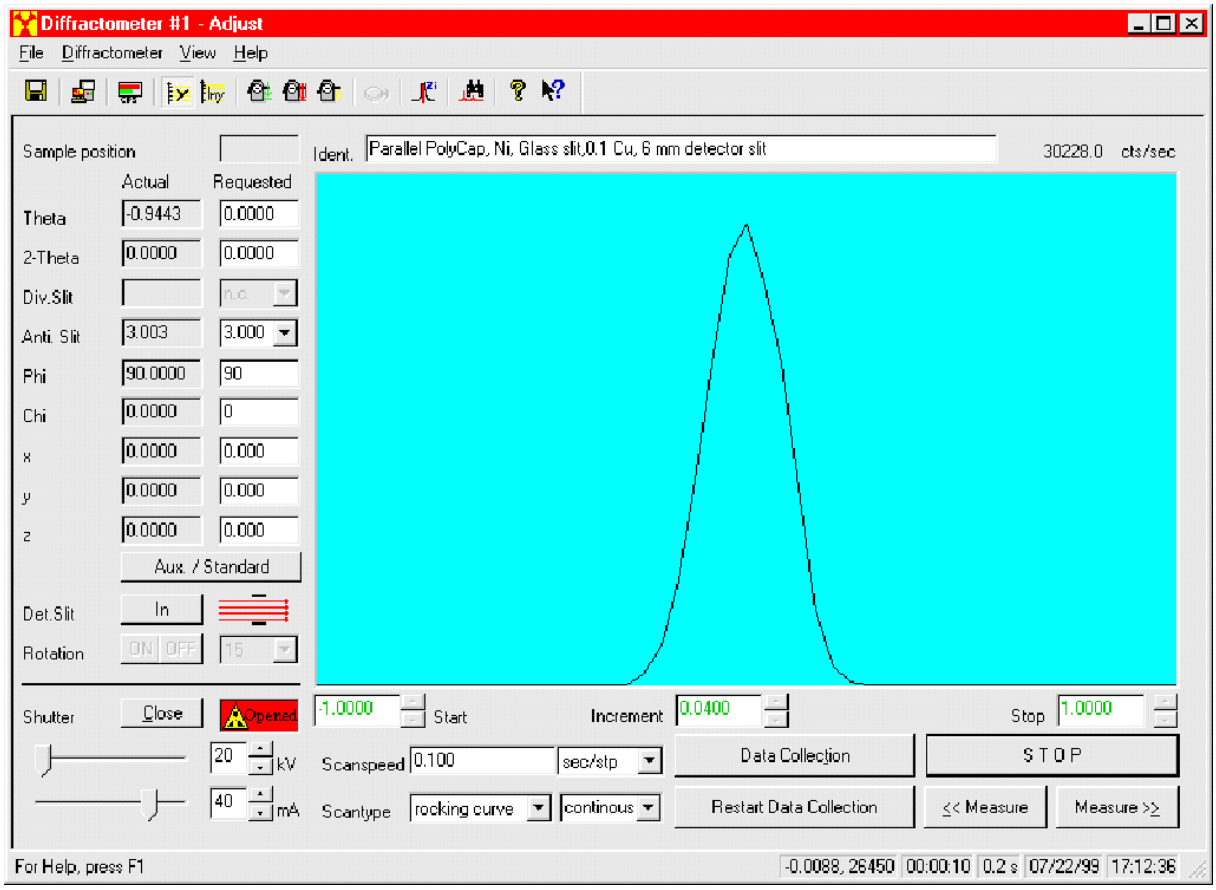


Fig. 3-13: Rocking curve with glass slit for parallel PolyCap (Cu radiation): Zero position of theta.

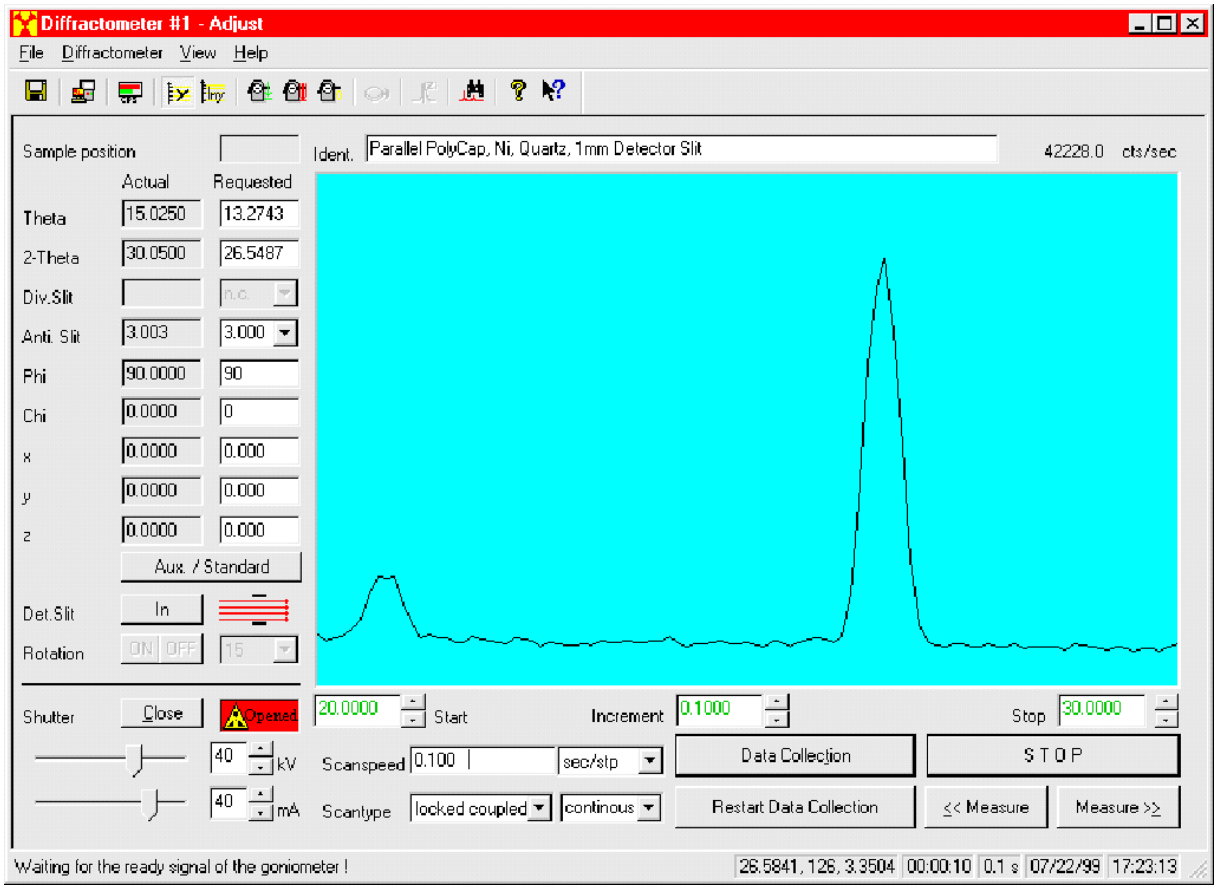


Fig. 3-14: 100% reflection of quartz measured with 1 mm detector slit (Cu radiation): Check of alignment.

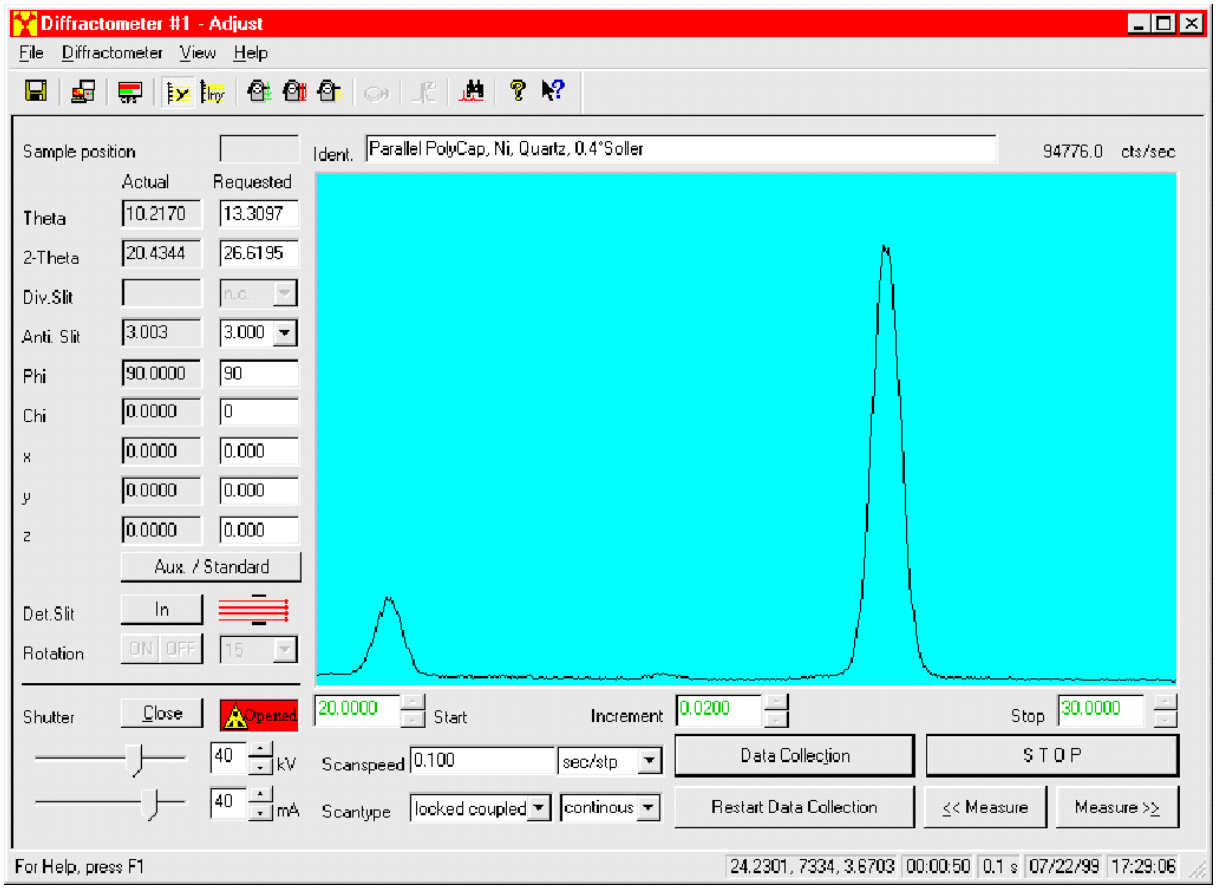


Fig. 3-15: 100% Reflection of quartz measured with 0.4° grazing incidence Soller (Cu radiation): Better intensity together with better resolution.

Alignment of Focusing PolyCap with Cu radiation on horizontal theta-2theta with cradle

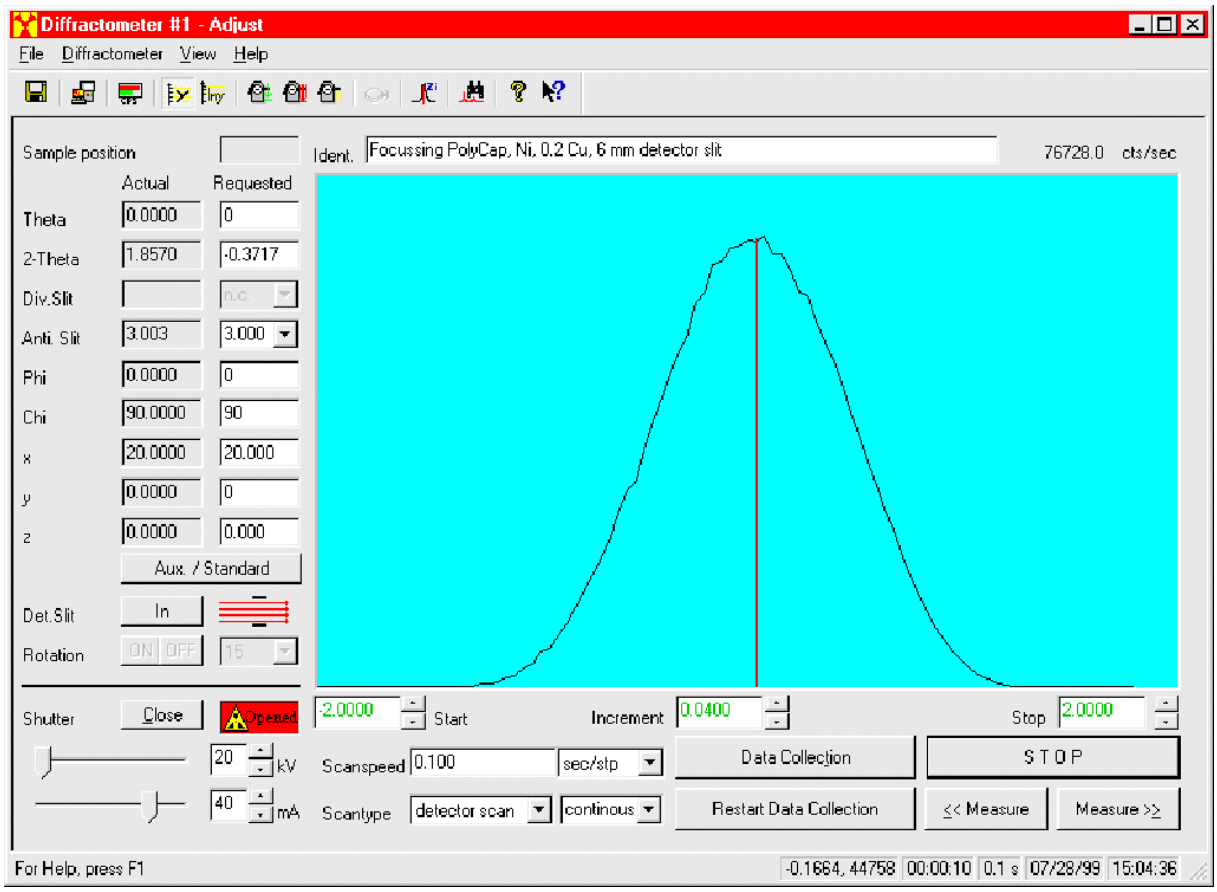


Fig. 3-16: Primary beam of focusing PolyCap with Cu tube, Ni filter, 0.2 mm Cu absorber, 6 mm detector slit, at 20 KV and 40mA. The maximum intensity is lower than that gained with the parallel PolyCap.

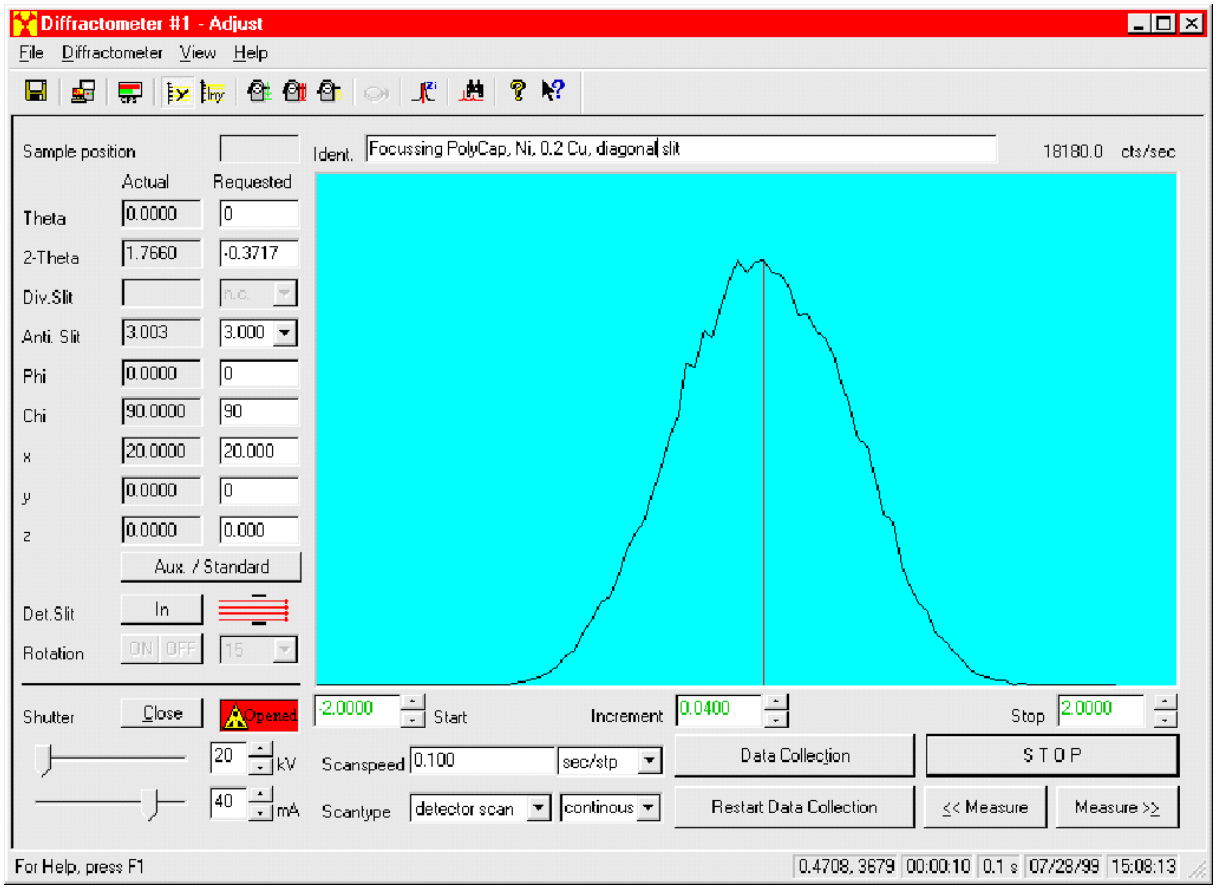


Fig. 3-17: Primary beam of focusing PolyCap measured with diagonal slit: Check of beam slope.

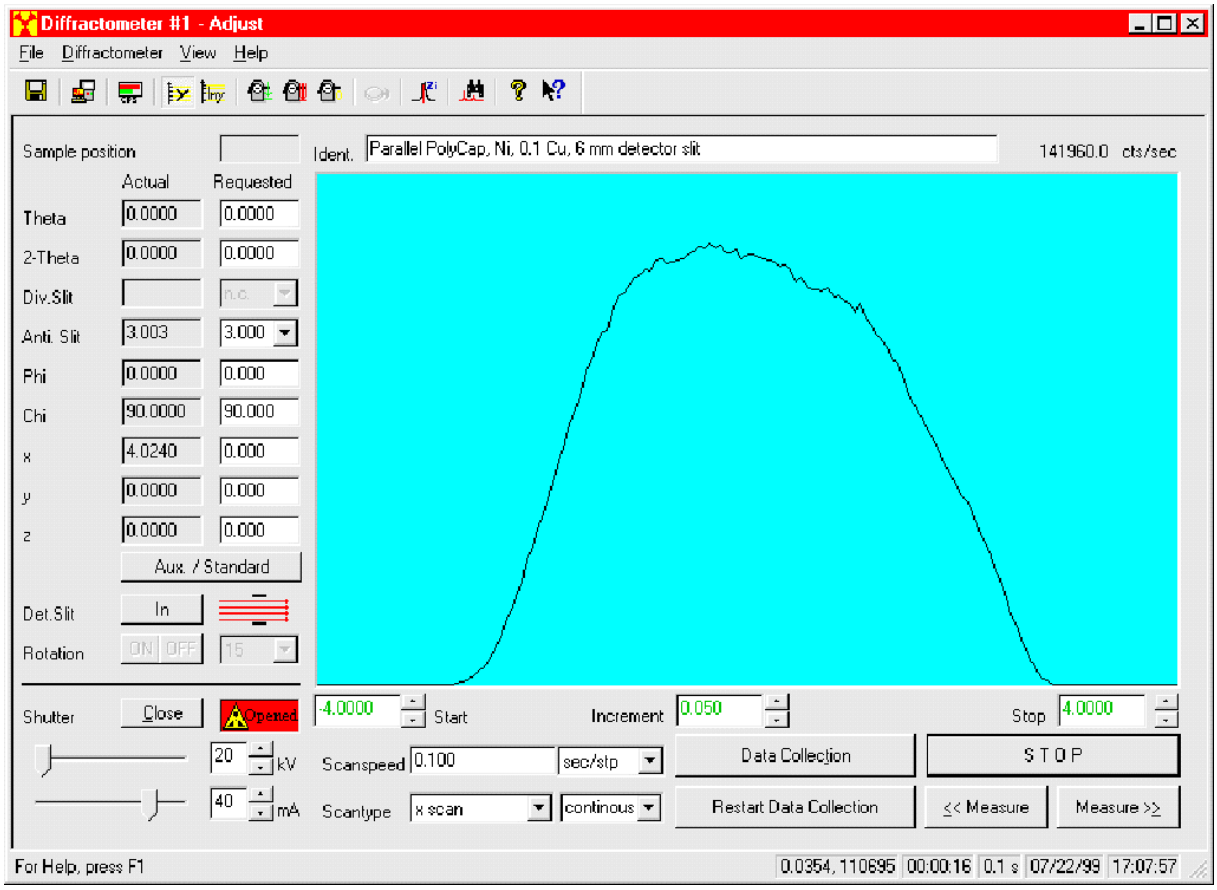


Fig. 3-18: Horizontal beam position for focusing PolyCap (Cu radiation) – X-Scan with pinhole in scattering plane. Note that the beam is much sharper compared to the parallel PolyCap.

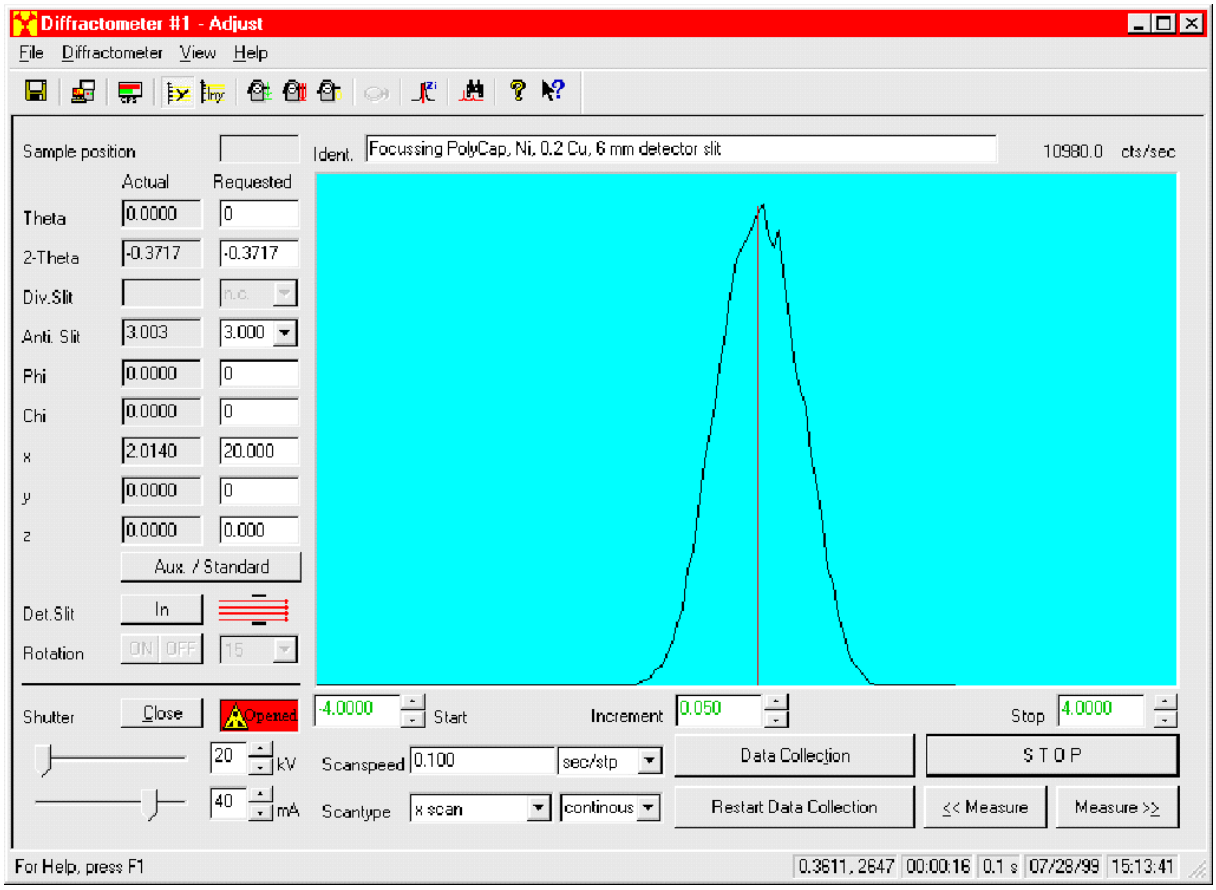


Fig. 3-19: Vertical beam position for focusing PolyCap (Cu radiation) – X-Scan with pinhole normal to scattering plane.

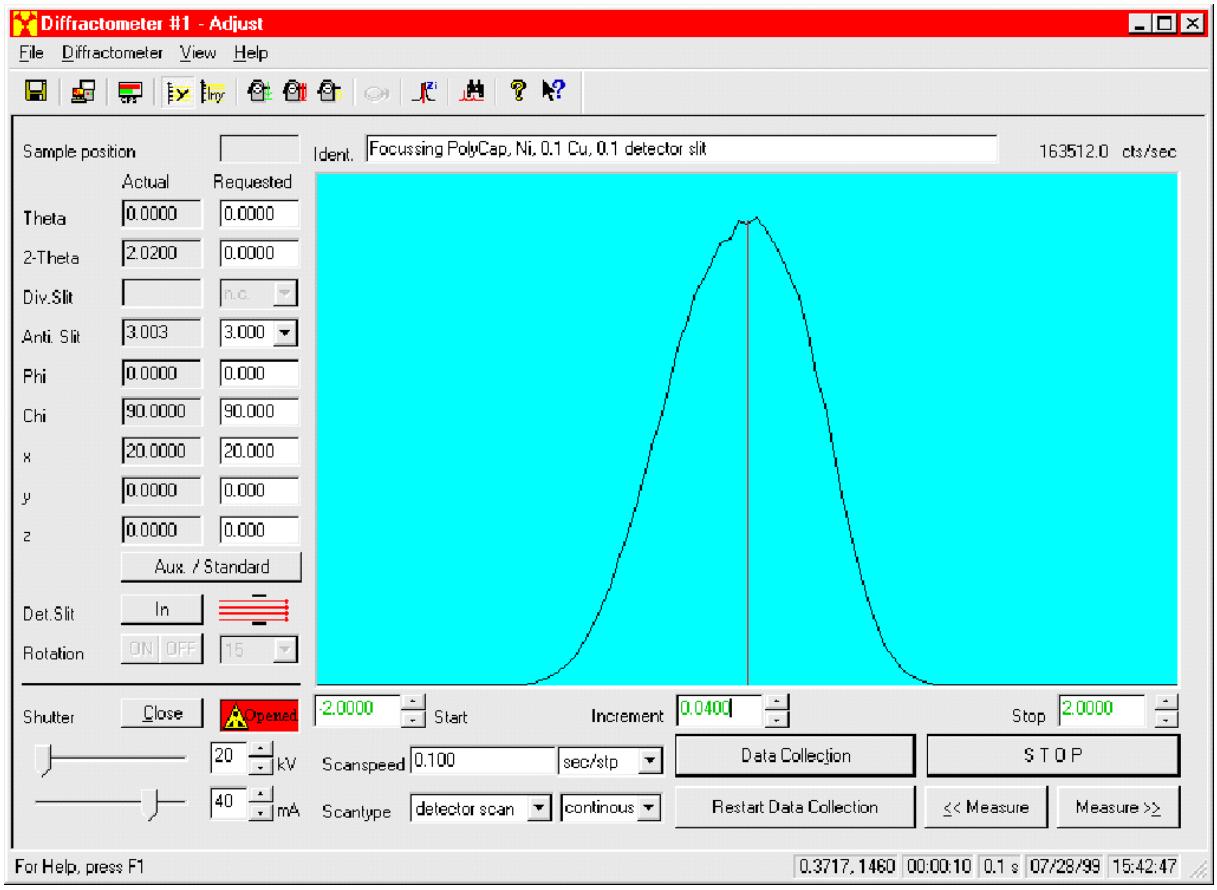


Fig. 3-20: Detector scan through primary beam for focusing PolyCap (Cu radiation): Zero Position of 2theta.

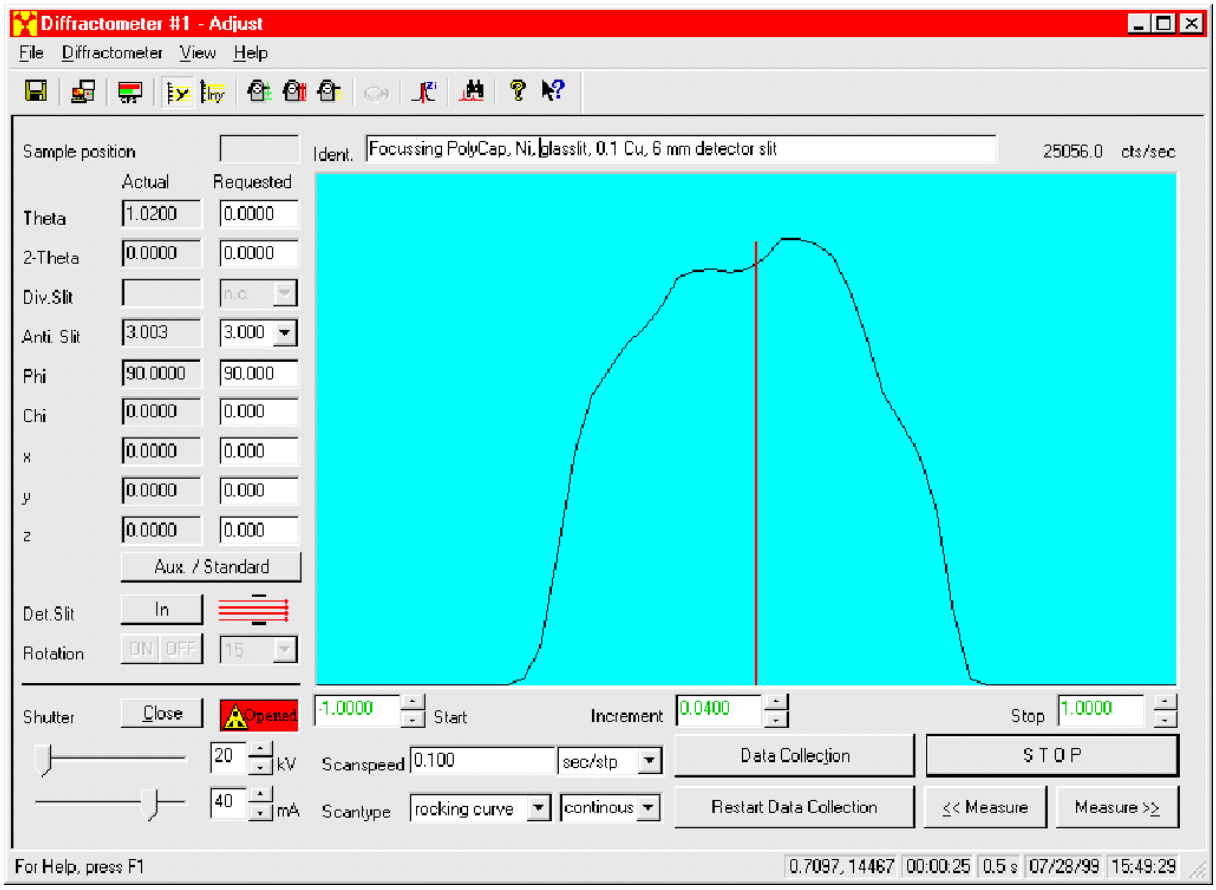


Fig. 3-21: Rocking curve with glass slit for focusing PolyCap (Cu radiation): Zero position of theta. The peak is broader than measured with parallel PolyCap because of the focusing, i.e. converging beam.

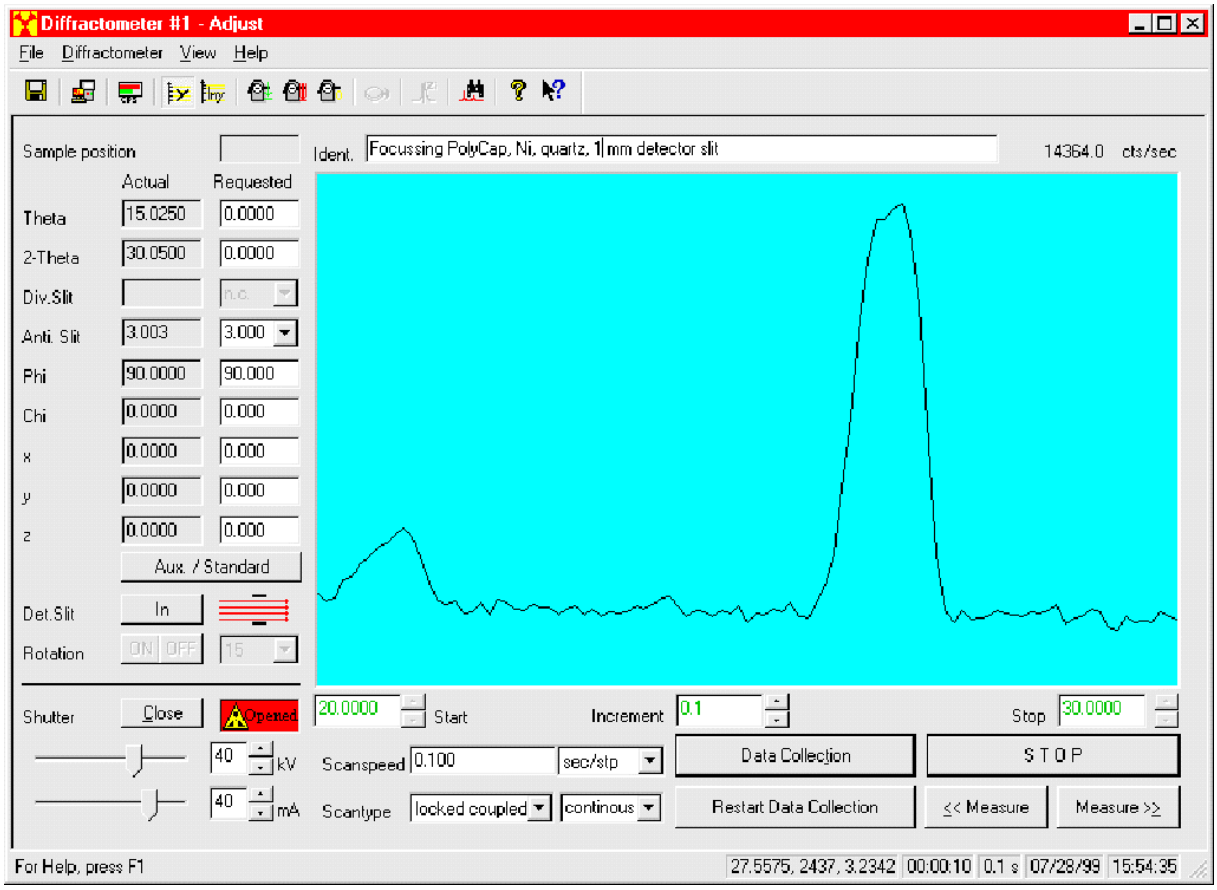


Fig. 3-22: 100% reflection of quartz measured with 1 mm detector slit (Cu radiation): Check of alignment. The intensity is lower than with the parallel PolyCap but the sample area from which the intensity stems is much smaller.

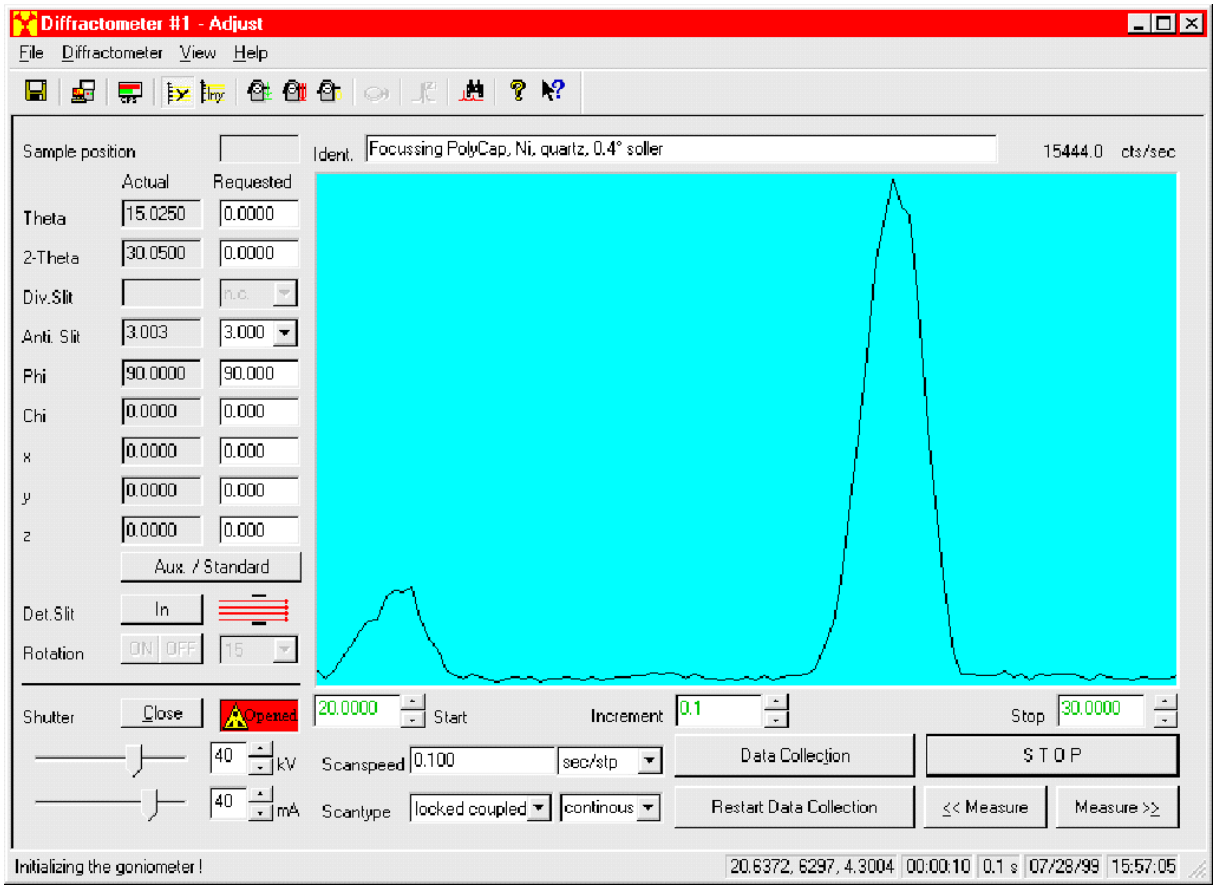


Fig. 3-23: 100% Reflection of quartz measured with 0.4° grazing incidence Soller (Cu radiation): There is no big advantage of the grazing incidence Soller for the focusing PolyCap.

Alignment of Focusing PolyCap with Mo radiation on horizontal theta-2theta with cradle

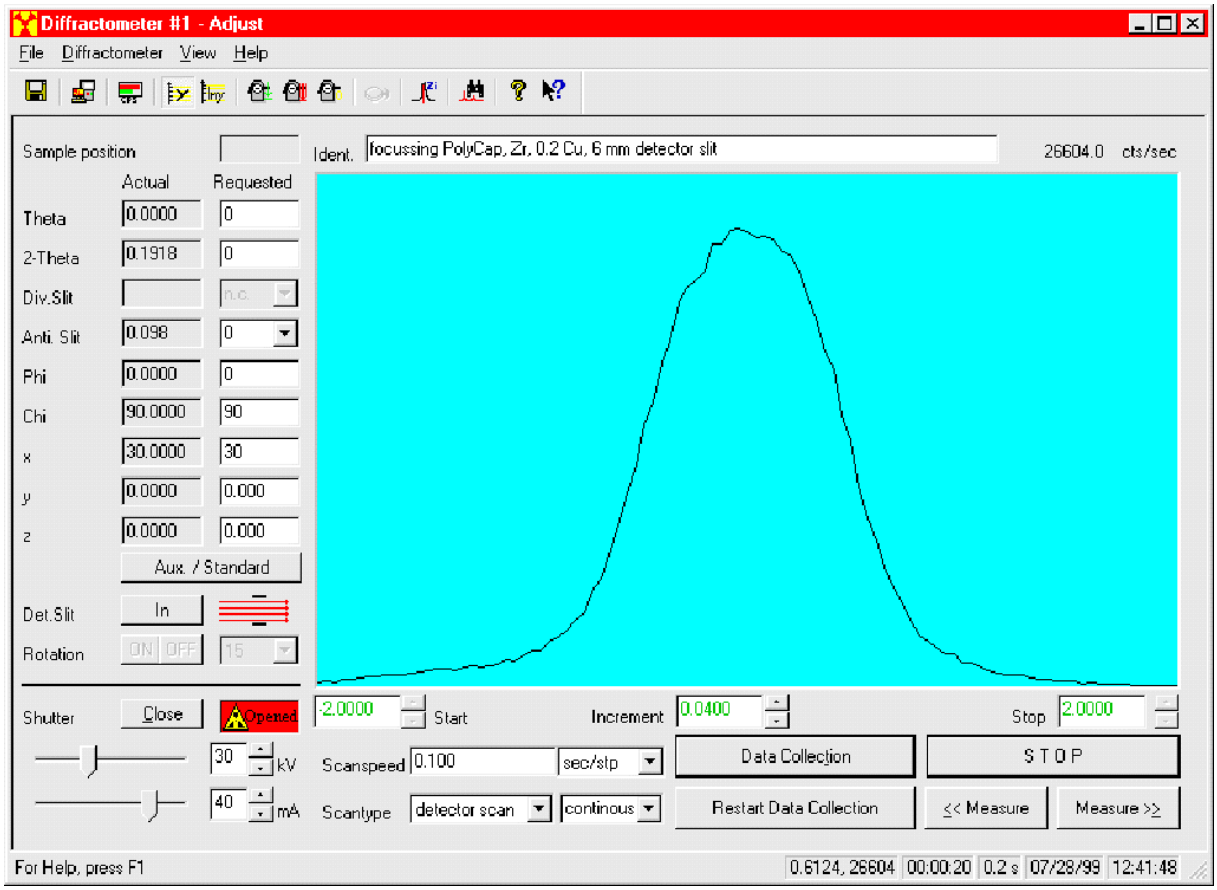


Fig. 3-24: Primary beam of focusing PolyCap with Mo tube, Zr filter, 0.2 mm Cu absorber, 6 mm detector slit, at 30 KV and 40mA. The maximum intensity is lower than that gained with the Cu tube.

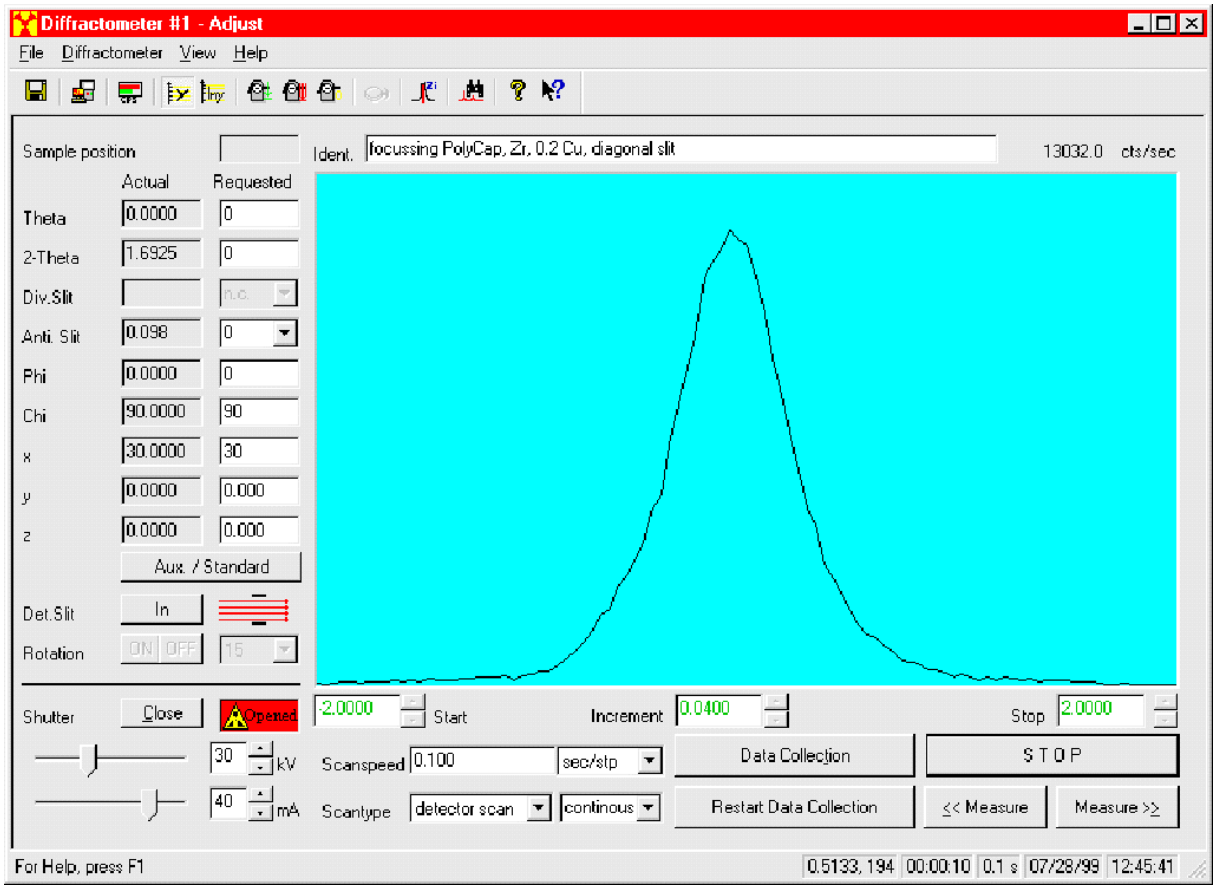


Fig. 3-25: Primary beam of focusing PolyCap and Mo tube measured with diagonal slit: Check of beam slope.

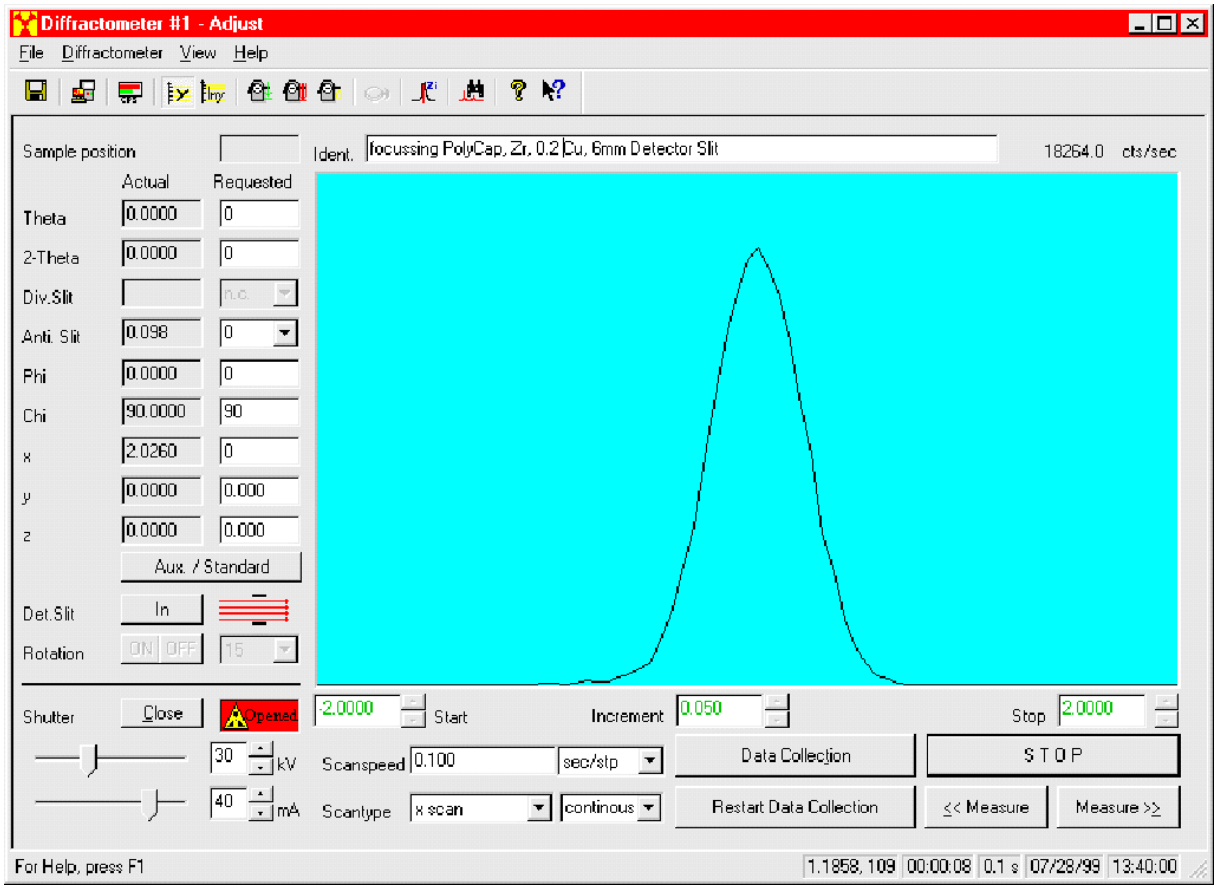


Fig. 3-26: Horizontal beam position for focusing PolyCap (Mo radiation) – X-Scan with pinhole in scattering plane.

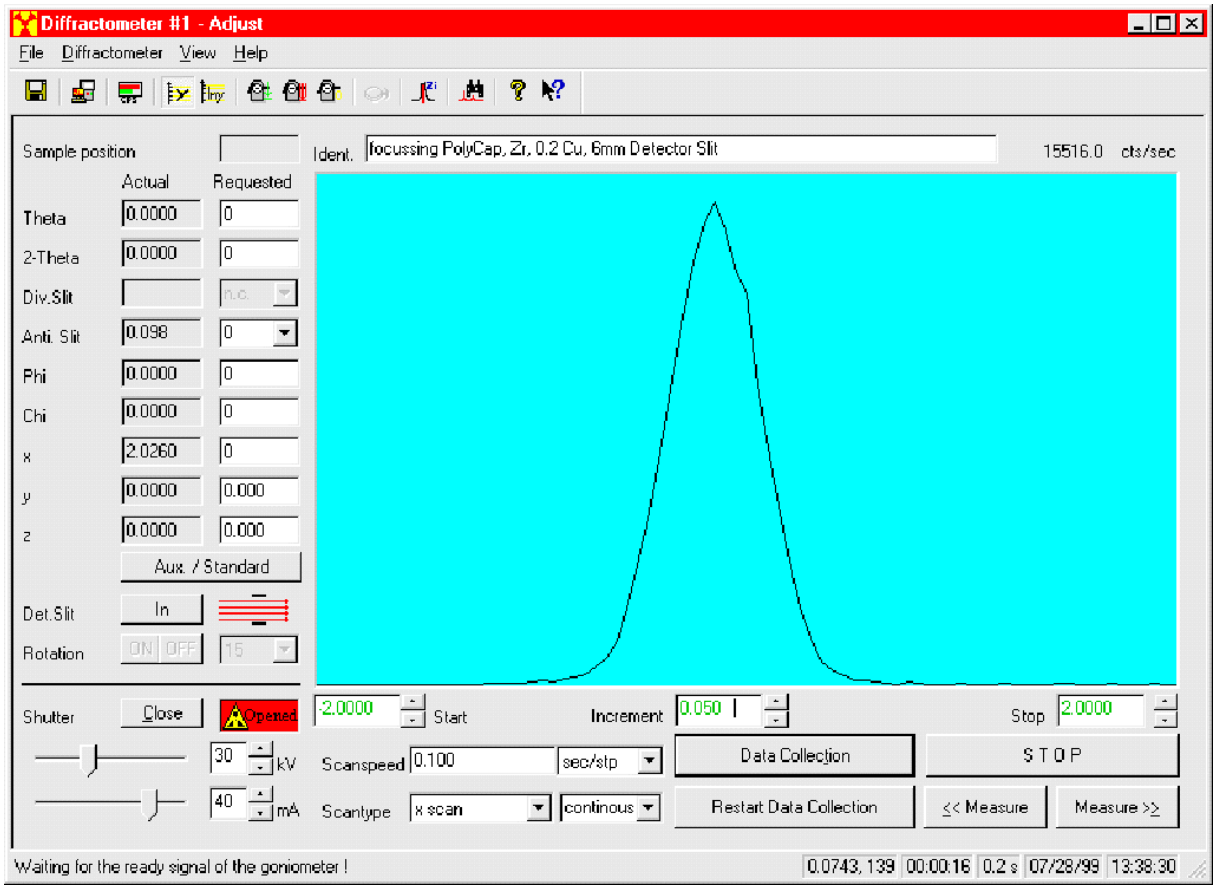


Fig. 3-27: Vertical beam position for focusing PolyCap (Mo radiation) – X-Scan with pinhole normal to scattering plane.

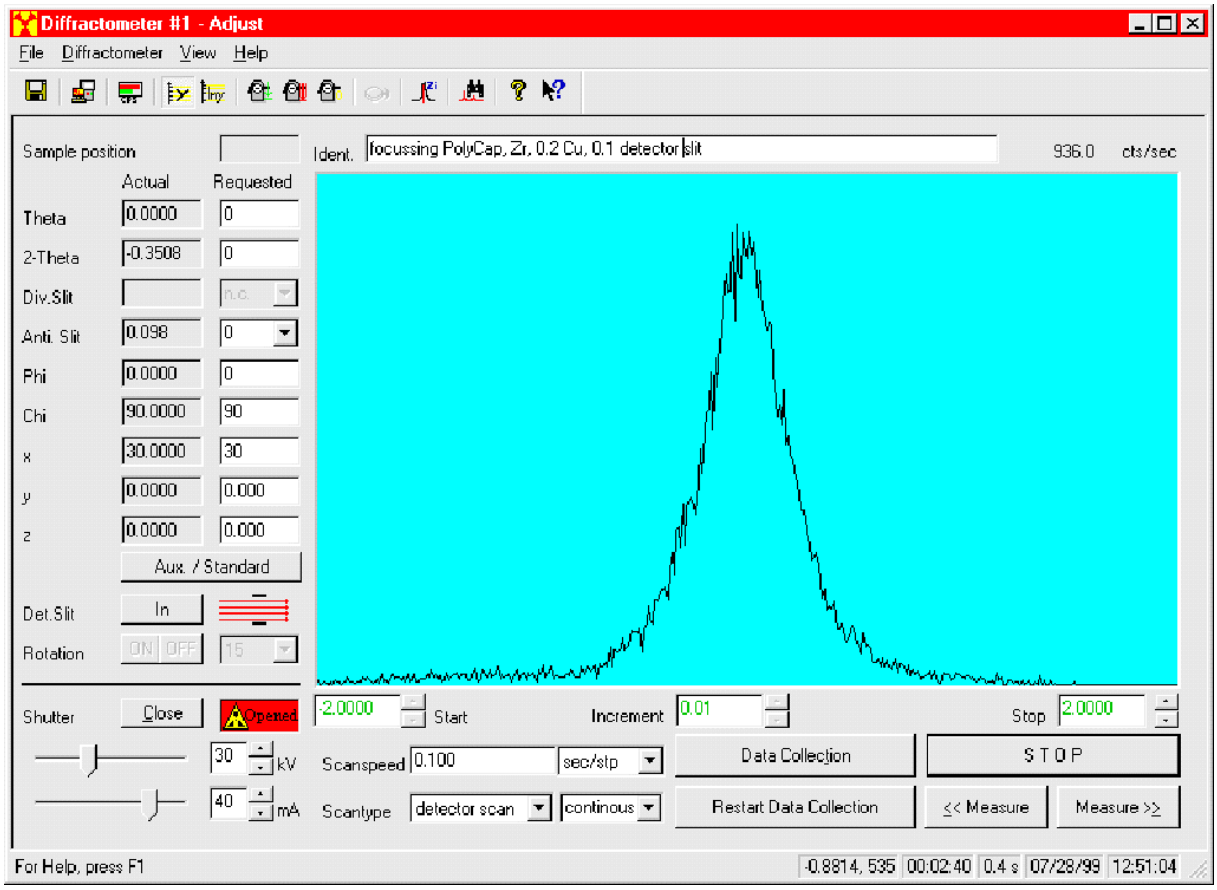


Fig. 3-28: Detector scan through primary beam for focusing PolyCap (Mo radiation): Zero Position of 2theta.

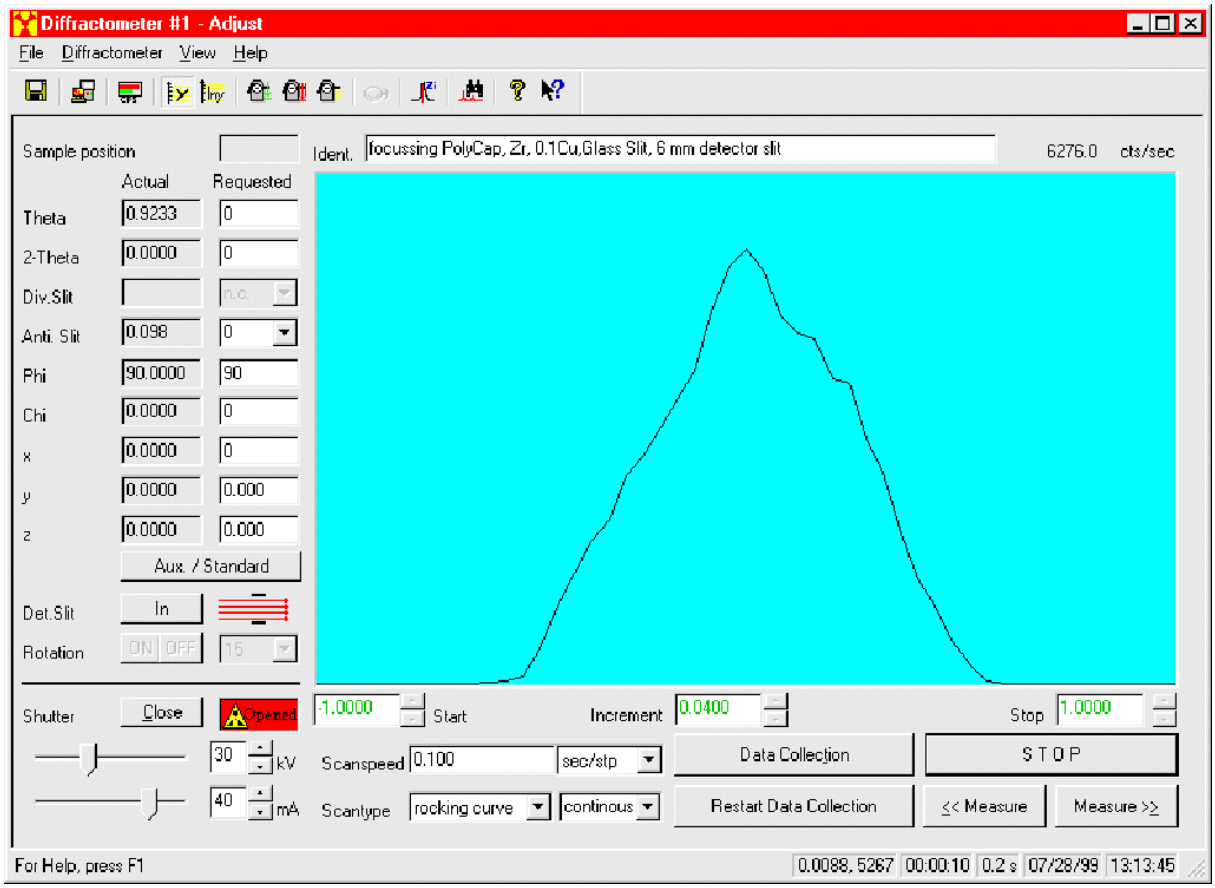


Fig. 3-29: Rocking curve with glass slit for focusing PolyCap (Mo radiation): Zero position of theta. The peak is broader than measured with parallel PolyCap because of the focusing, i.e. converging beam.

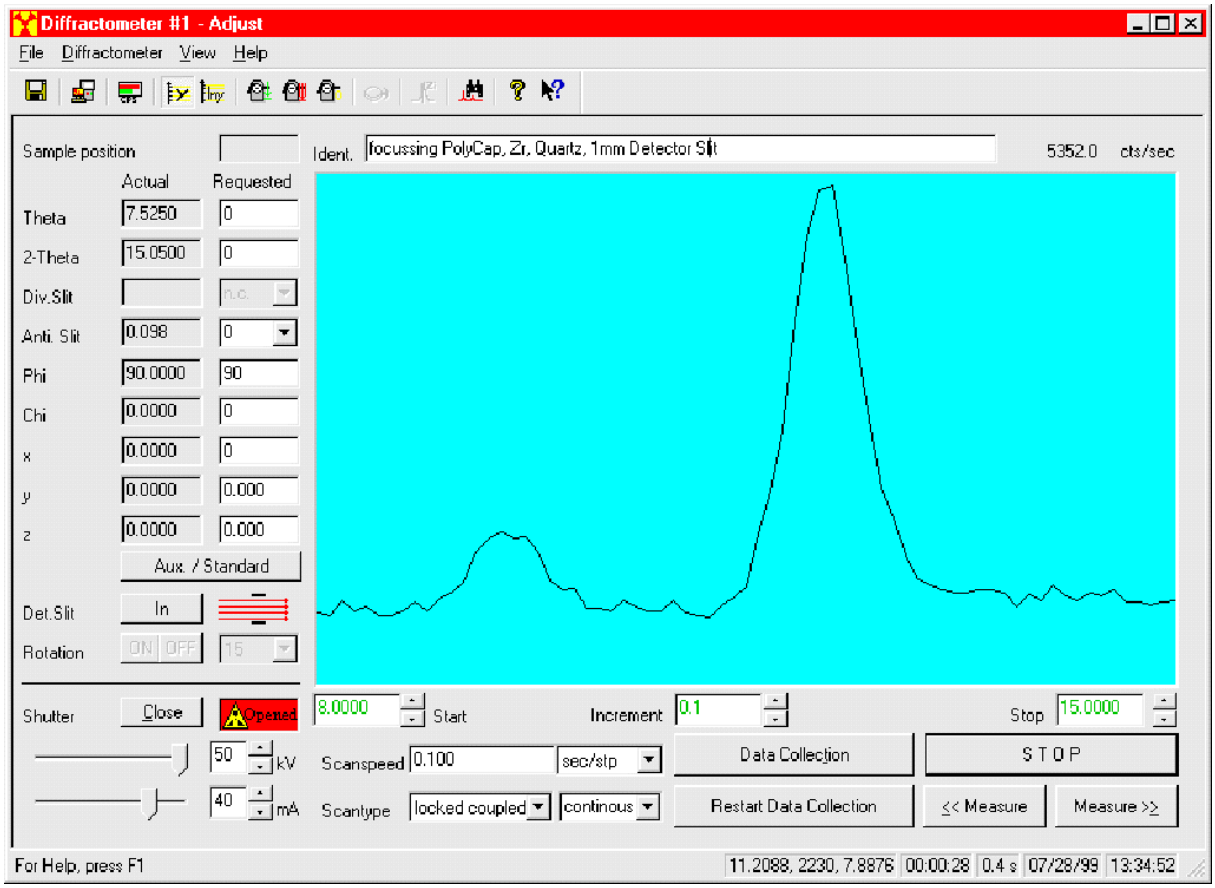


Fig. 3-30: 100% reflection of quartz measured with 1 mm detector slit (Mo radiation): Check of alignment. The intensity is lower than with Cu radiation

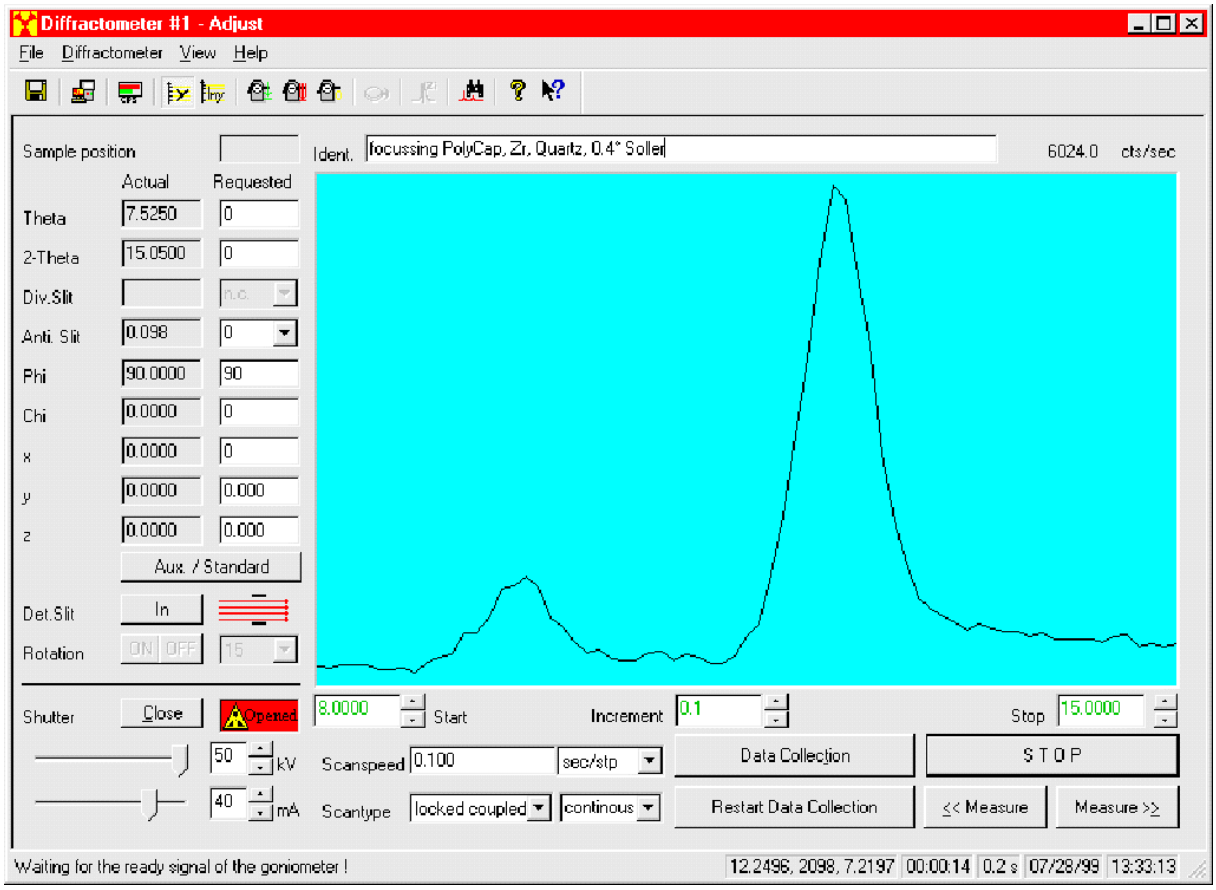


Fig. 3-31: 100% Reflection of quartz measured with 0.4° grazing incidence Soller (Mo radiation): There is no big advantage of the grazing incidence Soller for the focusing PolyCap.

Alignment of Parallel PolyCap on vertical theta-theta with standard sample stage

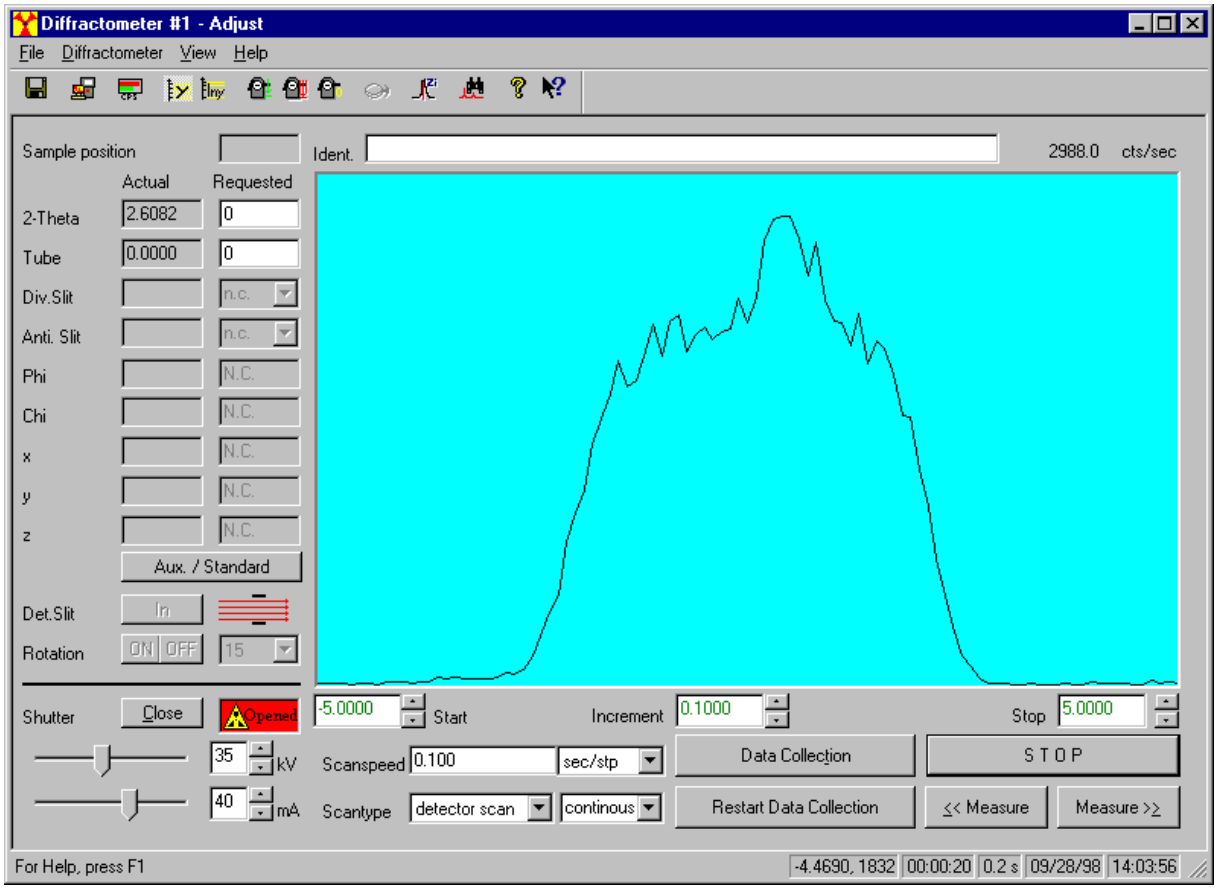


Fig. 3-32: Detector scan for parallel PolyCap. The Kα radiation is the small peak on top of the broad plateau. The centre of mass is not identical for both radiation types! (Cr radiation!)

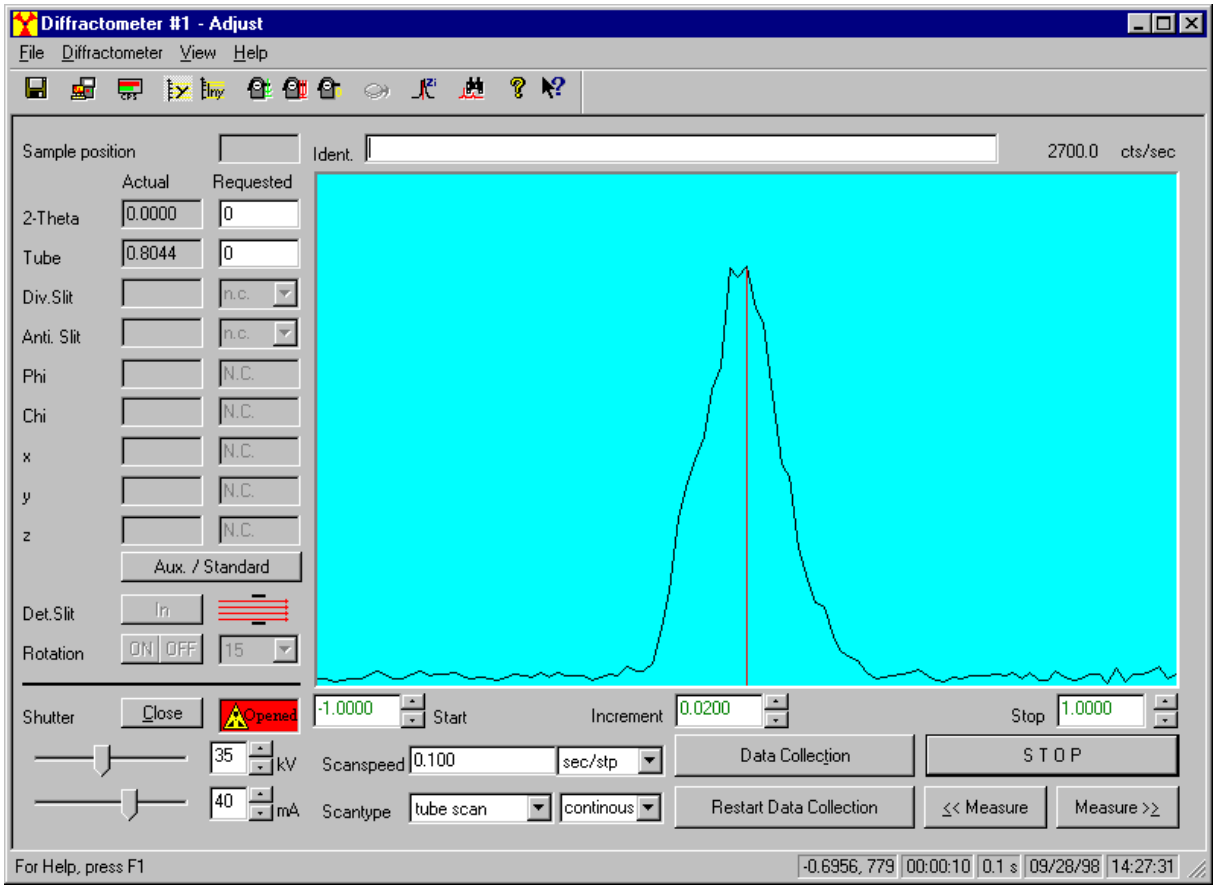


Fig. 3-33: Tube scan with glass slit to check the direction of the primary beam with respect to the goniometer (Cr radiation!).

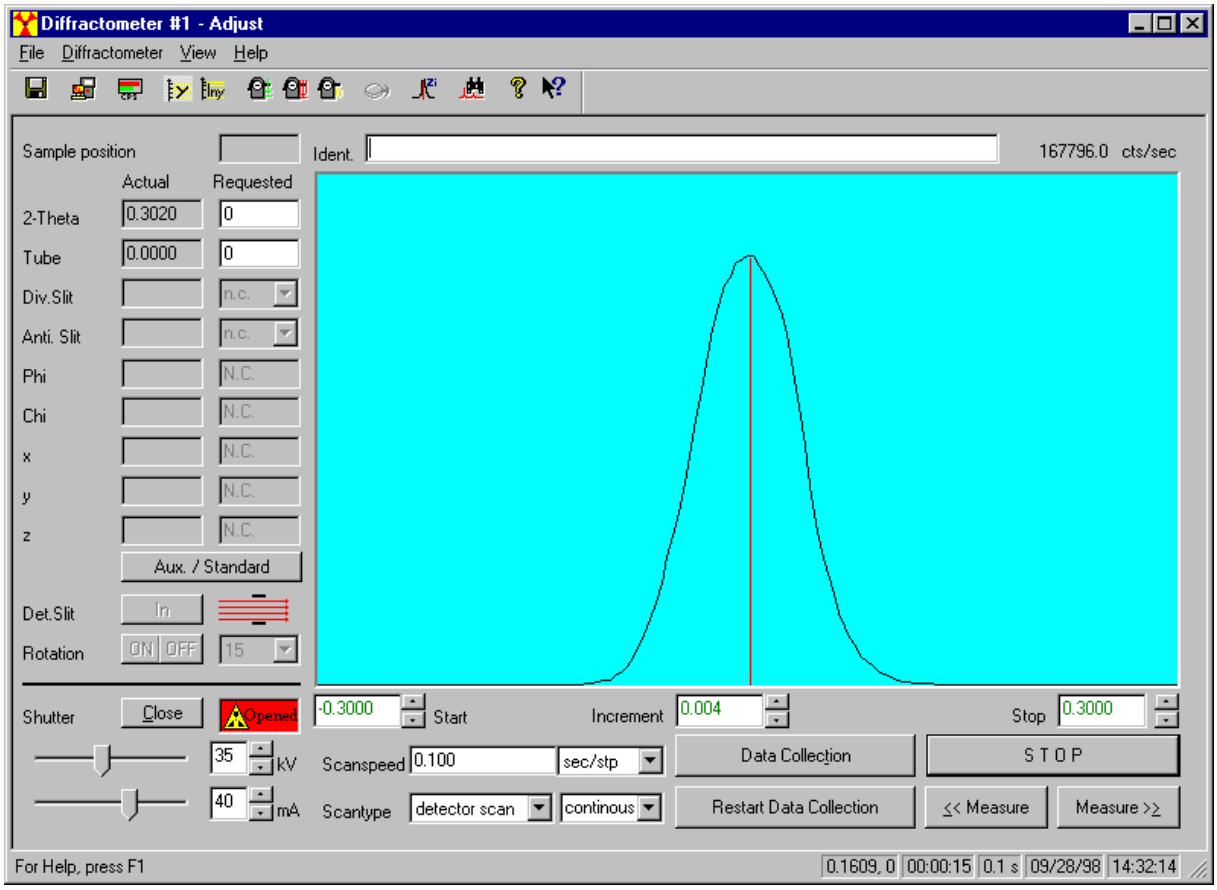


Fig. 3-34: Detector scan through the primary beam with glass slit as sample to check the zero position of the detector circle (Cr radiation!).

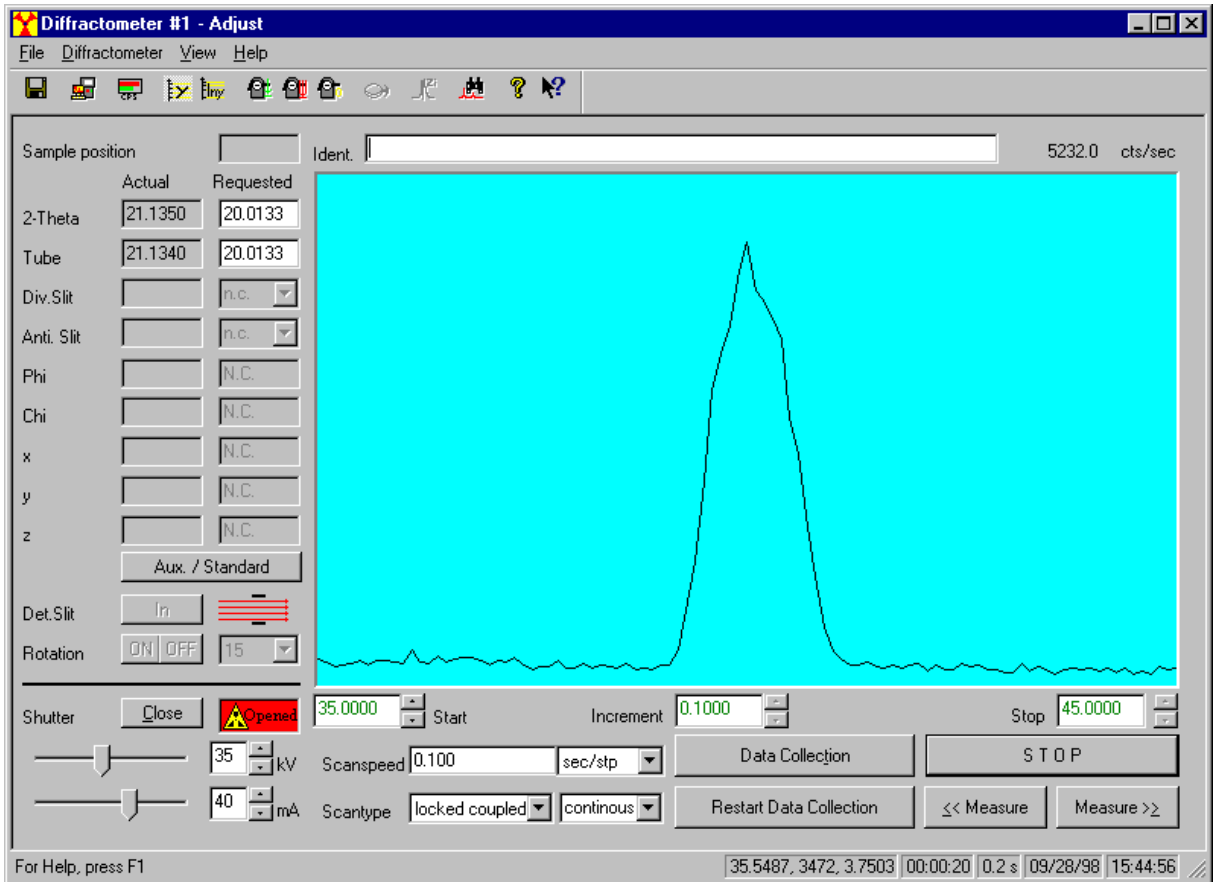


Fig. 3-35: Check of the alignment: 100% reflection of quartz (Cr radiation!)

4 Assembly and Alignment of Single Göbel Mirrors for D8 with Cu Radiation and Line Focus Tubes

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4 Assembly and Alignment of Single Göbel Mirrors for D8 with Cu Radiation and Line Focus Tubes

Introduction

The main component of a Bruker AXS Göbel Mirror is the bent gradient multilayer optics (cf. fig 4-1, 4-2). The standard lengths of the multilayer optics are 40 mm and 60 mm. The bending and lattice spacing is optimized for the D8 sealed tube housing. The Göbel Mirror converts the divergent beam 10 emitted from a line shaped X-ray source 3 into a parallel beam 8.

These special diffractive properties are valid for the Kα radiation of the source, so that the mirror is mainly diffracting the Kα part of the spectrum. Because the Göbel Mirror collects a relevant part of the divergent beam of a source, for many applications a primary beam is conditioned which is more brilliant than a beam created by slit optics.

The multilayer optics is in a radiation safe housing which has three degrees of freedom to align the mirror (fig. 4-1). The position of the mirror's surface (screws 1/2/4), the incident angle (screw 1) and the relative orientation of the mirror's surface with respect to the line focus (screw 2) can be aligned in a proper way to ensure the best performance.

The following sections describe in detail how the Göbel Mirror has to be mounted to the D8 tube housing, and the basic and the final alignments of the Mirror.

Preparation work

1. The D8 has to be set up and aligned as described in the D8 users manual, vol. I.
2. The detector and discriminator have to be set up for Cu-Kα radiation.
3. Make sure that the D8 has a sample stage and a fitting alignment glass slit. In some cases, sample stages are available in which no glass slit fit into - in this case another alignment procedure may be valid.

Removal of the primary slit assembly (fixed or motorized slit assembly)

To remove the primary optics from the dove tail track, loosen both fixing screws at the bottom clamp mechanism so that the optics can be switched above the dove tails.

Mounting the Göbel Mirror

Before mounting the mirror to the tube housing, make sure that the three alignment screws (1, 2 and 4) of the housing are flush (fig. 4-2). This is the mirror's starting position. Then move the incident beam knife edge 6 by turning screw 3 clockwise until the stop position (open knife edge). Do not use too much force for turning the screws!

Mount the mirror to the tube housing by use of four M4 screws shipped with the mirror (cf. fig. 4-3, screws 1a, 1b, 1c, and 1d). Before fastening the screws tightly make sure that the upper edge of the knife edge mounting plate 1 (fig. 4-4) is flush with the mounting plate of the tube housing 2 (fig. 4-4).

Alignment of the Göbel Mirror

The following sections describe the alignment of the mirror, starting with a rough alignment and finishing with the final alignment of the D8 Goniometer with the mounted Göbel Mirror. To execute the alignment DIFFRAC^{plus} ADJUST is necessary.

The steps of alignment are listed below:

- Rough setting of the tube mount
- Rough alignment of the incident angle by use of a fluorescence screen
- Prealignment of the beam direction with respect to the center of the goniometer
- Alignment of tilt and incident angle by use of the counter
- Final alignment of the position of the knife edge and the exit slit
- Final alignment of the beam direction with respect to the center of the goniometer
- Performance test: measuring the standard Quartz sample

Prealignment of the incident angle

The presetting of the alignment screws (fig. 4-2) and the incident beam knife edge was described in the previous sections. Within this section the prealignment of the incident angle of the mirror by means of a fluorescence screen is described. Please use the following settings:

Configuration	
Generator	40 kV, 40 mA
Exit slit of the mirror	open
Sample	mounted fluorescence screen
Anti-scatter slit	open
Kβ-filter, Kα-absorber	none
Receiving slit	0.1 mm
ϑ or tube	45°
2ϑ	0°

In a first step the tube mount and, therefore, the direction of the beam emitted by the X-ray source has to be roughly aligned with respect to the center of the goniometer. In general, the D8 tube mount to be used with a Göbel Mirror should have at least two degrees of freedom of alignment. The first one (fig. 4-5,1) is a translation to align the primary beam with respect to the edge of the incident beam track. The second one is a rotation (fig. 4-5, 2) with a pivot point roughly below the center of the Göbel Mirror. Fig. 4-5 shows the two degrees of freedom tube mount, measuring height 150 mm, in a version released in Mai 1999. Other tube mounts may look different but support the same or even more degrees of freedom.

If you intend to change the diameter of the measuring circle (435 mm, 500 mm, 600 mm) with a minimum of alignment work, you should align the Göbel mirror beam parallel to the primary track to the center of the goniometer. This task is described later in this manual. If you use only one diameter of the measuring circle (e.g. 435 mm), it is not necessary to align the beam parallel to the track, but you have to bring the beam to the goniometer center.

The rotation degree of freedom 1 (fig. 4-5) aligns the Göbel mirror beam to the center of the goniometer. Mount the fluorescence screen 1 (fig. 4-6) e.g. into the sample holder cup 2 (used with rotation sample stage) and move ϑ or the tube (for a theta/theta instrument) to the above given angular position. Close the enclosure doors and open the shutter with a software command. Observe the fluorescence screen and turn screw 2 (fig. 4-5) step by step (¼ of a turn) clockwise and watch the fluorescence screen again with open shutter.

The enclosure doors should be opened smoothly and slowly to make sure that the shutter closes before the doors are open, otherwise the safety circuit may switch off the generator. Continue turning 2 (fig. 4-5) until the illuminated area is seen in the right section of the screen (fig. 4-6, 3). Now the

rough alignment of the beam direction is done.

In the next step tune the setting of the incident angle to the mirror with screw 1 (fig. 4-2). Turn screw 1 approx. 1/8 turn clockwise (fig. 4-11) and observe the illuminated area on the screen. In case the incident angle is above 0° and below the Bragg angle of Kß and Kα radiation a total external reflection of the mirror surface can probably be observed (fig. 4-7). In general, this beam overlaps with the direct beam passing the mirror's surface. Therefore, this beam can easily be distinguished from the first order multilayer Bragg reflection, which is the alignment goal.

Continue turning screw 1 (fig. 4-2, fig. 4-11) clockwise in steps of an 1/8 of a turn. As long as the total external reflection is visible it will successively move in the direction of the arrow (fig. 4-7) with each turn. After about 2 ¼ turns - the total external reflection has already disappeared – a new illuminated line appears on the screen. This is the first order Kß reflection of the mirror.

The intensity of the Kß beam is less than that of the Kα mirror reflection. Both reflections cannot be clearly distinguished by means of the fluorescence screen only. This has to be done in a later step with the use of a Ni filter (Cu Kß suppression) and the counter.

Continue turning screw 1 (fig. 4-2, fig. 4-11) less than an 1/8 of a turn. Within about a ¼ of a turn the Kß line should go down in intensity and another line – the Kα reflection 1 (fig. 4-8) of the mirror - appears. At this point use screw 3 (fig. 4-2) to raise the knife edge (fig. 4-12) and suppress the direct beam passing the mirror (fig. 4-8, 4-9, and 4-10). A narrow direct beam should remain visible (fig. 4-9).

Use screw 1 (fig. 4-2) again to optimize the incident angle as far as the fluorescence screen is sensitive. Now use screw 2 (fig. 4-5) to move the beam 1 (fig. 4-9) by means of the fluorescent screen to the goniometer centre.

Finding the mirror's optimum angle

In this section the goal is to find the maximum intensity and optimum Bragg angle of the reflected Kα beam of the mirror. For the next step of alignment use the following settings:

Configuration:	
Generator	20 kV, 5 mA
Exit slit of mirror housing	open
Sample	none
Anti-scatter slit	6 mm
Kβ-filter, Kα-absorber	Ni-filter, 1 Cu-absorber
Receiving slit	0.1 mm
ϑ	90° for rotating sample stage, 0° for standard sample stage
2ϑ	-2° to 5°

First, you have to measure the reflection and direct beam with a detector scan using ADJUST. Use -2° for the start, 5° for the stop angle and 0.02° as increment. After this first scan, you should observe the mirror's reflection and the direct beam. If not, decrease the start and/or increase the stop angle.

If the reflection and the direct beam appear, set the increment to 0.01°. Move the detector to the reflection angle and exchange the receiving slit to a 2 mm one. Start the ratemeter, open the shutter and observe the integrated beam intensity during the optimization of the mirror's settings. Now turn screw 1 (fig. 4-2, fig. 4-11) in small amounts (1/32 turns or less), close the enclosure door and control intensity. Set screw 1 to the maximum intensity.

Now measure the mirror's reflection (detector scan with 0.1 mm receiving slit) with and without the Ni-filter. Comparing both measurement results you can clearly decide whether the peak you found is Kα. The Cu Kβ peak is reduced by two orders of magnitude of intensity by the filter, whereas Cu Kα is reduced by a factor of about 2 (cf. fig. 4-13).

Aligning the mirror's optimum side tilt angle

If you are sure that the mirror's angle is set to the characteristic Cu-Kα radiation, align the mirror's optimum side tilt angle with screw 2 (fig. 4-2, fig. 4-14) in this section. Leave the Ni-filter in place to be sure that you do not leave the Kα Bragg condition. Use the 2ϑ scan parameters from the last section and find the maximum intensity as follows:

1. Record the angular position of the mirror's reflection with a detector scan. This is the starting position B of screw 2 (fig. 4-2).
2. Set the detector to the reflection angular position and change the receiving slit to a 2 mm one. Open shutter, start the ratemeter and measure the integrated intensity.
3. Turn screw 2 (fig. 4-2) counterclockwise ¼ turn (position A of screw 2) and control the integrated intensity.
4. Again, find the optimum incident angle, by adjusting screw 1 (1/32 turn or less clockwise or counterclockwise) to maximum intensity. If the maximum is less than in position B go back to the starting position B of screw 2 and turn screw 2 clockwise ¼ turn (position C of screw 2).
5. Decide whether + ¼ (pos. A), 0 (pos. B) or – ¼ (pos. C) turn of screw 2 results in the maximum intensity after optimizing screw 1 (fig. 4-2) to maximum intensity.
6. Set screw 2 to the optimum position (A, B, C) and continue with no. 1)

If you have found the best position of screw 2 repeat the procedure (1-6) for +/- 1/8 turn of screw 2.

Testing the mirror quality

To be sure that the mirror gives the proper gain, test the mirror's quality a follows:

1. Run a detector scan and measure the mirror's reflection. Adjust the zero reference by the ZI adjust button.
2. Repeat the detector scan (with or without Ni-filter) with –2° and 5° for the start and stop angle and an increment of 0.01°.
3. Compare the intensity of the peak you just maximized to the intensity of the plateau. For this step a proper and correct setting of the discriminator and detector is very important. Be sure that the lower and upper discriminator level are set to Cu-Kα radiation.
4. The peak should have at least 1.8 times the intensity of the direct beam plateau, proving that the intensity is higher than the direct beam (cf. fig. 4-15).
5. Record the peaks intensity and position.
6. The width of this reflection should be more than 0.5 mm or 0.15° for 435 mm measuring circle. Take the full width at 10% maximum value! Fig. 4-16 shows a typical Göbel mirror reflection, measured with and without a Ni-filter.

Finding the mirror's optimum angle with high power

In this section the optimum incident angle of the mirror is aligned at high generator power.

Settings	
Generator	40kV/40mA (typical for Cu FL 4 KE tubes, lower for FF tubes)
Mirror housing exit slit	open
Sample	none
Anti-scatter slit	open
Ni-filter, Cu-absorber	yes, 2 x Cu-absorber
Receiving slit	0.1 mm
ϑ	90° for rotating sample stage otherwise 0°
2ϑ	+/- 0.5°

Align the mirror using the following steps:

1. After increasing the generator power, wait for 30 minutes until thermal equilibrium between tube, housing and Göbel mirror is reached.
2. Measure the beam profile with the detector scan from –0.5° to +0.5° and increment of 0.01°.
3. Set the detector to the angular maximum peak position and replace the 0.1 mm receiving slit with a 2 mm one.
4. Start the ratemeter and open shutter.

5. Turn screw 1 1/32 of a turn or less and search for the maximum integrated intensity.
6. After setting the mirror to maximum, repeat the detector scan.
7. A typical reflection profile is shown in fig. 4-17. Don't worry about the two shoulders (without slit) you see in this figure. They are quite normal because of bending tolerances on the edges of the multilayer. These shoulders and similar deviations from the ideal profile may occur in typical profiles and can be cut off with slits.

Align the knife edge window

Now the knife edge has to be closed as far as possible without cutting intensity from the mirror's reflection. Insert the 0.1 mm receiving slit and measure a detector scan from –1° to 5° with an increment of 0.02°. Look where the knife edge cuts off the direct beam (cf. fig. 4-18) and turn screw 3 (fig. 4-2) counterclockwise to close the knife edge 6 (fig. 4-2). Control again by measuring a detector scan. A small amount of the direct beam must remain (cf. fig. 4-18, a/b) in order to avoid cutting the mirror's profile.

Align the mirror's exit slit

In this section the exit slit of the mirror housing will be set to the optimum position with an optimum width. As you see in fig. 4-17, the reflection profile shows irregularities. This is quite normal because of tolerances in the bending of the mirror which are located at the edges of the mirror and irregularities of the tube focus (compare fig. 4-16 and 4-17). In order to find the optimum width and position of the slit follow these steps.

Settings	
Generator	40 kV / 40 mA (typical for Cu FL 4 KE tubes, lower for FF tubes)
Mirror housing exit slit	2 mm
Sample	none
Anti-scatter slit	open
Ni-filter, Cu-absorber	yes, 2 x Cu-absorber
Receiving slit	2 mm
ϑ	90° for rotating sample stage otherwise 0°
2ϑ	+/- 0.5°

1. Set the detector to the angular peak position, replace the 0.1 mm receiving slit by a 2 mm one.
2. Insert a 2 mm slit into the Göbel mirror housing, open shutter and start the ratemeter.
3. Turn screw 2 (fig. 4-3) for ½ turn.

4. Control intensity and maximize with screw 2 (fig. 4-3)
5. Insert 1 mm slit and maximize intensity with screw 2 and ¼ turns
6. Insert 0.6 mm slit and maximize intensity with screw 2 and 1/8 turns or less.

Control the reflection profile by measuring a detector scan. If you see a shoulder at the left or right side of the profile move, screw 2 (fig. 4-3) by a small amount in the direction which minimizes the shoulder. Fig. 4-17 presents a measured profile with optimum alignment of a 0,6 mm slit in contrast to a profile without any slit.

Aligning the reflection parallel to the track through the goniometer centre

If you want to change the diameter of the measuring circle (from 435 mm to 500 mm or to 600 mm and vice versa) with a minimum of alignment work, you should align the Göbel mirror beam parallel to the primary track to the center of the goniometer. This work is described in this section. If you use only one diameter of the measuring circle (e.g. 435 mm), it is not necessary to align the beam parallel to the track, but you have to bring the beam through the centre of the goniometer; use the centralize procedure as described here and omit the parallel-track part of the procedure.

1. In the previous section the rough adjustment of the beam to the goniometer centre parallel to the primary track by means of a fluorescent screen has been described. In the first step the beam has to be set parallel to the track (1 of fig. 4-19) (omit this, if you use one measuring circle diameter!). Slide the tube with optics along the track 3 (fig. 4-19, 1) and measure detector scans in two positions – near the sample and far away from the sample. In case of divergency (beam and track are not parallel) the maximum of the beam profile has to be on a fixed angular position, otherwise the tube has to be inclined by screw 2 (fig. 4-5). In fig. 4-19 part 1 you see the beam path in the case of a divergency α. If the beam is parallel to the track then a=0 mm, i.e. the angular positions of the beam – near the goniometer centre and far away – do not change. It might happen that the beam doesn't hit the goniometer centre, this alignment will be described next.
2. Now the beam must pass the goniometer centre. Move screw 1 (fig. 4-5) for this alignment. Use the fluorescence screen for a rough alignment in order to set the beam to the centre. After that, a detector scan has to be performed, use ADJUST and set the detector to the maximum intensity position. Remove the 0.1 mm receiving slit and insert the 6 mm one.

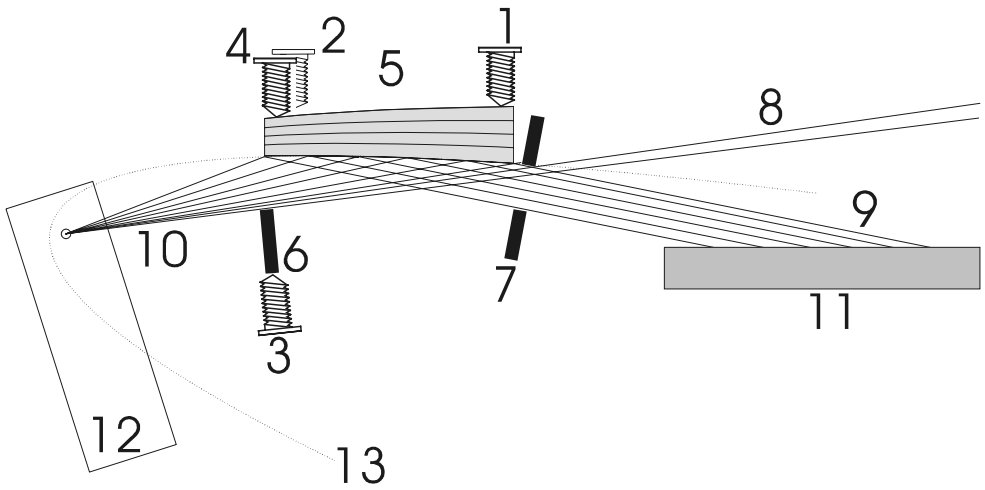
3. Insert the glass slit into the sample holder and proof whether the beam passes the glass slit. The beam now has a width of 0.6 mm (if you use the 0.6 mm slit to minimize the shoulders). Proof by means of a rocking curve scan whether the beam passes the glass slit. In most cases this condition is fulfilled after the rough adjustment because the beam is relatively wide. If not, move screw 1 (fig. 4-4) a little bit and repeat the measurement.
4. Optimize the intensity through the glass slit by screw 1. When you have reached the maximum, repeat the rocking curve scan and control the maximum again. This procedure has to be repeated with slit widths of 0.2 mm and 0.1 mm so that the centre of the beam passes the glass slit. As a last step the ZI determination must be done for the glass slit (setting 'rocking curve').
5. Move the glass slit to position 0°, insert a 0.1 mm receiving slit and determine the ZI value for the detector (setting 'detector scan').
6. If the intensity through the glass slit with 0.1 mm slit width (Göbel mirror slit) is at maximum, the zero point deviations of ϑ and 2ϑ are 0 or less than +/- 0.01 after ZI-determination and divergency is about 0.1° to 0.5° (angle between beam and track) the final alignment is reached.

Examples of typical diffraction scans

Here, some examples of diffraction scans are presented in order to test the final quality of the Göbel mirror beam, i.e. divergency and intensity. Test sample is quartz and test reflections are the main peak and the 5 finger set of quartz. The parameters for these plots are 40 kV and 40 mA, a 0,6 mm exit slit at the mirror's housing, the 4° soller slit, a 0,2 mm anti-scatter slit and a 0,1 mm receiving slit. The profile with the higher intensity is recorded without the soller with same parameters as above.

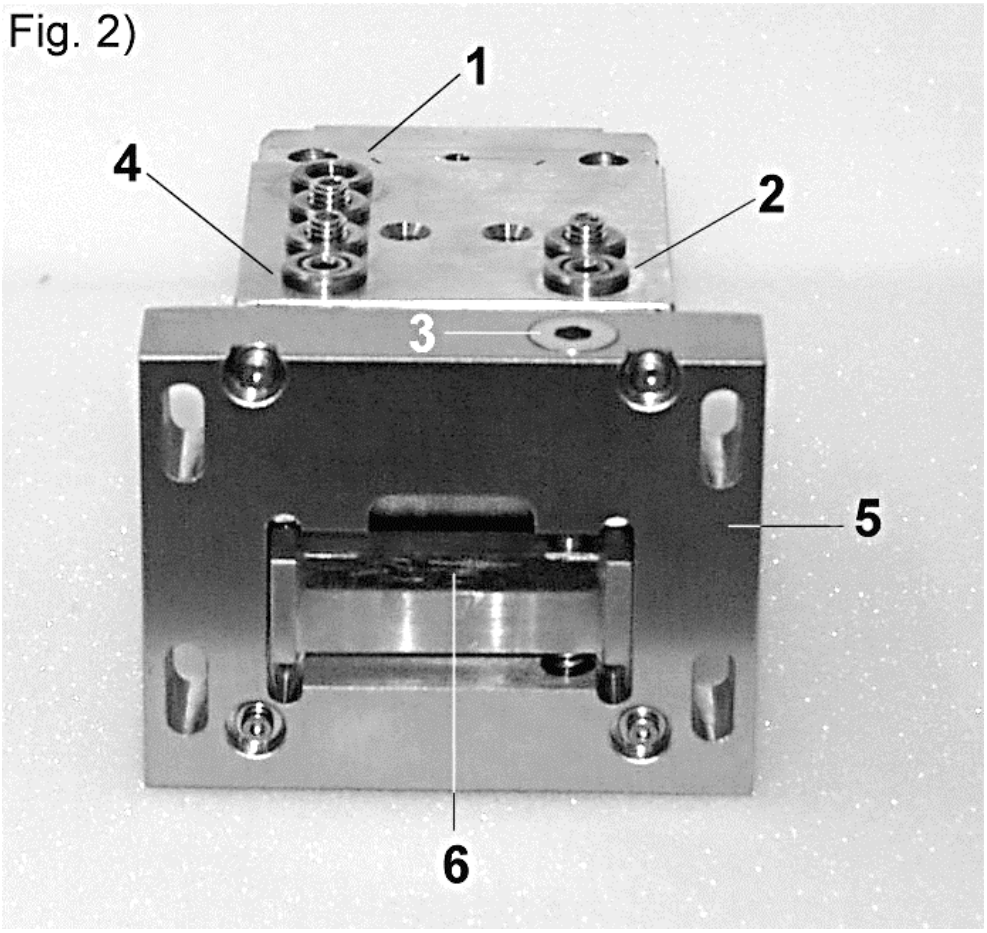
Figures

Fig. 1)



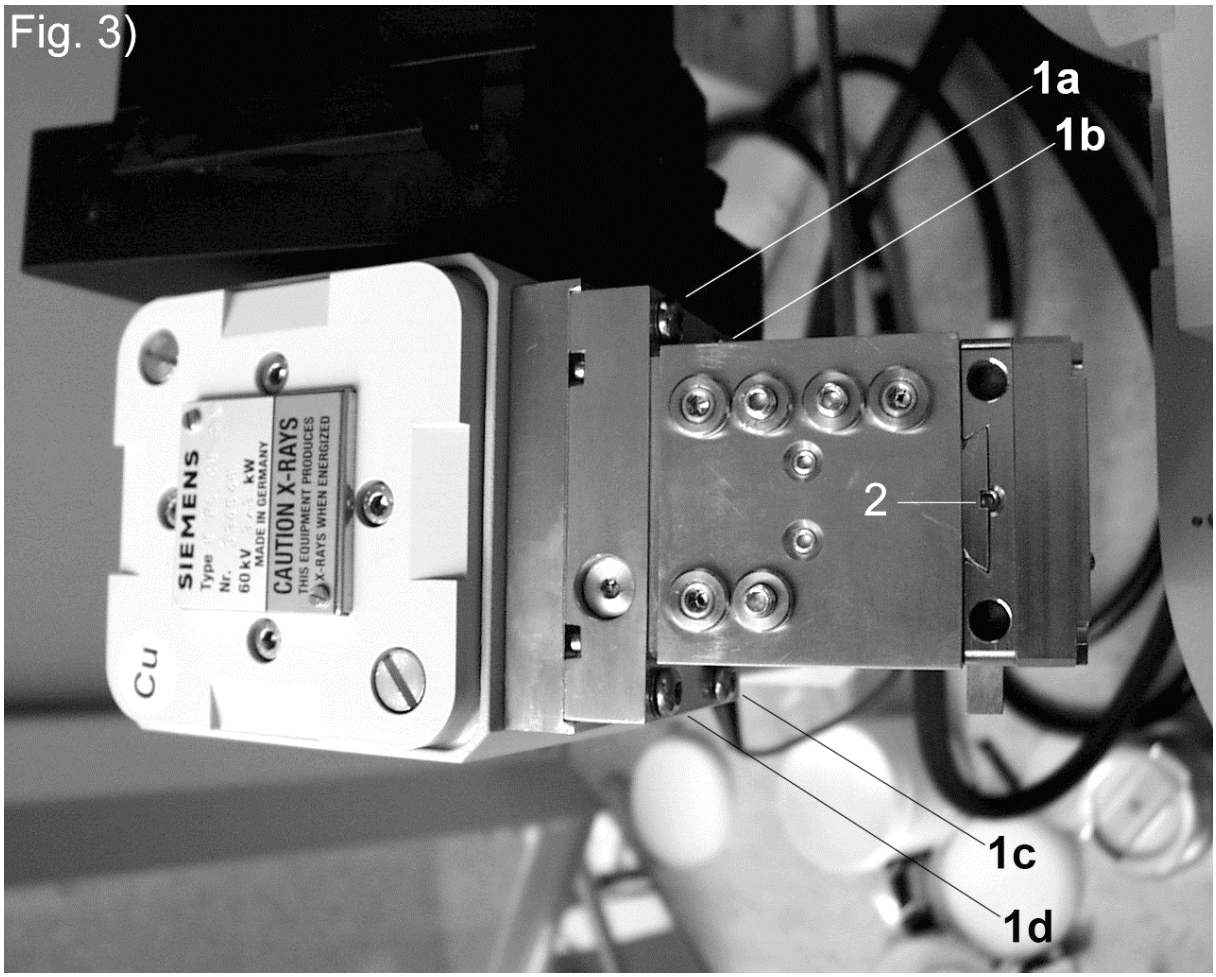
1) The incident angle can be varied with screw 1.
2) By turning screw 2, the mirror's surface can be set parallel to the line focus of the tube.
3) Screw for the knife edge.
4) By turning screws 1/2/4 simultaneously the mirror can be translated to set the take off angle.
5) Bent gradient multilayer optics.
6) Knife edge.
7) Exit slit.
8) Direct beam.
9) Parallel Kα reflection.
10) Divergent beam from tube focus.
11) Sample.
12) X-ray tube.
13) Parabola.

Fig. 4-1: Beam path for Kα radiation at Bragg condition of first order with the Göbel mirror



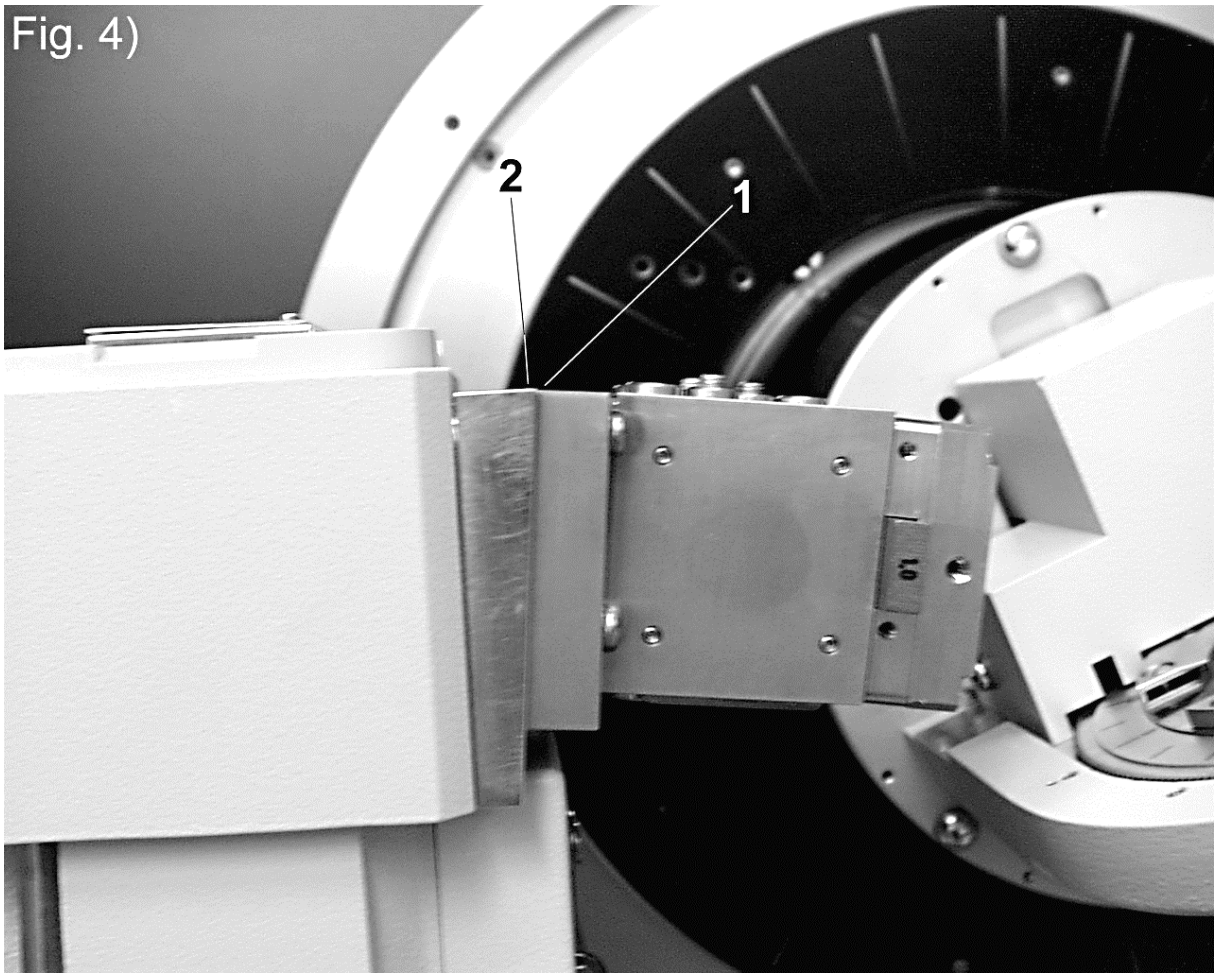
1) Variation af incident angle.
2) Set the mirror's surface parallel to the line focus with this screw.
3) Knife edge setting screw.
4) Simultaneous turning of screws 1/2/4 translates the mirror to change the take off angle.
5) Mounting plate.
6) Knife edge.

Fig. 4-2: Göbel mirror housing with mounting plate and alignment screws



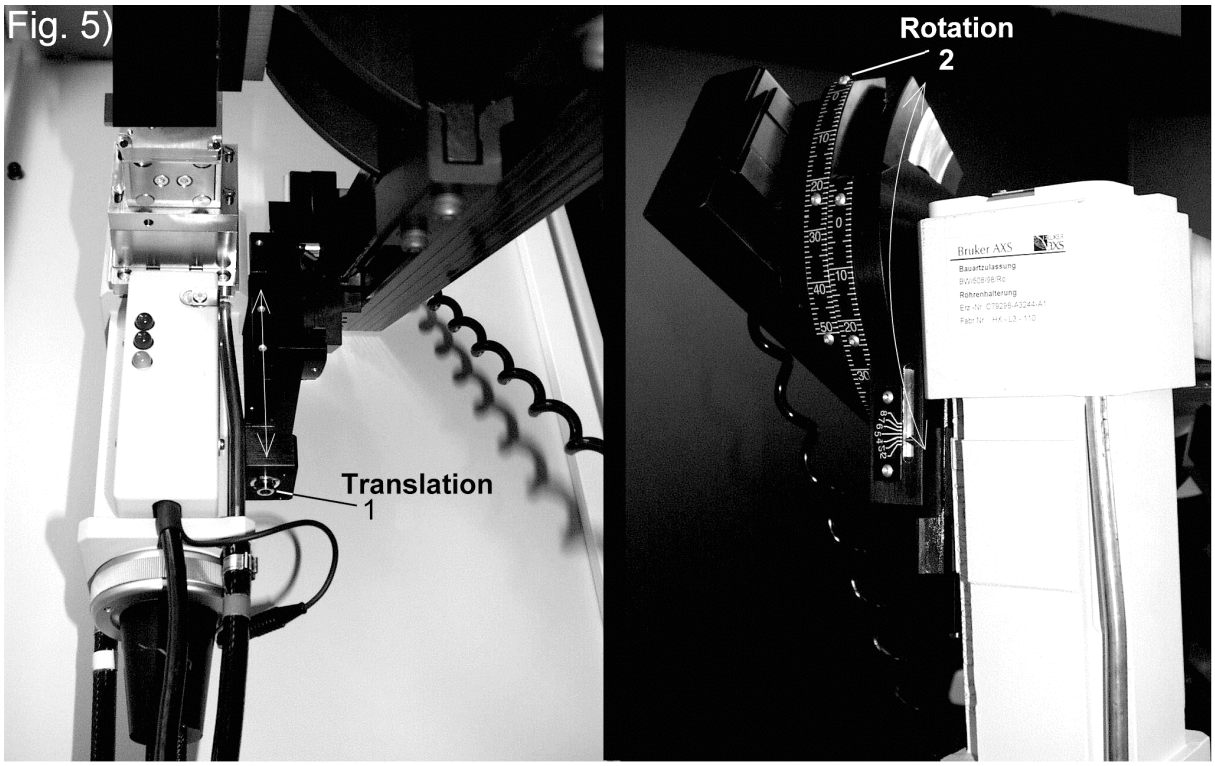
1) a/b/c/d mounting screws M4 each.
2) Alignment screw for the position of the exit slit.

Fig. 4-3: Göbel mirror housing mounted to the tube housing



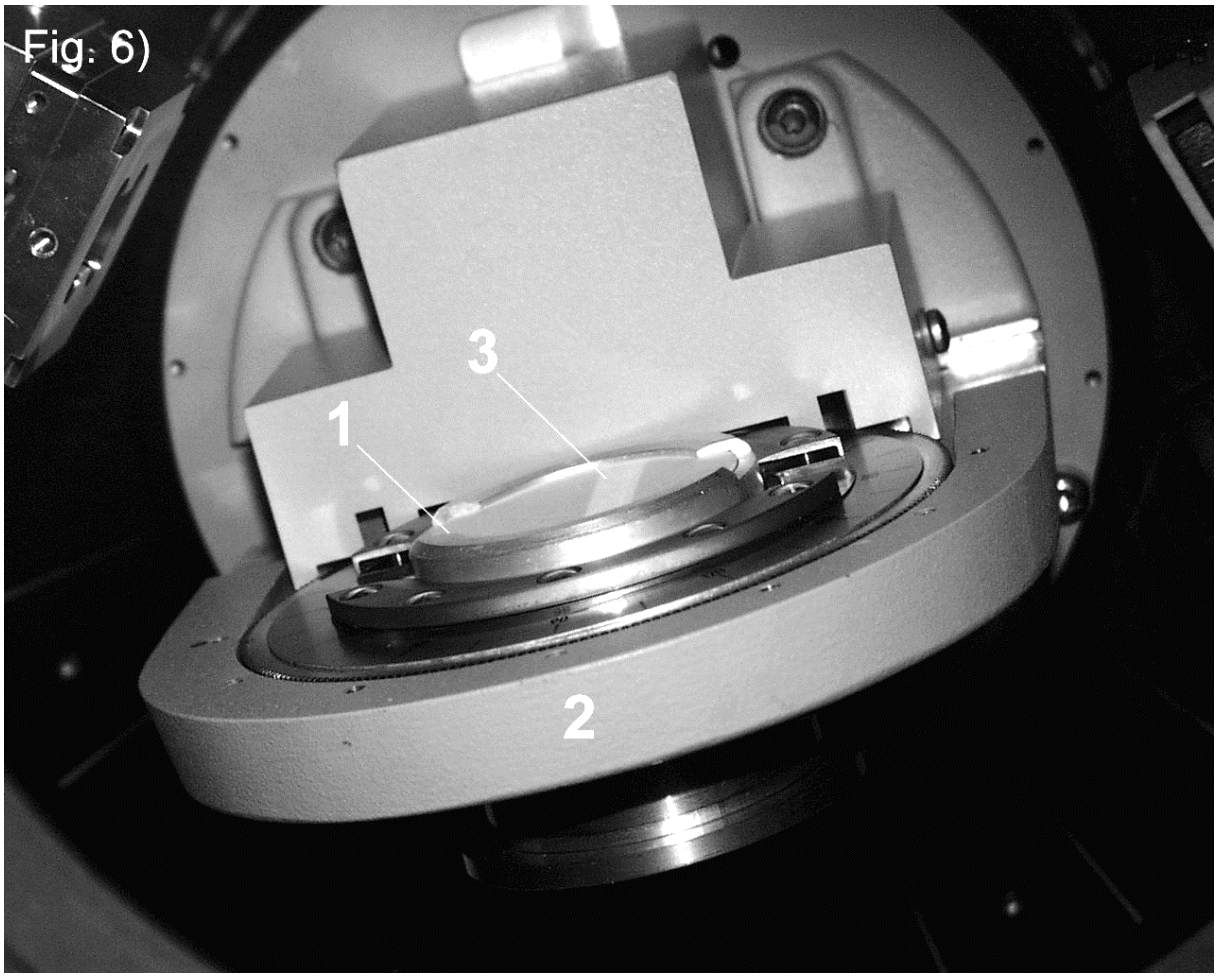
1) Mounting plate edge flush with tube housing mounting plate
2) See above.

Fig. 4-4: Göbel mirror housing mounted to the tube housing



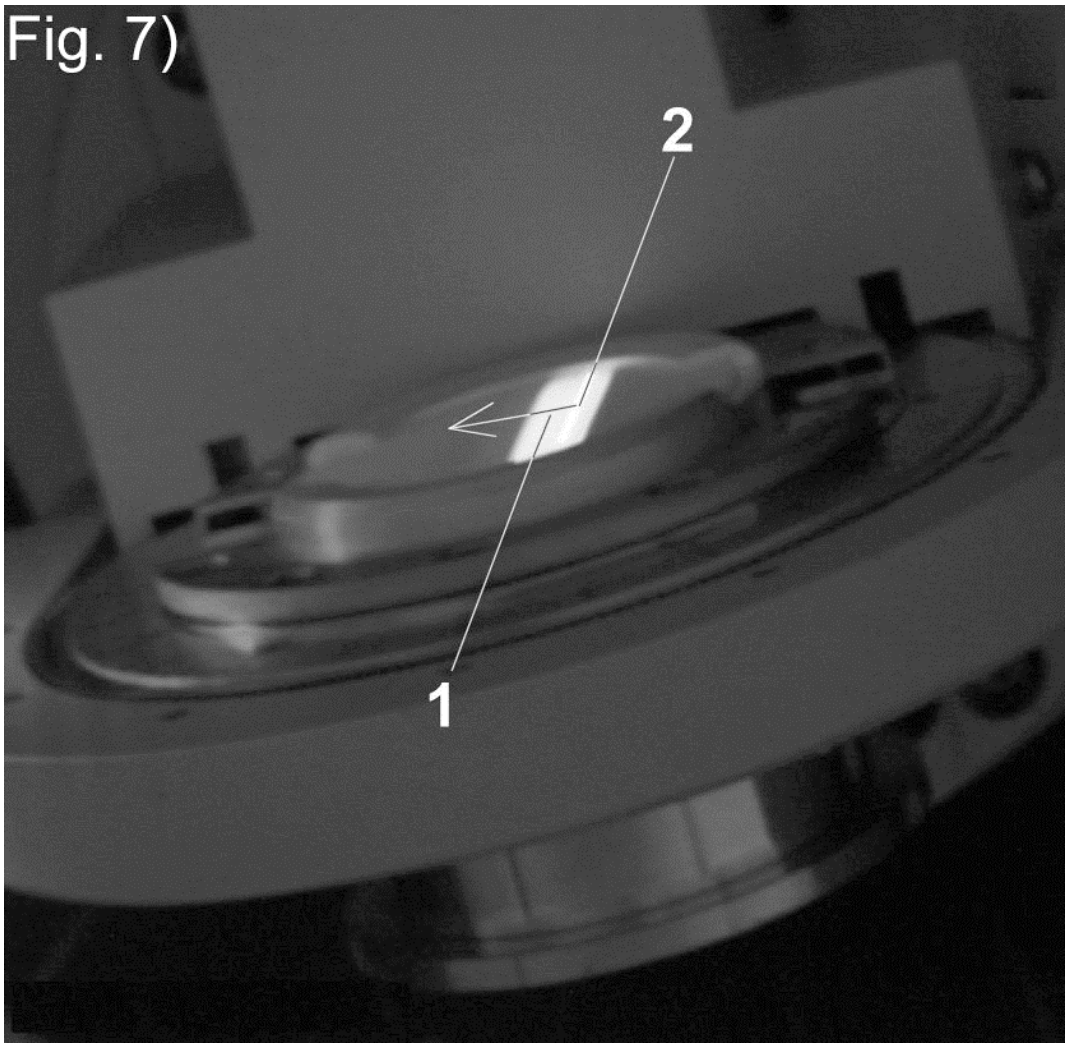
1) Interlock.
2) Rotation.
3) Bottom clamp fixing screws for the tube mount.
4) Translation.

Fig. 4-5: Tube mount with at least two degrees of freedom



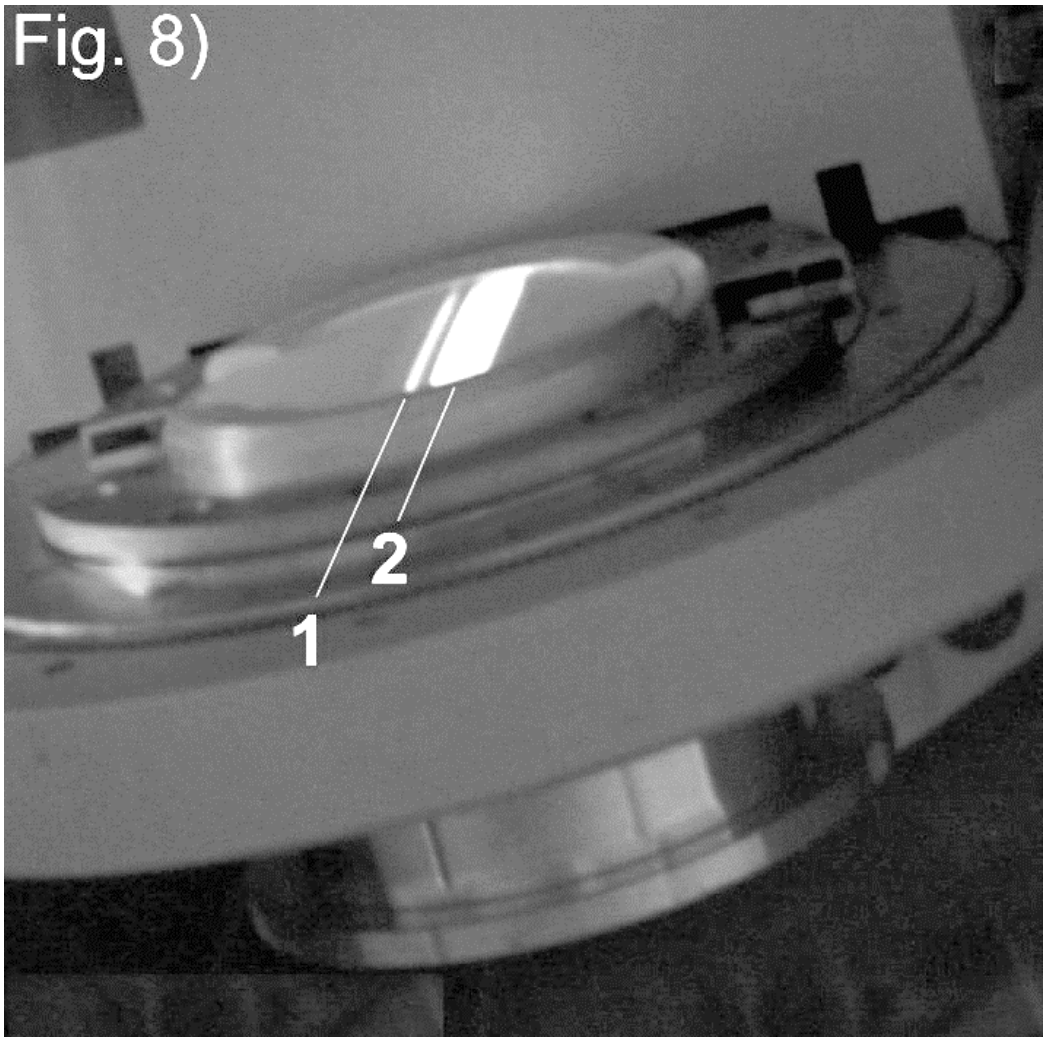
1) Fluorescence screen.
2) Sample holder.
3) Direct beam passing the Göbel mirror housing.

Fig. 4-6: Sample holder with mounted fluorescent screen



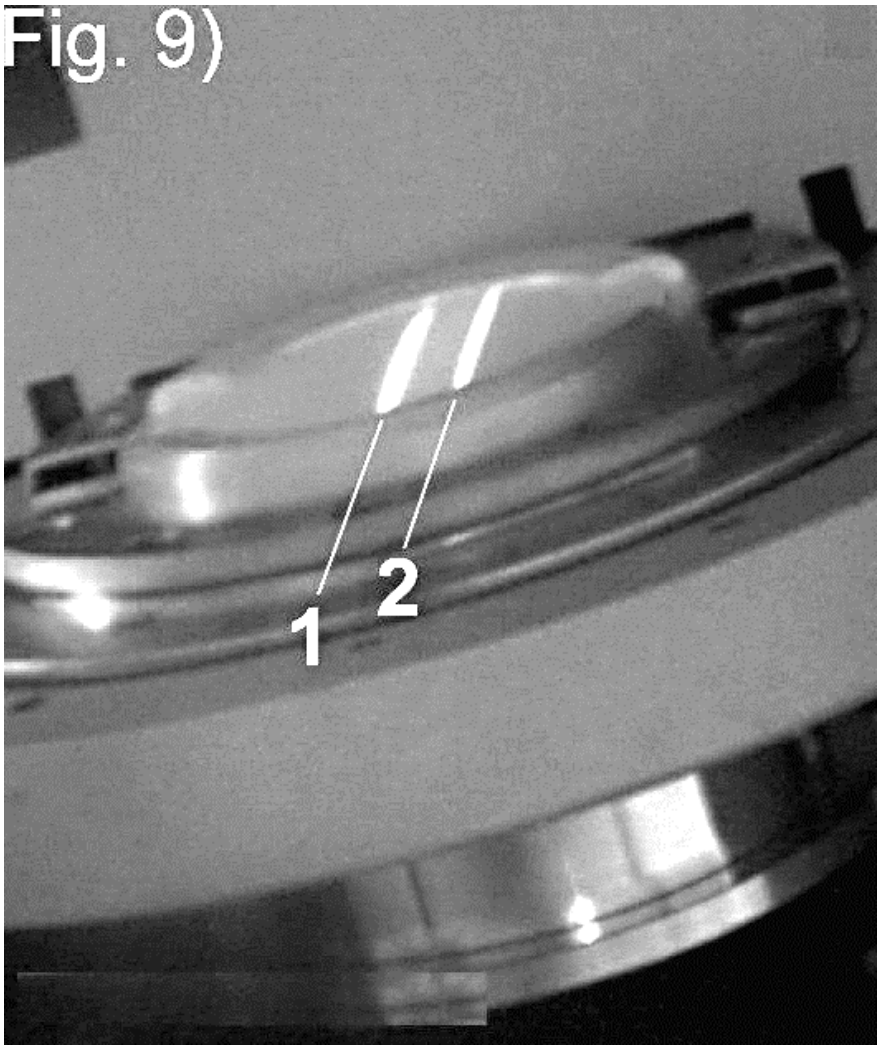
1) Direct beam.
2) Total external reflection with moving direction.

Fig. 4-7: Total external reflection from the mirror's surface



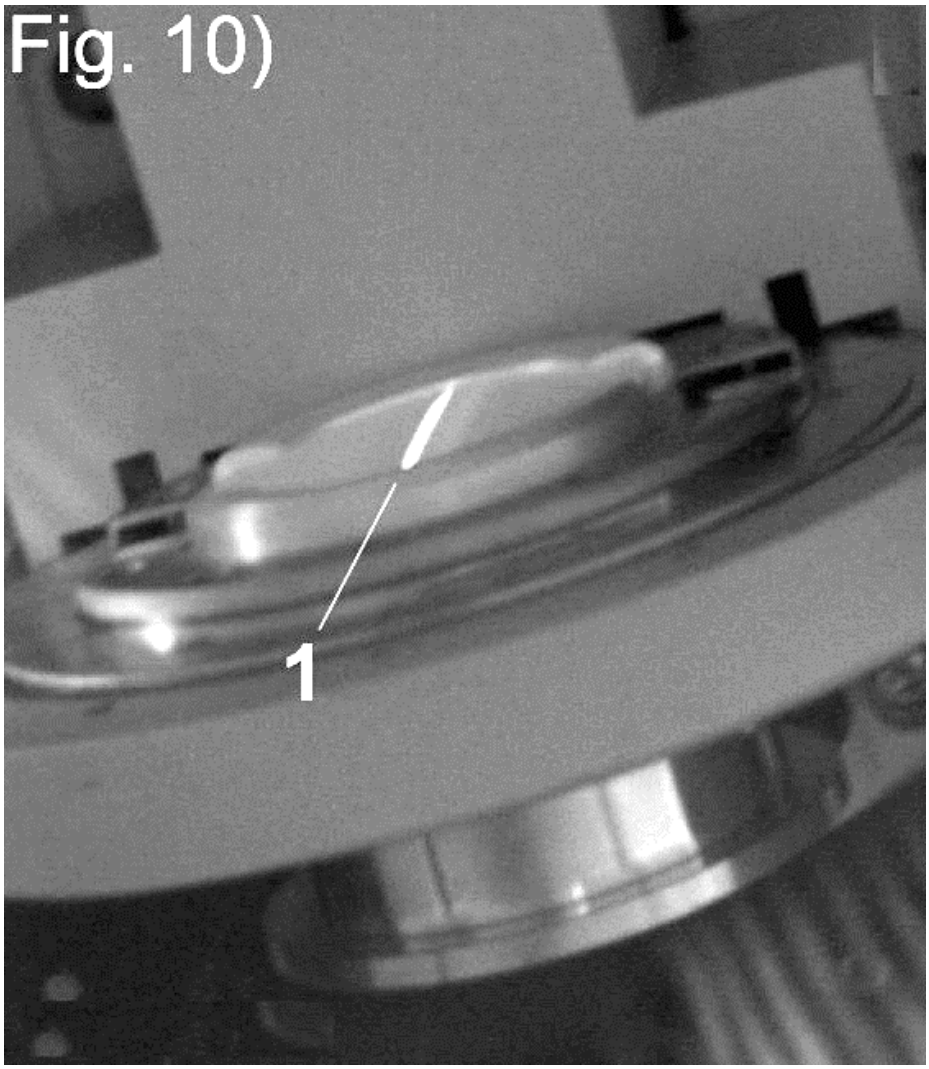
1) Kα reflection of Göbel mirror.
2) Direct beam from X-ray tube.

Fig. 4-8: Direct beam and Göbel mirror reflection



1) Kα reflection of Göbel mirror.
2) Direct beam with nearly closed knife edge.

Fig. 4-9: Direct beam and Göbel mirror with nearly closed knife edge



1) Kα reflection from Göbel mirror. Knife edge is closed completely.

Fig. 4-10: Göbel mirror reflection without direct beam



Fig. 4-11: Turning screw 1 to set the Göbel mirror to the Bragg angle

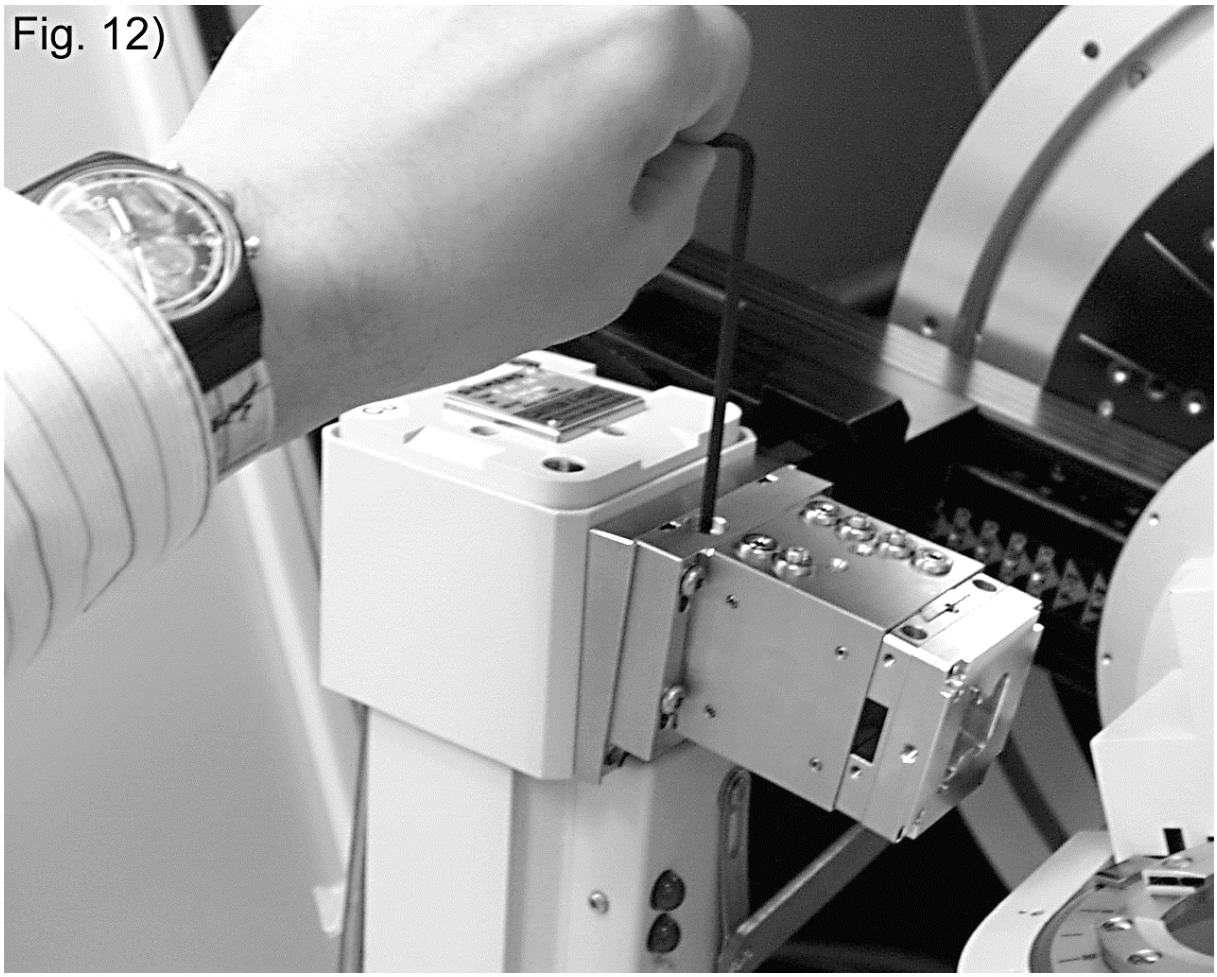


Fig. 4-12: Turning screw 3 to close the knife edge

Fig. 13)

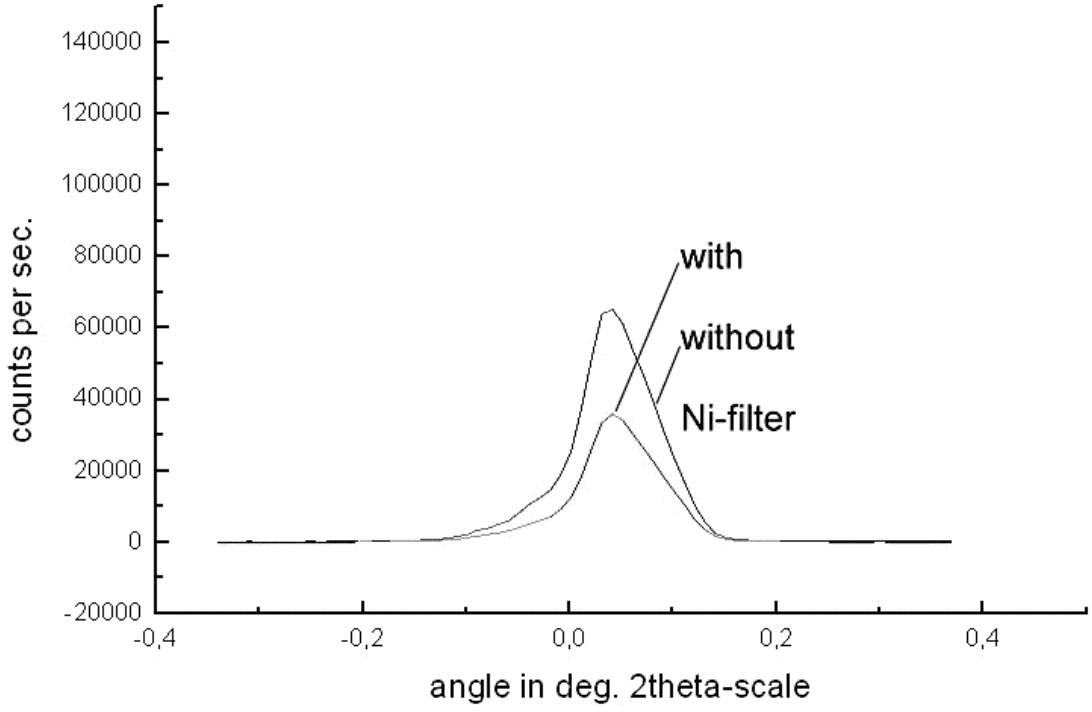


Fig. 4-13: Parallel Kα beam of Göbel mirror with and without Ni-filter

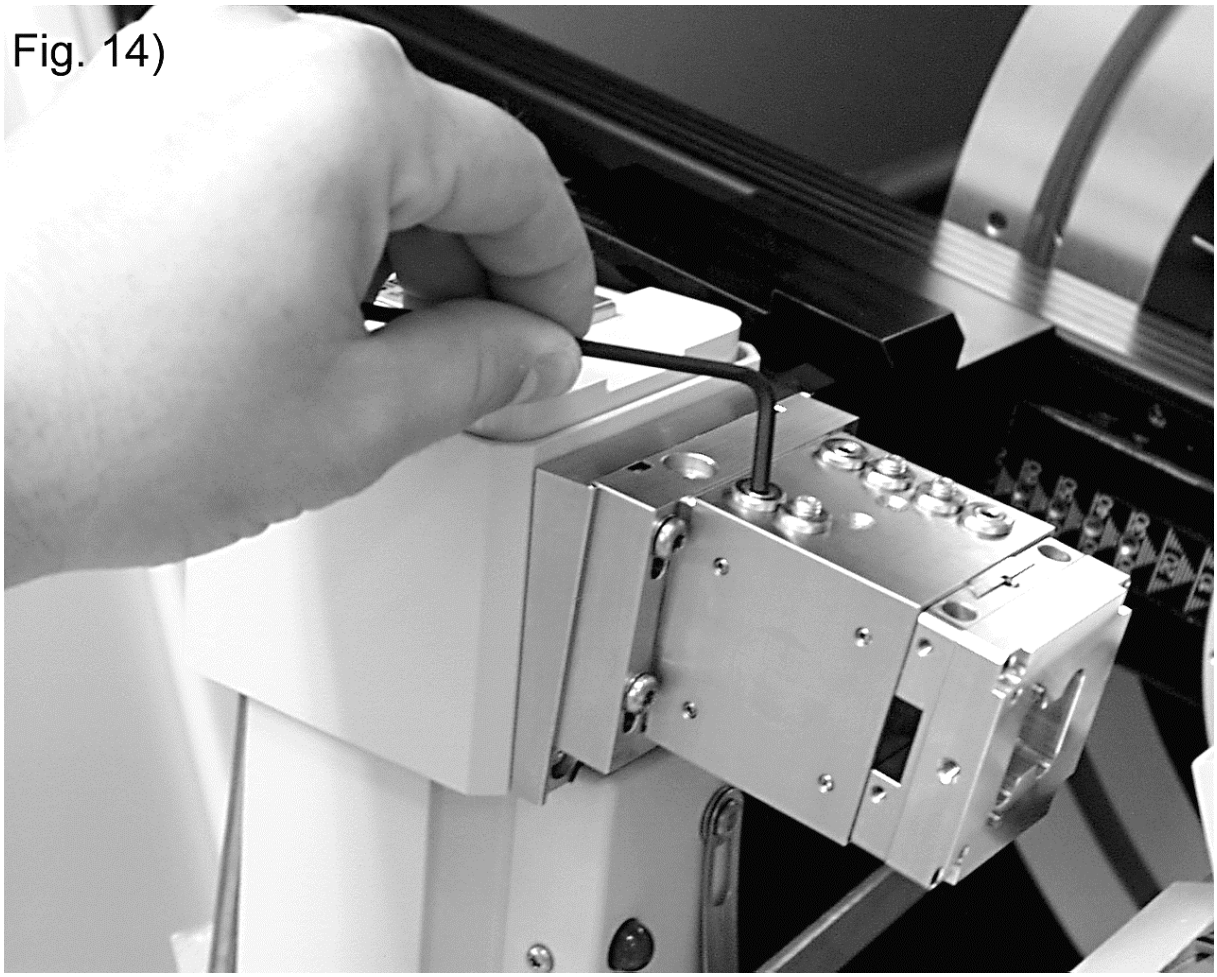


Fig. 4-14: Turning screw 2 to find the optimum side tilt angle

Fig. 15)

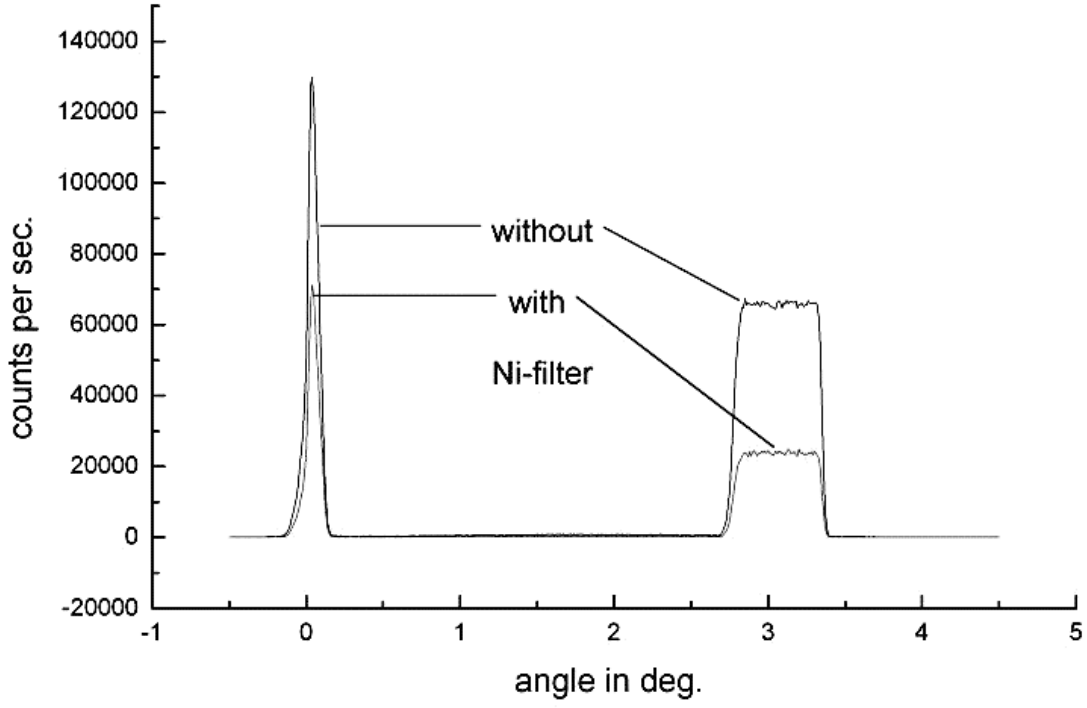


Fig. 4-15: Göbel mirror reflection and direct beam with and without the Ni-filter

Fig. 16)

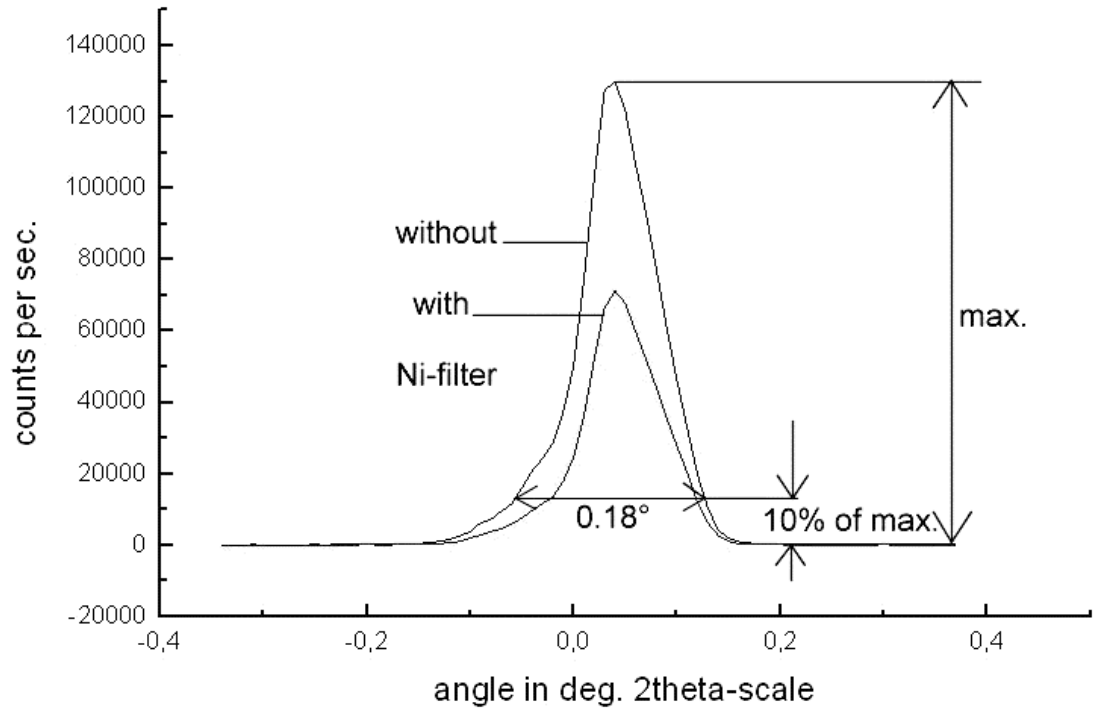


Fig. 4-16: A typical Göbel mirror reflection with full width at 10% max. measured with and without Ni-filter

Fig. 17)

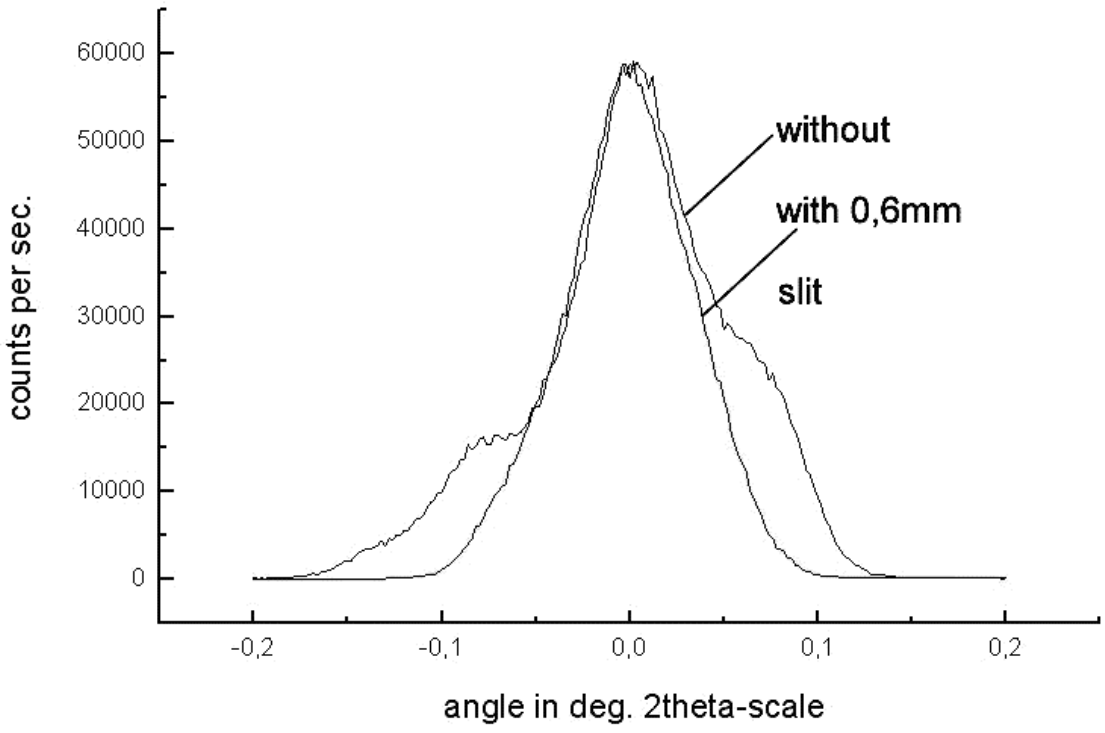


Fig. 4-17: A typical Göbel mirror reflection at high power (40kV/40mA) measured with an 0.6 mm exit slit and without

Fig. 18)

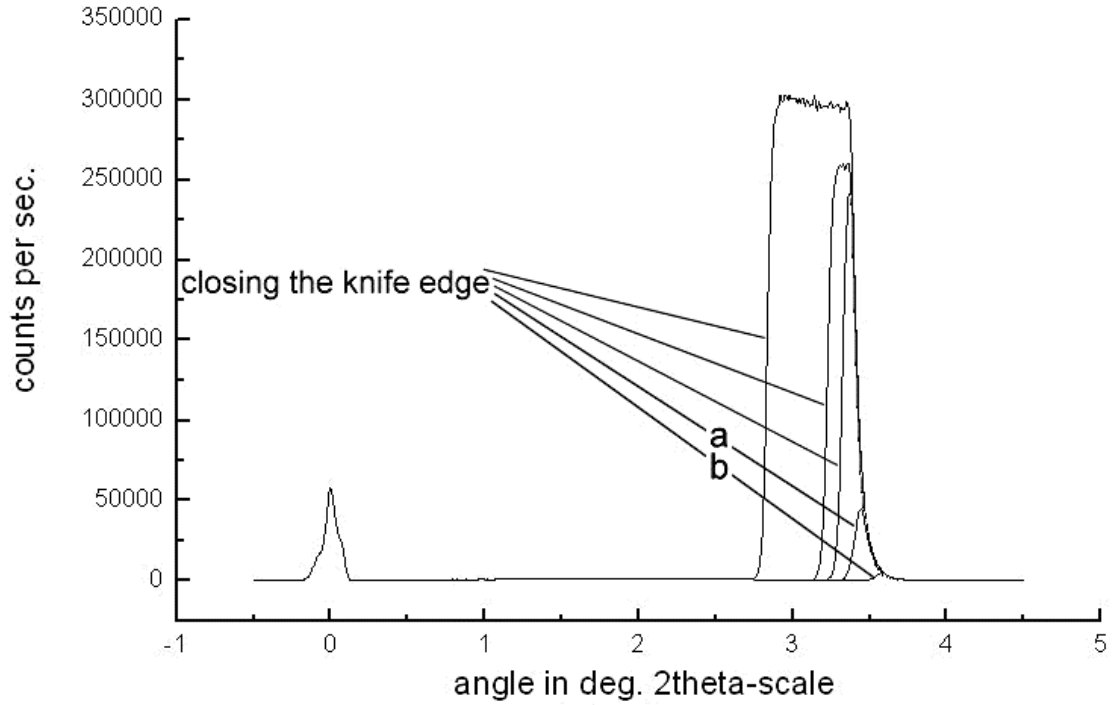
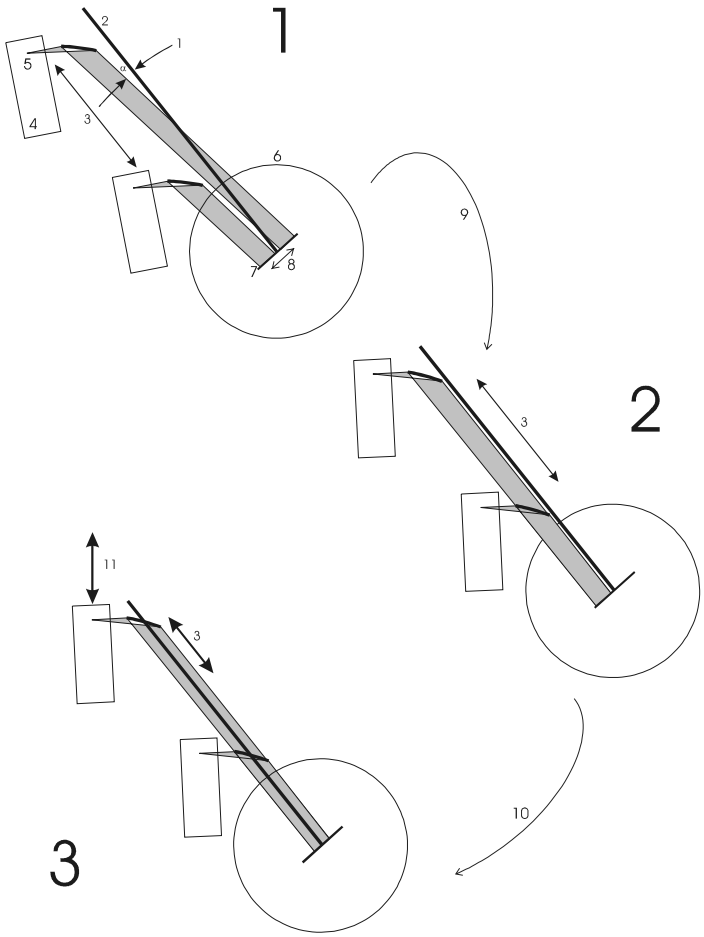


Fig. 4-18: Disappearing direct beam during the closing of the knife edge. For the end setting of the knife edge, a narrow part of the direct beam a/b has to remain in order to avoid cutting off intensity from the mirror's beam

Fig. 19)



1) Divergency α (beam and track are not parallel).
2) Direction of the primary track.
3) Moving direction of the tube along the track.
4) X-ray tube.
5) Tube focus
6) Measuring circle
7) Fluorescent screen.
8) Beam misfit caused by divergency of beam with respect to the track direction.
9) Rotation procedure step.
10) Translation procedure step.
11) Translation direction of tube mount.

Fig. 4-19: Alignment procedure and beam paths for nonparallel beams which do not hit the goniometer centre

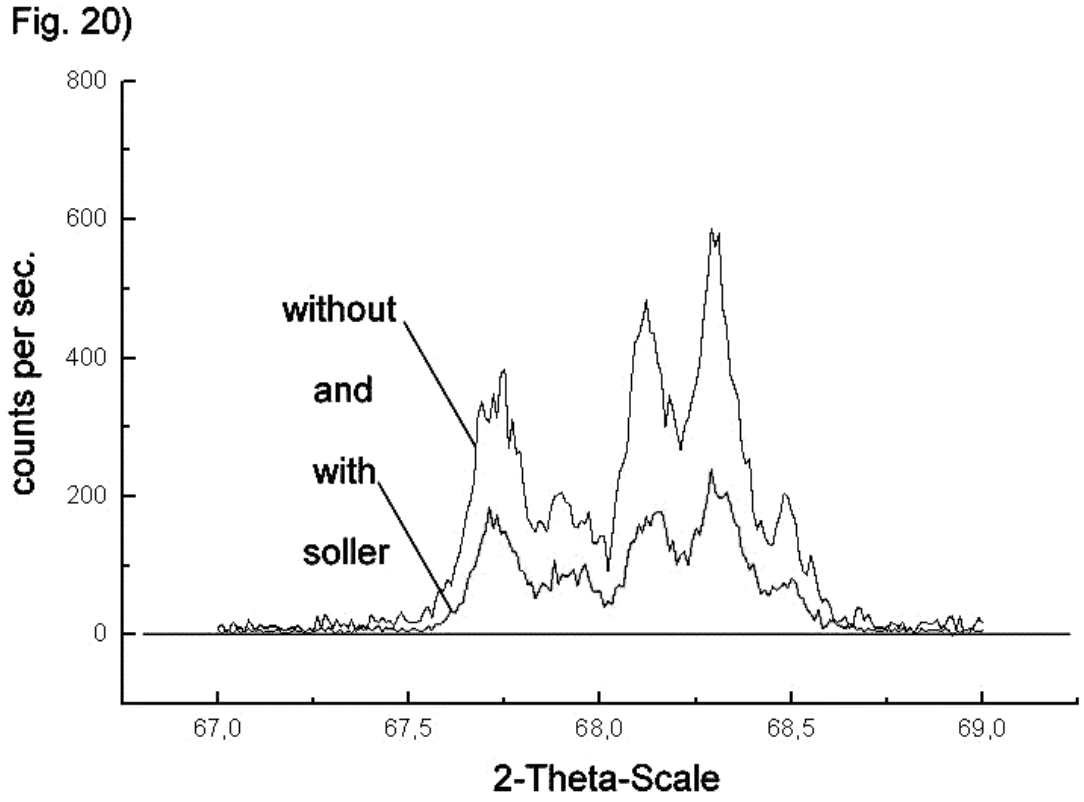


Fig. 4-20: 5 fingers of quartz measured with and without a primary 4° soller

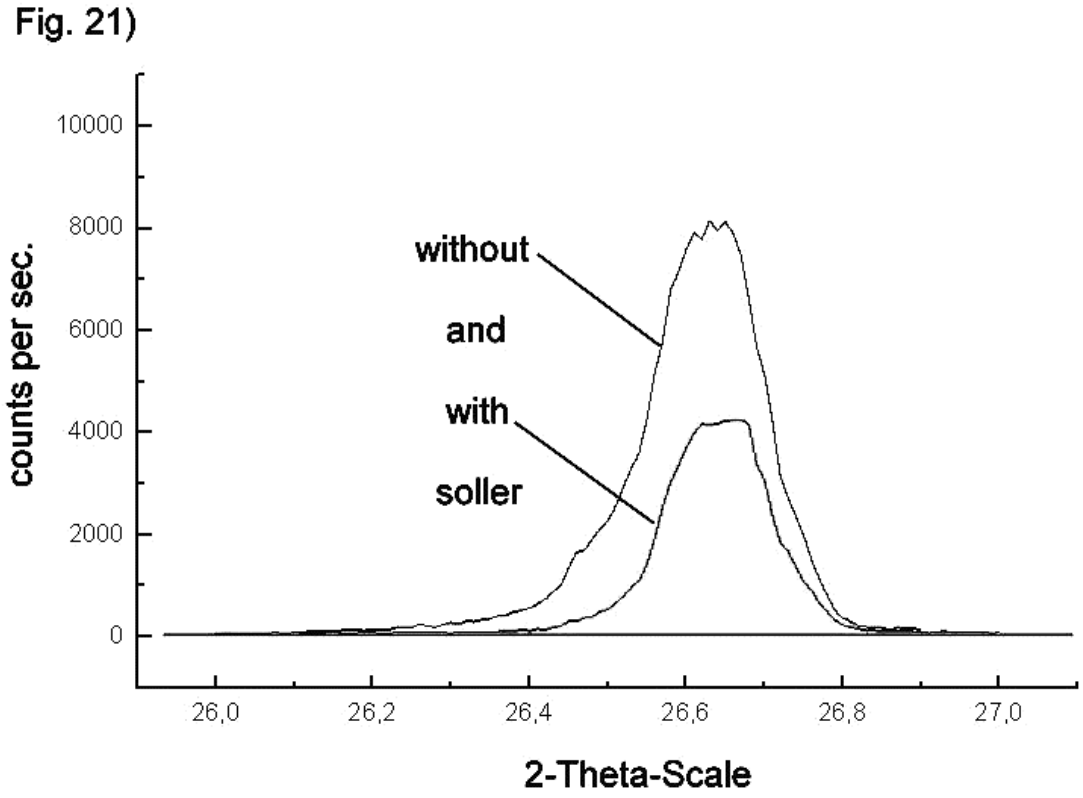


Fig. 4-21: Main peak of quartz measured with and without a primary 4° soller

5 Assembly and Alignment of Single Göbel Mirrors 3rd Generation for D8 with Cu Radiation and Line Focus Tubes

Contents

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5 Assembly and Alignment of Single Göbel Mirrors 3rd Generation for D8 with Cu Radiation and Line Focus Tubes

Introduction

The main component of a Bruker-AXS Göbel Mirror of 3rd Generation (GM III) is the multilayer optics on a prefigured substrate (fig. 2). The standard lengths of the multilayer optics are 40 mm and 60 mm. The substrate surface (part of a parabola) and lattice spacing gradient are optimized for the D8 sealed tube housing. The Göbel Mirror converts the divergent beam emitted from a line shaped X-ray source into a parallel beam (fig. 1).

These special diffractive properties apply to the Kα radiation of the source, so that the mirror is mainly diffracting the Kα part of the spectrum. Other wavelengths are supressed by several orders of magnitude. Since the Göbel Mirror collects divergent source radiation, a parallel primary beam is conditioned which is more brilliant than the radiation created by slit optics. Further, the parabolic surface of the substrate is made with very high precision as is known from space technology. In this way bending tolerances are completely avoided.

The multilayer optics is in a radiation safe housing which has three degrees of freedom to align the mirror (fig. 3a). The position of the mirror's surface (moving screws 1/2/4 simultaneously), the Bragg angle (screw 1) and the relative orientation of the mirror's surface (screw 2) with respect to the line focus can be aligned in a proper way to ensure the best performance.

The following sections describe how the Göbel Mirror has to be mounted in detail to the D8 tube housing, the basic and the final alignments of the mirror.

Preparation work

1) The D8 has to be set up and aligned as described in the D8 users manual.
2) The detector and discriminator have to be set up for Cu-Kα radiation.
3) Make sure that the D8 has a sample stage and a fitting alignment glass slit. In some cases, sample stages are available in which no glass slit fits into - in this case another alignment procedure may be valid.

Removal of the primary slit assembly (fixed or motorized slit assembly)

To remove the primary optics from the dove tail track, loosen both fixing screws at the bottom clamp mechanism so that the optics can be switched above the dove tail.

Mounting the Göbel Mirror

Before mounting the mirror to the tube housing, make sure that the three alignment screws (1, 2 and 4) of the housing are in position as shown in fig. 4b (**60 mm mirror**: A=flush=0 mm, B=4.5 mm and C=4 mm. **40 mm mirror**: A=3.2 mm, B=4.4 mm, C=3.9 mm). This is the mirror's starting position. Then move the incident beam knife edge 6 by turning screw 3 (fig. 3a) clockwise until the stop position (open knife edge). Do not use too much force during moving the screws! Mount the mirror flush to the tube housing by use of four M4 screws shipped with the mirror (fig. 3b, screws 8a, 8b, 8c, and 8d).

Alignment of the Göbel Mirror

The following sections describe the alignment of the mirror, starting with a rough alignment and finishing with the final alignment of the D8 Goniometer with the mounted Göbel Mirror. To execute the alignment job DIFFRAC^{plus} ADJUST is necessary.

The steps of alignment are listed below:

- Rough setting of the tube mount
- Rough alignment of the mirror's incident angle by use of a fluorescence screen
- Prealignment of the beam direction with respect to the center of the goniometer
- Alignment of tilt and incident angle by use of the counter
- Final alignment of the position of the knife edge and the exit slit
- Final alignment of the beam direction with respect to the center of the goniometer
- Performance test: measuring the standard Quartz sample

Prealignment of the incident angle

The presetting of the alignment screws (fig. 3a, 4b) and the incident beam knife edge was described in the previous sections. Within this section the prealignment of the Bragg angle of the mirror by means of a fluorescence screen is described. Please use the following settings:

Configuration:	
Generator	40kV, 40mA
Exit slit of the mirror	open, centered dove tail
Sample	mounted fluorescence screen
Anti-scatter slit	none
Kβ-filter, Kα-absorber	none
Receiving slit	-
ϑ or tube	45°
2ϑ	-

In a first step the tube mount and, therefore, the direction of the beam emitted by the X-ray source has to be roughly aligned with respect to the center of the goniometer. In general, the D8 tube mount to be used with a Göbel Mirror should have at least two degrees of freedom of alignment. The first one (fig. 5, 1) is a translation to align the primary beam parallel to the edge of the primary beam track. The second one is a rotation (fig. 5, 2) with a pivot point roughly below the center of the Göbel Mirror. Fig. 5 shows the tube mount with two degrees of freedom in a version released in Mai 1999. Other tube mounts may look different but support the same or even more degrees of freedom.

If you intend to change the diameter of the measuring circle (435 mm, 500 mm, 600 mm) with a minimum of alignment work, you should align the Göbel mirror beam parallel to the primary track to the center of the goniometer. This task is described later in this manual. If you use only <u>one</u> diameter of the measuring circle (e.g. 435 mm), it is not necessary to align the beam parallel to the track, but you have to bring the beam to the goniometer center.

The rotation degree of freedom 1 (fig. 5) sets the Göbel mirror beam to the center of the goniometer. Mount the fluorescence screen 1 (fig. 6) e.g. into the sample holder cup 2 (used with rotation sample stage) and move ϑ or the tube (for a theta/theta instrument) to the above given angular position. Close the enclosure doors and open the shutter with a software command. Observe the fluorescence screen and turn screw 2 (fig. 5) step by step (1/8 of a turn) clockwise and watch the fluorescence screen again with open shutter.

The enclosure doors should be opened smoothly and slowly to make sure that the shutter closes before the doors are open, otherwise the safety circuit may switch off the generator. Continue turning screw 2 (fig. 5) until the illuminated area is seen in the right section of the screen (fig. 6, 3). Now the rough alignment of the direct beam is done.

In the next step close the knife edge until a direct beam of approx. 2 mm remains. Tune the setting of the incident angle to the mirror with screw 1 (fig. 3a). Turn screw 1 approx. 1/16 to 1/8 turn clockwise (fig. 11) and observe the illuminated area on the screen. In case the

incident angle is above 0° and below the Bragg angle of Kß and Kα radiation a total external reflection of the mirror surface can probably be observed (fig. 7). In general, this beam overlaps with the direct beam passing the mirror's surface. Therefore, this beam can easily be distinguished from the first order multilayer Bragg reflection, which is the alignment goal.

Continue turning screw 1 (fig. 3a, fig. 11) clockwise in steps of an 1/16 to 1/8 of a turn. If the direct beam becomes smaller and disappeares open the knife edge until the direct beam is approx. 2 mm again. As long as the total external reflection is visible it will successively move in the direction of the arrow as shown in fig. 7 with each turn. After about a few turns - the total external reflection has already disappeared – a new illuminated line appears on the screen. This is the first order Kß reflection of the mirror.

The intensity of the Kß beam is less than that of the Kα mirror reflection. Both reflections cannot be clearly distinguished by means of the fluorescence screen only. This has to be done in a later step with the use of a Ni filter (Cu Kß suppression) and the counter.

Continue turning screw 1 (fig. 3a, fig. 11) less than an 1/8 of a turn. Within about a ¼ of a turn the Kß line should go down in intensity and another line – the Kα reflection 1 (fig. 8) of the mirror - appears. At this point use screw 3 (fig. 3a) to raise the knife edge (fig. 12) and suppress the direct beam passing the mirror (2, fig. 8 and 9) until a narrow direct beam remains visible (fig. 9).

Use screw 1 (fig. 3a) again to optimize the incident angle as far as the fluorescence screen is sensitive. Now use screw 2 (fig. 5) to move the beam 1 (fig. 9) by means of the fluorescent screen to the goniometer centre.

Finding the mirror's optimum angle

In this section the goal is to find the maximum intensity and optimum Bragg angle of the reflected Kα beam of the mirror. For the next step of alignment use the following settings:

Configuration:	
Generator	20kV, 5mA
Exit slit of mirror housing	open
Sample	none
Anti-scatter slit	6 mm
Kβ-filter, Kα-absorber	Ni-filter, 1 Cu-absorber with 0.1 mm
Receiving slit	0.1 mm

First, you have to measure the reflection and direct beam with a detector scan using ADJUST. Use -2° for the start, 5° for the stop angle and 0.02° as an increment. After this first scan, you should observe the mirror's reflection and the direct beam. If not, decrease the start and/or increase the stop angle.

If the reflection and the direct beam appear, set the increment to 0.01°. Close the knife edge until a small part of the direct beam remains visible (flourescent screen), move the detector to the centre of the reflection angle and remove

the receiving slit. Start the ratemeter, open the shutter and observe the integrated beam intensity during the optimization of the mirror's settings. Now turn screw 1 (fig. 3a, fig. 11) in small amounts (1/32 turns or less), close the enclosure door and control intensity. Set screw 1 to the maximum intensity.

Now measure the mirror's reflection (detector scan with 0.1 mm receiving slit) with and without the Ni-filter. Comparing both measurement results you can clearly decide whether the peak you found is Kα. The Cu Kβ peak is reduced by two orders of magnitude of intensity by the filter, whereas Cu Kα is reduced by a factor of about 2.

Aligning the mirror's optimum side tilt angle

If you are sure that the mirror's angle is set to the characteristic Cu-Kα radiation, align the mirror's optimum side tilt angle with screw 2 (fig. 3a, fig. 13) in this section. Leave the Ni-filter in place to be sure that you do not leave the Kα Bragg condition. Use the 2ϑ scan parameters from the last section and find the maximum intensity as follows:

1. Record the angular position of the mirror's reflection with a detector scan. This is the starting position B of screw 2 (fig. 3a).
2. Set the detector to the reflection angular position and remove the receiving slit. Open shutter, start the ratemeter and measure the integrated intensity.
3. Turn screw 2 (fig. 3a) counterclockwise 1/8 turn (position A of screw 2) and control the integrated intensity.
4. Again, find the optimum incident angle, by adjusting screw 1 (1/32 turn or less clockwise or counterclockwise) to maximum intensity. If the maximum is less than in position B go back to the starting position B of screw 2 and turn screw 2 clockwise 1/8 turn (position C of screw 2).
5. Decide whether + 1/8 (pos. A), 0 (pos. B) or - 1/8 (pos. C) turn of screw 2 results in the maximum intensity after optimizing screw 1 (fig. 3a) to maximum intensity.
6. Set screw 2 to the optimum position (A, B, C) and continue with 1)

If you have found the best position of screw 2 repeat the procedure (1-6) for +/- 1/16 turn of screw 2.

Testing the mirror's quality

To be sure that the mirror gives the proper gain, test the mirror's quality a follows:

1. Run a detector scan and measure the mirror's reflection. Adjust the zero reference by the ZI adjust button.
2. Repeat the detector scan (without Ni-filter) from - 2° to 5° for the start and stop angle and an increment of 0.01°, one copper absorber (0.1 mm) and 0.1 mm receiving slit (2° soller slit within the secondary optics).

3. Compare the intensity of the peak you just maximized to the intensity of the plateau of the direct beam. For this step a proper and correct setting of the discriminator and detector is very important. Be sure that the lower and upper discriminator level are optimized to Cu-Kα radiation.
4. The peak should have at least 1.2 (mean value of maximum and minimum) times the intensity of the direct beam plateau, proving that the intensity is higher than the direct beam (fig. 14).
5. Record the peaks intensity and position.
6. The FWHM of this reflection should be more than 1.05 mm (0.277° for 435 mm measuring circle) and for 60 mm mirrors and 0.7 mm (0.184° for 435 mm measuring circle) for 40 mm mirrors. The shape of the peak should be approximately rectangular. Fig. 14a/b/c shows typical Göbel mirror reflections of 40/60 mm mirrors, measured without Ni-filter at 20kV, 5mA, 0.1 mm Cu absorber, 6 mm antiscatter slit, 2° Soller, 0.1 mm detector slit.
7. The plateau of the Bragg peak shows a decrease in intensity from lower to higher angles. This decrease is normal and comes from a decrease of the beam density along the mirror's length (from the beginning to its end i.e. sample side). This decrease is partly compensated by an increasing reflectivity from the mirror's beginning to its end. The decrease from the max. to the min. value should be no more than 30%.

Finding the mirror's optimum angle with high power

In this section the optimum incident angle of the mirror is aligned at high generator power.

Settings:	
Generator:	40kV/40mA (typical for Cu FL 4 KE tubes, lower for FF tubes)
Mirror housing exit slit	open
Sample	none
Anti-scatter slit	open (6 mm slit)
Ni-filter, Cu-absorber	yes, 2 x Cu-absorber each 0.1 mm
Detector slit	0.1 mm

Align the mirror using the following steps:

1. After increasing the generator power, wait for 30 minutes until thermal equilibrium between tube, housing and Göbel mirror is reached.
2. Measure the beam profile with the detector scan from –0.5° to +0.5° and increment of 0.01°.
3. Set the detector to the angular middle peak position and remove the 0.1 mm receiving slit.
4. Start the ratemeter and open shutter.
5. Turn screw 1 1/32 of a turn or less and search for the maximum integrated intensity.

6. After setting the mirror to maximum, repeat the detector scan and go on with 2).

7. Don't worry about the structures you can see on the reflection's plateau. They are quiet normal because of d-spacing tolerances and tolerances within the electron focus of the tube. Bending tolerances have been avoided completely by using a prefigured substrate with highest precision.

Multiply the integrated intensity by a factor of 20000 (absorption correction of copper and nickel). You have to get at least 1.6 x 10^9 cps for 40 mm mirrors and at least 2.5 x 10^9 cps for the 60 mm mirrors (no soller on primary and secondary side).

Align the knife edge window

Now the knife edge has to be closed as far as possible without cutting intensity from the mirror's reflection. Insert the 0.1 mm receiving slit and measure a detector scan from –1° to 5° with an increment of 0.02°. Look where the knife edge cuts off the direct beam (fig. 9 and 10) and turn screw 3 (fig. 3a) counterclockwise to close the knife edge 6 (fig. 3a). Control again by measuring a detector scan. A small amount of the direct beam must remain in order to avoid cutting the mirror's profile.

Align the mirror's exit slit

In this section the exit slit of the mirror housing will be set to the optimum position. In order to find the optimum position of the slit follow these steps.

Settings:	
Generator:	40kV/40mA (typical for Cu FL 4 KE tubes, lower for FF tubes)
Mirror housing exit slit	1.2 mm (60 mm mirrors), 1 mm (40 mm mirrors)
Sample	none
Anti-scatter slit	open (6 mm slit)
Ni-filter, Cu-absorber	yes, 2 x Cu-absorber each 0.1 mm
Detector slit	2 mm

1. Set the detector to the central angular peak position, replace the 0.1 mm receiving slit by a 2 mm one.

2. Insert a 1.2 mm slit for 60 mm GMs and 1 mm slit for 40 mm GMs into the Göbel mirror housing, open shutter and start the ratemeter.

3. Turn screw 7 (fig. 3b) for ½ turn.

4. Control intensity and maximize with screw 7 (fig. 3b)

Control the reflection profile by measuring a detector scan. The direct beam should be totally cutted by the 1.2 mm (1 mm) slit by a minimum of intensity decrease of the mirror's Bragg reflection.

Aligning the reflection parallel to the track through the goniometer centre

If you want to change the diameter of the measuring circle (from 435 mm to 500 mm or to 600 mm and vice versa) with a minimum of alignment work, you should align the Göbel mirror beam parallel to the primary track to the center of the goniometer. This work is described in this section. If you use only one diameter of the measuring circle (e.g. 435 mm), it is not necessary to align the beam parallel to the track, but you have to bring the beam through the centre of the goniometer; use the procedure as described here and omit the parallel-track part of the procedure.

1. In the previous section the rough adjustment of the beam to the goniometer centre parallel to the primary track by means of a fluorescent screen has been described. In the first step the beam has to be set parallel to the track (1 of fig. 17)(omit this, if you use only one measuring circle diameter!). Slide the tube with optics along the track 3 (fig. 19, 1) and measure detector scans in two positions – near the sample and far away from the sample. In case of divergency (beam and track are not parallel) the maximum of the beam profile has to be on a fixed angular position, otherwise the tube has to be inclined by screw 2 (fig. 5). In fig. 17 part 1 you see the beam path in the case of a divergency α. If the beam is parallel to the track then a=0 mm, i.e. the angular positions of the beam – near the goniometer centre and far away – do not change. It might happen that the beam doesn't hit the goniometer centre, this alignment will be described next.

2. Now the beam must pass the goniometer centre. Move screw 1 (fig. 5) for this alignment. Use the fluorescence screen for a rough alignment in order to set the beam to the centre. After that, a detector scan has to be performed, use ADJUST and set the detector to the maximum intensity position. Remove the 0.1 mm receiving slit and insert the 6 mm one.

3. Insert the glass slit into the sample holder and proof whether the beam passes the glass slit. Proof by means of a rocking curve scan whether the beam passes the glass slit. In most cases this condition is fulfilled after the rough adjustment because the beam is relatively wide. If not, move screw 1 (fig. 5) a little bit and repeat the measurement (tube's ZI determination for theta/theta goniometer).

4. Optimize the intensity through the glass slit by screw 1. When you have reached the maximum, repeat the rocking curve scan and control the maximum again. This procedure has to be repeated with slit widths of 0.2 mm and 0.1 mm so that the centre of the beam passes the glass slit. As a last step the ZI determination has to be done for the glass slit (setting ‚rocking curve').

5. Move the glass slit to position 0° insert a 0.1 mm receiving slit and determine the ZI value for the detector (setting ‚detector scan').

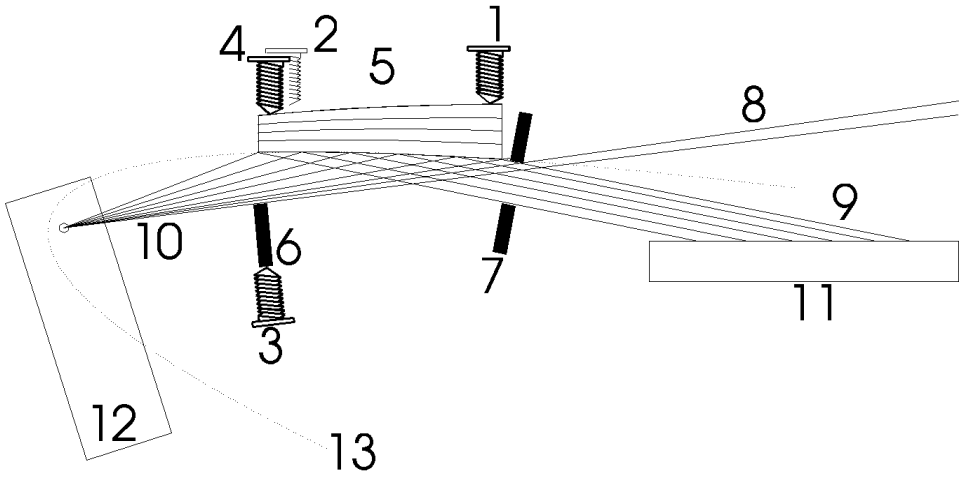
6. If the intensity through the glass slit with 0.1 mm slit width (Göbel mirror slit) is at maximum, the reference deviations of ϑ and 2ϑ are 0 or less than +/- 0.01 after ZI-determination and divergency is about 0.1° to 0.5° (angle between beam and track) the final alignment is reached.

Examples of typical diffraction scans

Here diffraction scan examples are presented in order to test the final quality of the Göbel mirror beam, i.e. divergency and intensity. Test sample is quartz and test reflections are the main peak and the 5 finger set of quartz. The parameters for these plots are 40kV and 40mA, a 1 mm exit slit at the mirror's housing, a 40 mm mirror, the 4° soller slit (secondary), a 0,2 mm anti-scatter slit and a 0,1 mm receiving slit.

Figures

Fig. 1)



1) The incident angle (Bragg's angle) can be varied with screw 1.
2) By turning screw 2, the mirror's surface can be set parallel to the line focus of the tube.
3) Screw for the knife edge.
4) Turning screws 1/2/4 simultaneously the mirror can be translated to set the take off angle.
5) Gradient multilayer optics on a prefigured substrate.
6) Knife edge.
7) Exit slit.
8) Direct beam.
9) Parallel Kα reflection.
10) Divergent beam from tube focus.
11) Sample.
12) X-ray tube.
13) Parabola.

Fig. 5-1: Beam path for Kα radiation at Bragg condition of first order with the Göbel mirror.

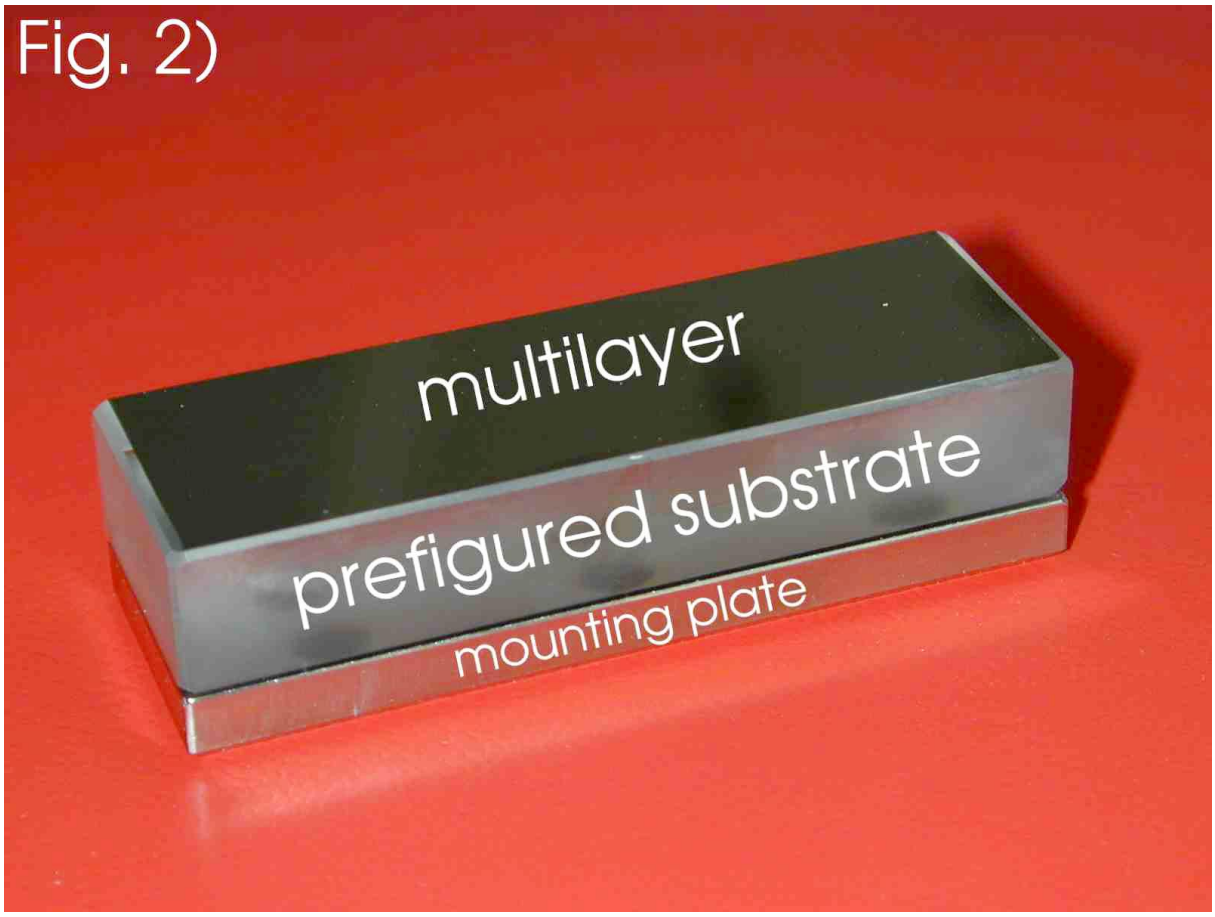
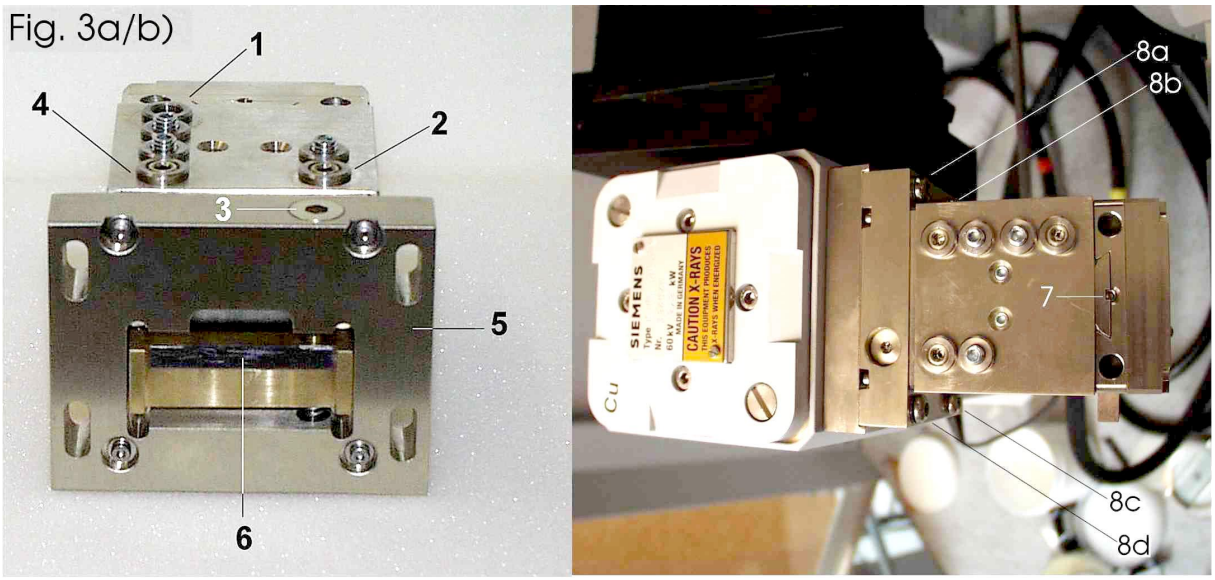
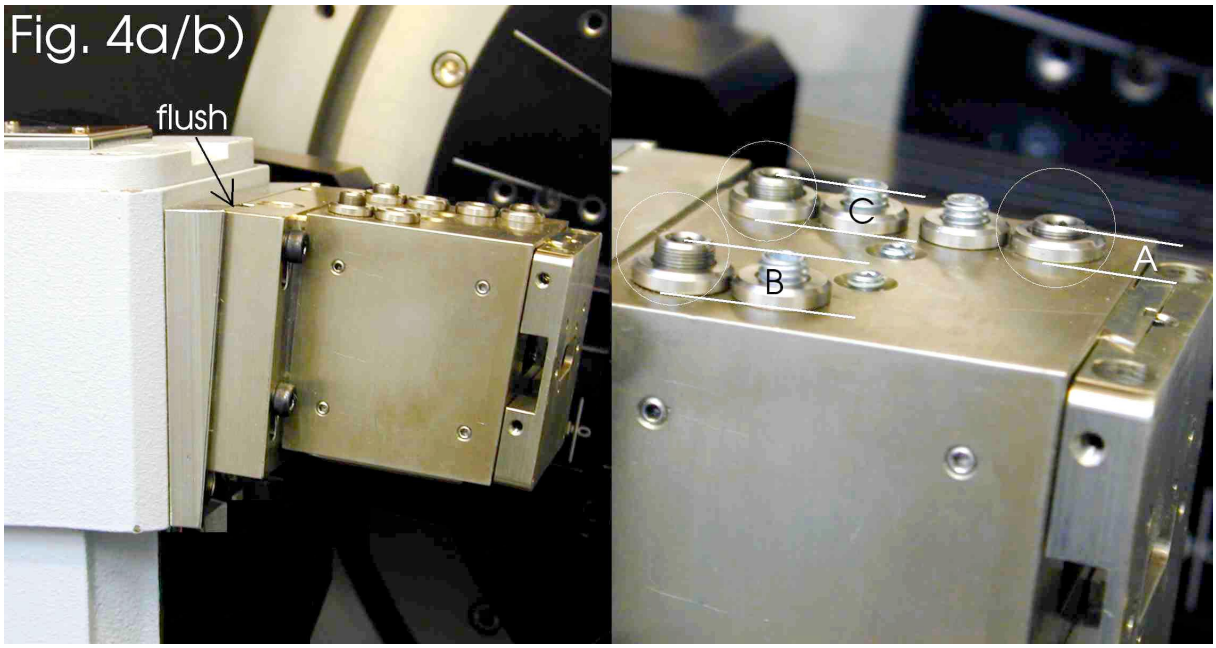


Fig. 5-2: Multilayer on a parabolic prefigured substrate which is mounted on a metal mounting plate. This example shows a 60 mm mirror.



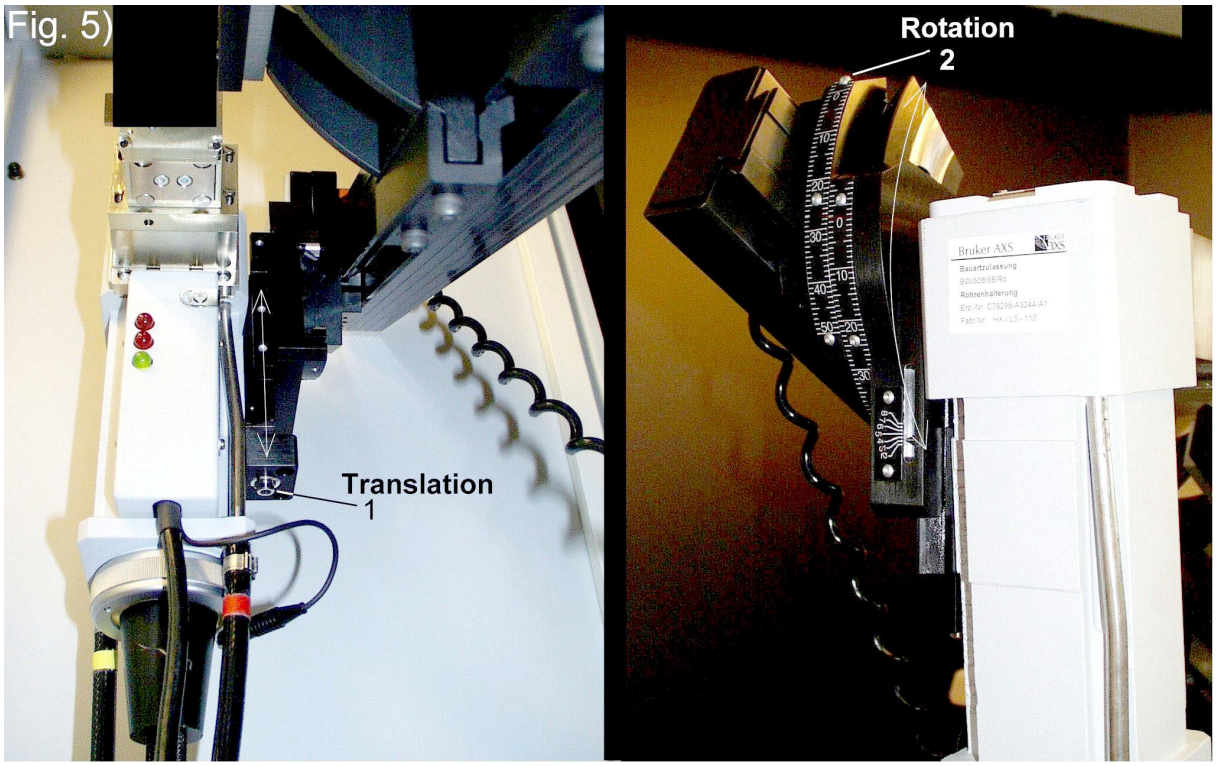
1) Variation af incident angle.
2) Set the mirror's surface parallel to the line focus with this screw.
3) Knife edge setting screw.
4) Simultaneous turning of screws 1/2/4 translates the mirror to change the take off angle.
5) Mounting plate.
6) Knife edge.
7) Screw to translate the mirror's exit slit.
8) a/b/c/d mounting screws, M4 each.

Fig. 5-3: Göbel mirror housing with mounting plate and alignment screws



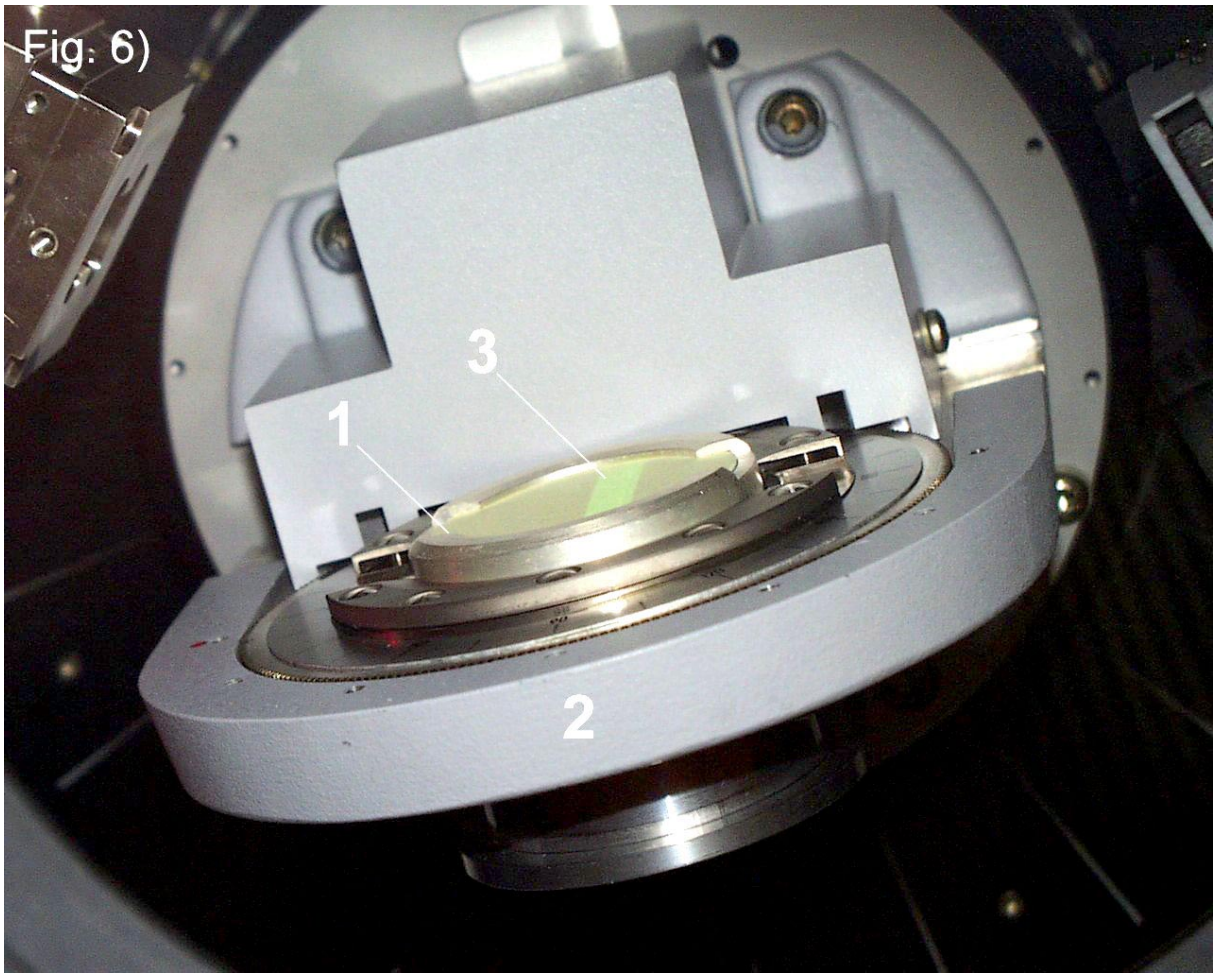
A/B/C/D) The screw's presetting (see text).

Fig. 5-4: Göbel mirror housing mounted flush to the tube housing



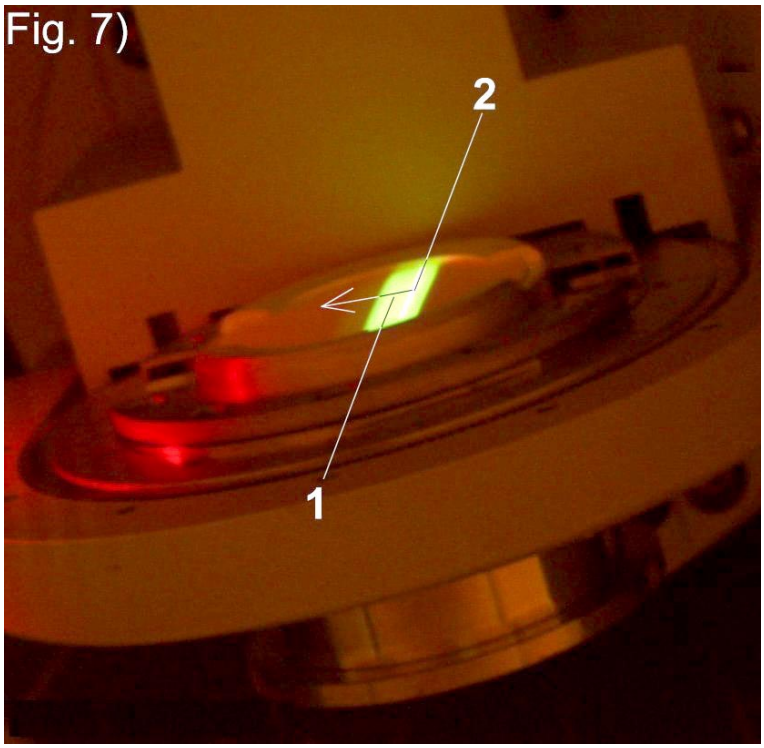
1) Translation.
2) Rotation.

Fig. 5-5: Tube mount with two degrees of freedom



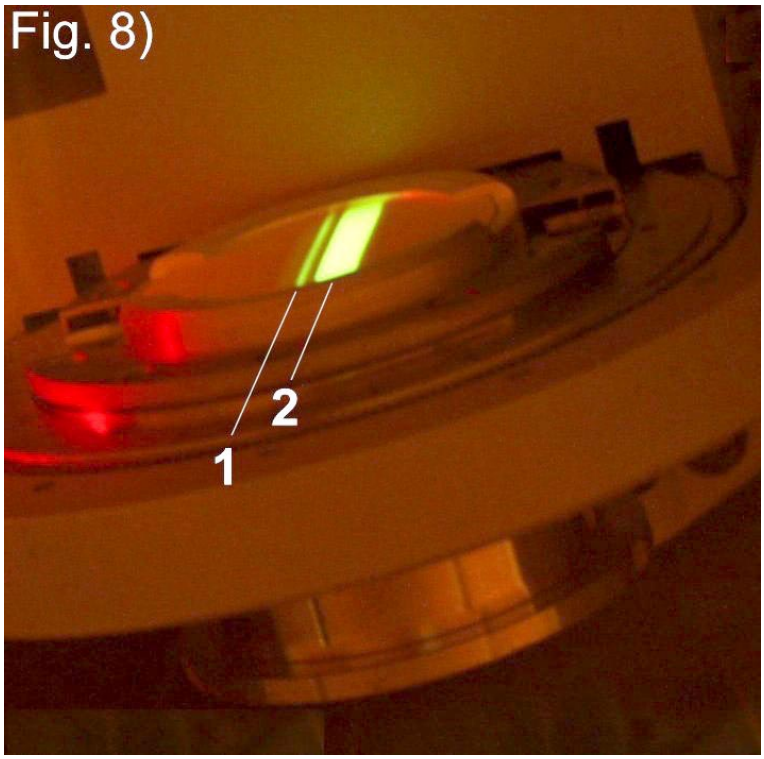
1) Fluorescence screen.
2) Sample holder.
3) Direct beam passing the Göbel mirror housing.

Fig. 5-6: Sample holder with mounted fluorescent screen



1) Direct beam.
2) Total external reflection with moving direction.

Fig. 5-7: Total external reflection from the mirror's surface



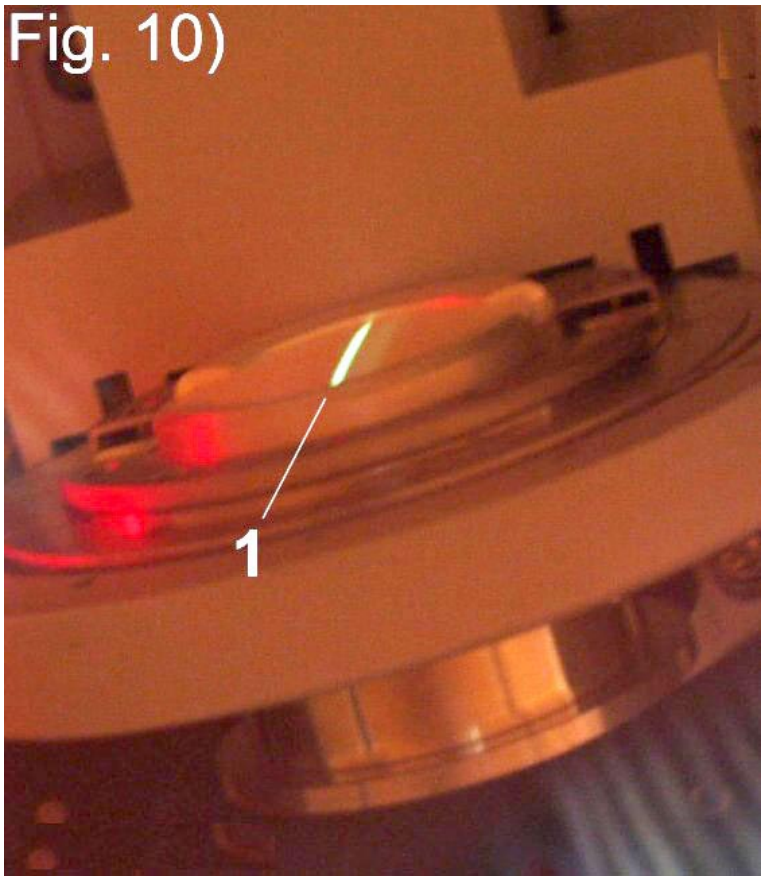
1) Kα reflection of Göbel mirror.
2) Direct beam from X-ray tube.

Fig. 5-8: Direct beam and Göbel mirror reflection



1) Kα reflection of Göbel mirror.
2) Direct beam with nearly closed knife edge.

Fig. 5-9: Direct beam and Göbel mirror with nearly closed knife edge



1) Kα reflection from Göbel mirror. Knife edge is closed completely.

Fig. 5-10: Göbel mirror reflection without direct beam

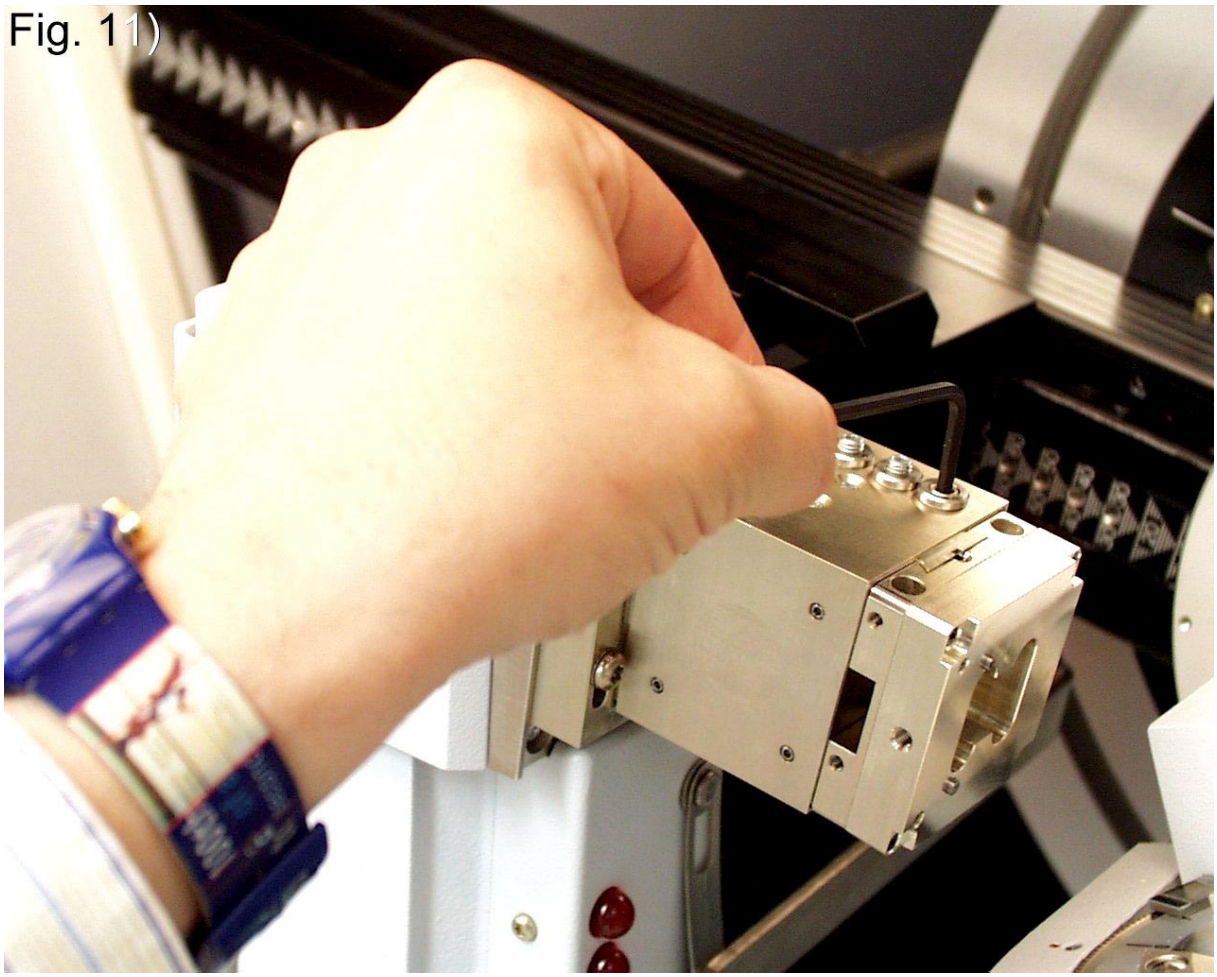


Fig. 5-11: Turning screw 1 to set the Göbel mirror to the Bragg angle.

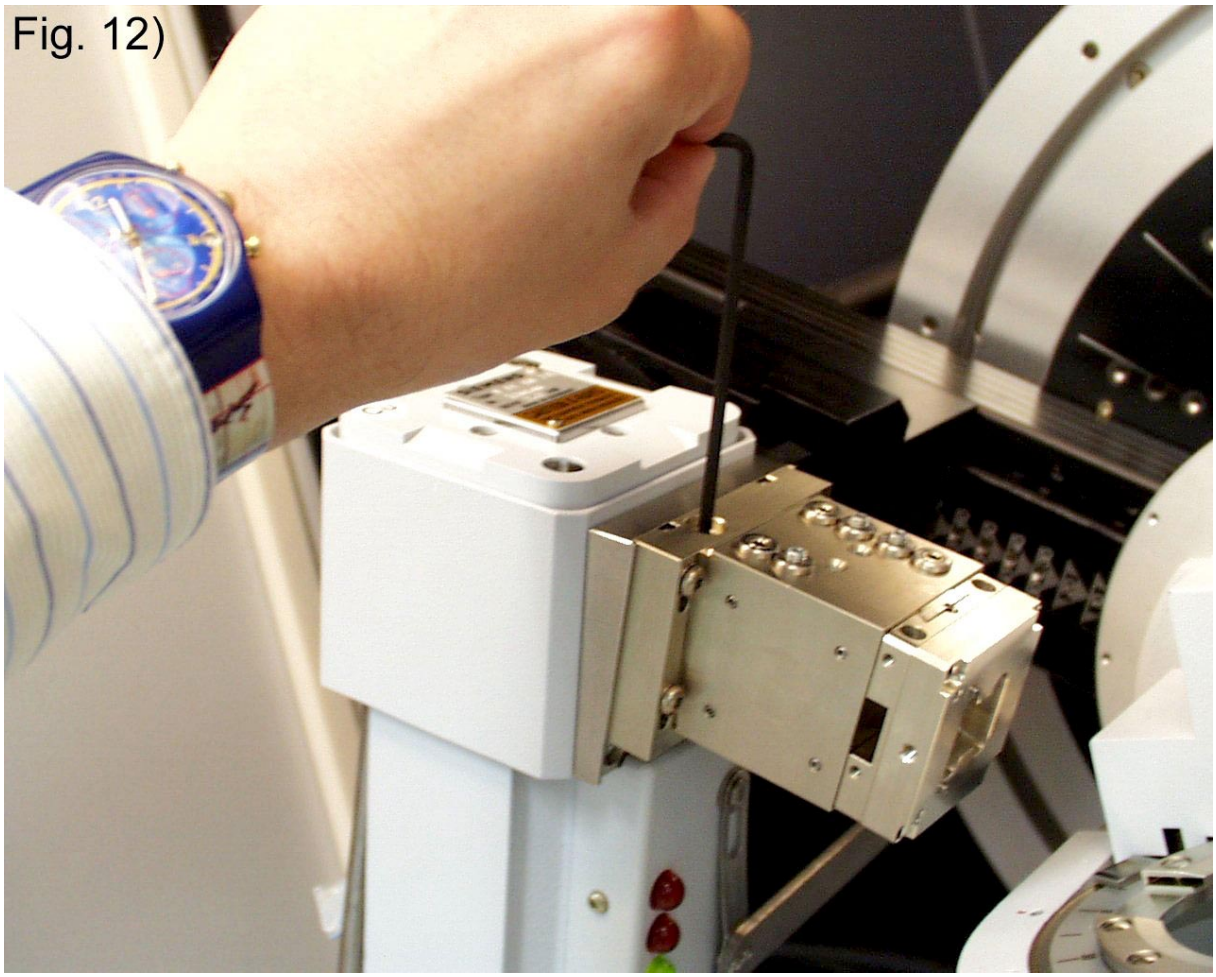


Fig. 5-12: Turning screw 3 to close the knife edge.

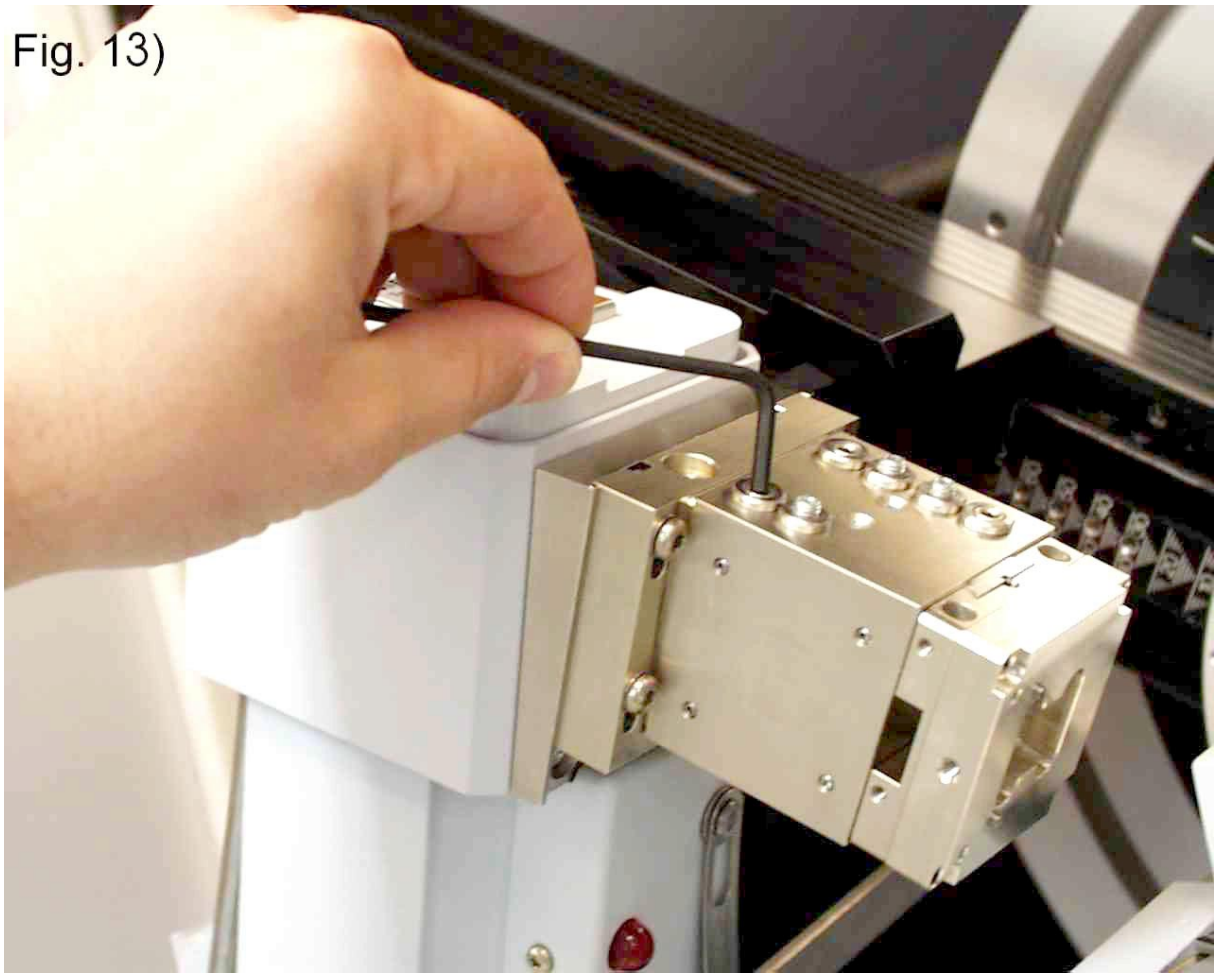
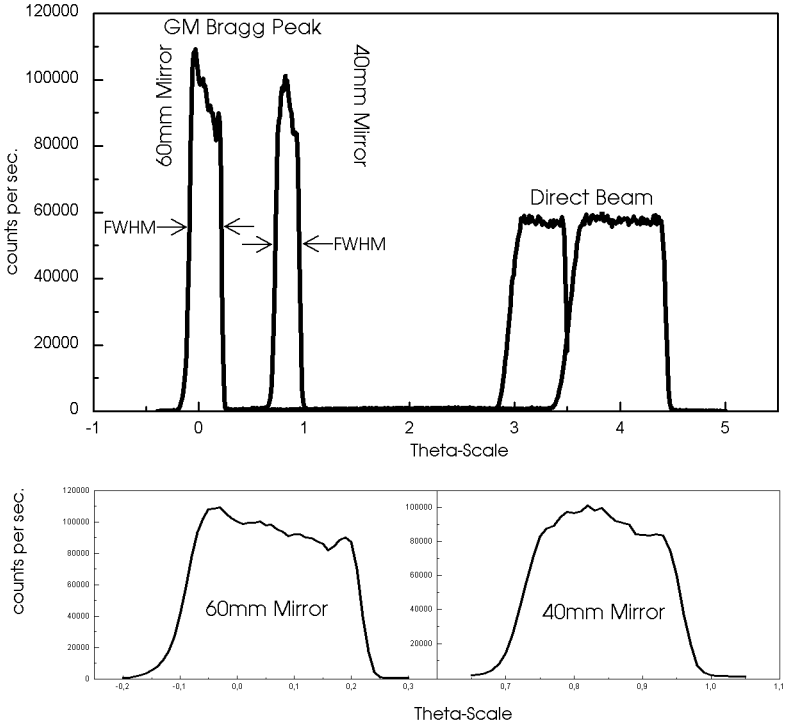


Fig. 5-13: Turning screw 2 to find the optimum side tilt angle.

Fig. 14a/b/c)



Parameters: 20kV, 5mA, no exit slit in mirror's housing, 6 mm antiscatter slit with 0.1 mm Cu absorber. 2° soller, 0.1 mm detector slit, no nickel filter.

Fig. 5-14: Göbel mirror reflections and direct beams

Fig. 15)

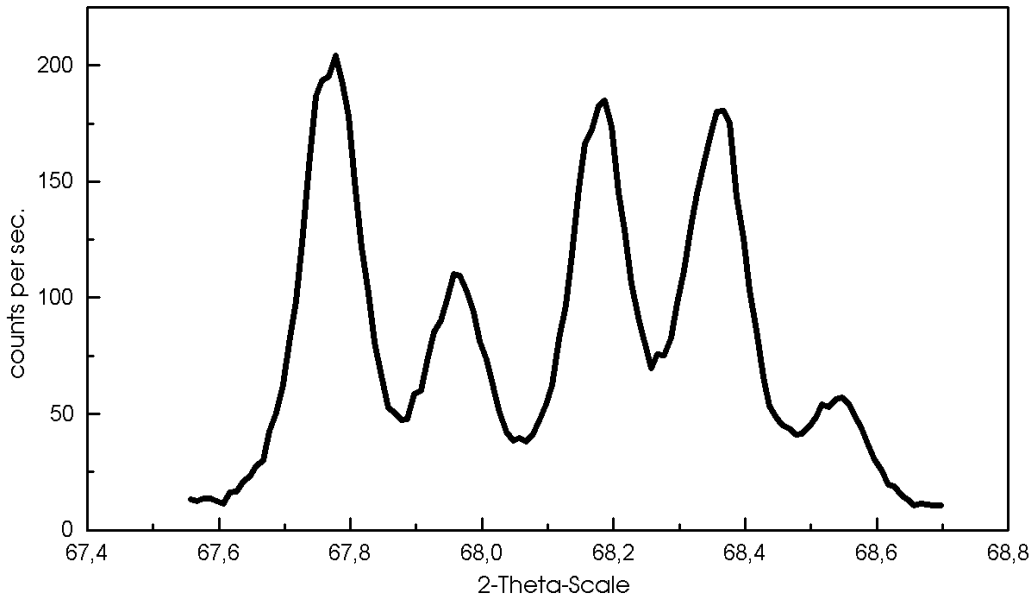


Fig. 5-15: A typical reflection profile of the 5 finger quartz, measured at 40kV, 40mA, 40 mm mirror, 1 mm exit slit, 0.2 mm antiscatter slit, 2° soller, 0.1 mm detector slit.

Fig. 16)

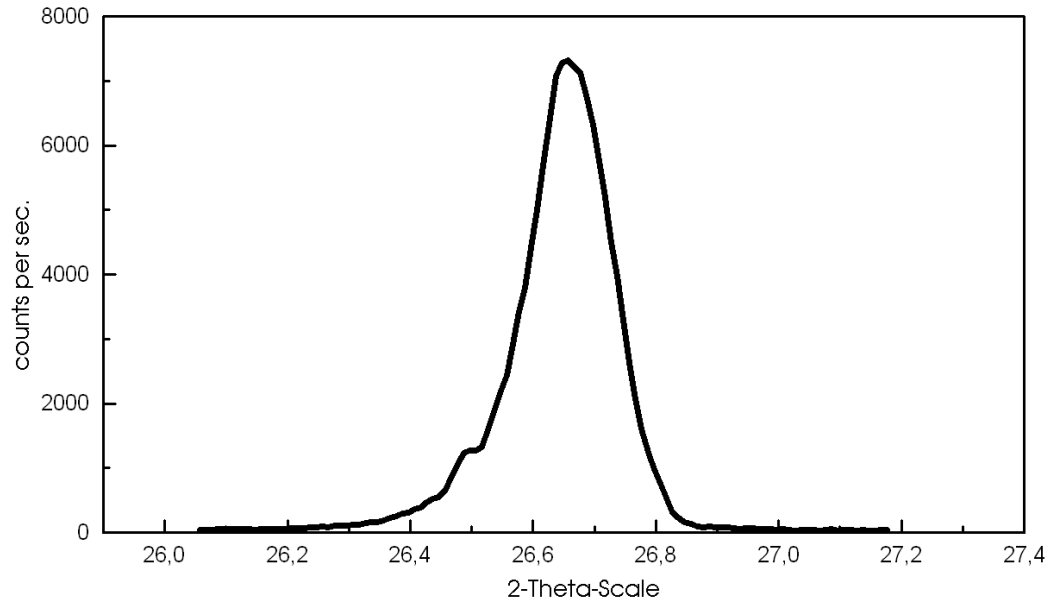
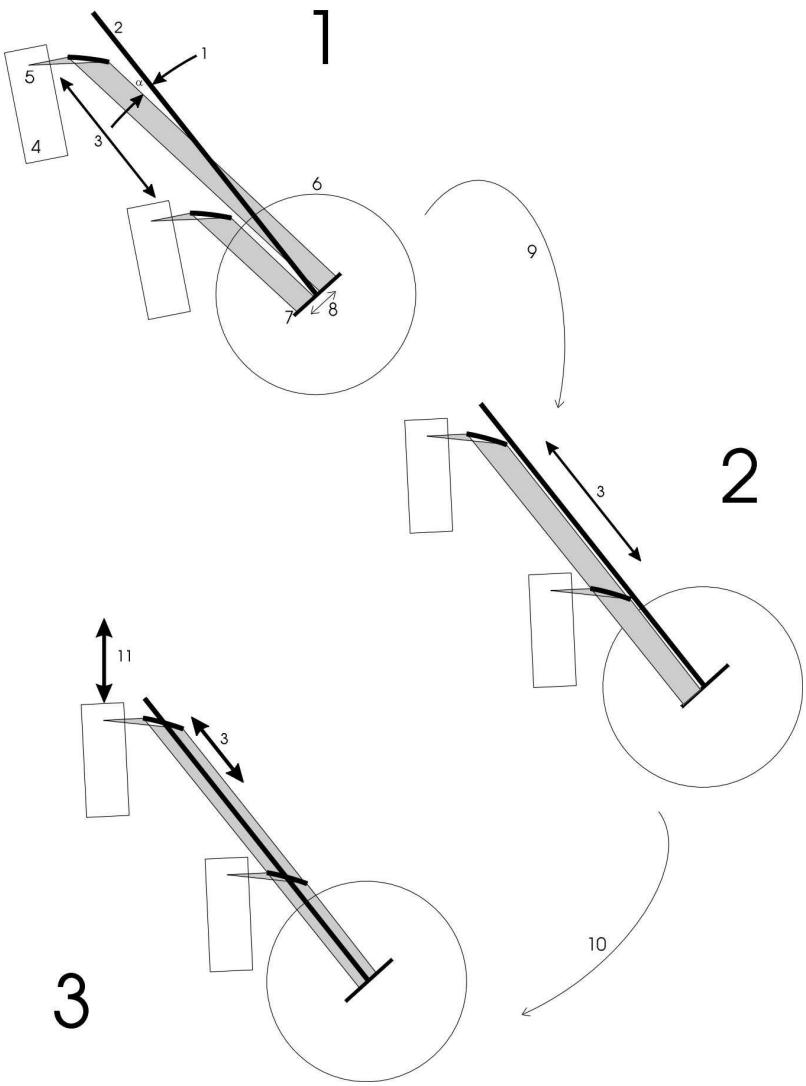


Fig. 5-16: A typical reflection profile from the main peak of quartz, measured at 40kV, 40mA, 40 mm mirror, 1 mm exit slit, 0.2 mm antiscatter slit, 2° soller, 0.1 mm detector slit.

Fig. 17)



continued next page

1) Divergency α (beam and track are not parallel).
2) Direction of the primary track.
3) Moving direction of the tube along the track.
4) X-ray tube.
5) Tube focus
6) Measuring circle
7) Fluorescent screen.
8) Beam misfit caused by divergency of beam with respect to the track direction.
9) Rotation procedure step.
10) Translation procedure step.
11) Translation direction of tube mount.

Fig. 5-17: Alignment procedure and beam paths for nonparallel beams which do not hit the goniometer centre

Tab. 5-1: Min. and max. parameters of 40 mm and 60 mm prefigured GMs III:

	40 mm GM III	**60 mm GM III**
Min. integrated intensity in cps after Absorption correction, measured at 40kV 40mA, measuring circle 435 mm, 2 Cu + 1 Ni absober, 1 mm detector slit, 6 mm antiscatter slit	1 600 000 000	2 500 000 000
Min. value of relative peak height of GM Bragg reflection to direct beam plateau	1.2	1.2
Min. value of FWHM of GM Bragg reflection (435 mm) in units of mm	0.7	1.05
Max. value of divergency in deg. (measured with (400)-Si rocking curve)	0.04	0.04

6 D8 DISCOVER Factory Alignment

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6 D8 DISCOVER Factory Alignment

Most parts of this manual are meant for factory alignment. For standard use and for in field installation the chapter ”Standard operation of the optics” should be sufficient.

⚠ **Warning:**
Factory alignment should only be performed by very experienced personnel. If unqualified personnel tries to perform the complete alignment routine, servicing by Bruker-AXS might be necessary.

Introduction

The logical sequence for aligning the high resolution monochromators on a D8 DISCOVER is as following:

I. Perform basic alignment of D8 according to ”Prüfung-D8.doc” or according chapter in D8 user’s manual vol. I (chapter 3, Alignment of the diffractometer)

II. Installation and optical centreing of eulerian cradle

III. Alignment of Goebel mirror intensity

IV. Alignment of Goebel mirror beam parallel to primary track

V. Positioning of monochromator base

VI. Alignment of channelcut crystals

VII. Alignment of beam tilt parallel to goniometer axis

VIII. Alignment of near sample pinholes (Standard: C79298-A3158-B153)

In case of systems without Goebel mirror, the D8 should be prealigned for usage with Goebel mirror. The steps 3 to 6 should be performed with a borrowed Goebel mirror. Steps 7 to 8 should then be performed after taking out the Goebel mirror and pushing the tube to contact with the channelcut monochromator. The rotation of the DOF must be tuned to get the beam through the monochromator. Therefor the tube flange should be approx. parallel to the monochromator (the Bragg angle - 3 ° - of the Goebel mirror must be compensated).

Without Goebel mirror the line or point focus may be used, depending on applications to be done with the instrument. Changing from line to point source requires some realignment as described in ”Standard Operation of the Optics”, see below.

Conditions

The following conditions have to be fulfilled in step III. to VIII. if nothing different is defined.

- 40 kV, 40 mA, KFLCu2K x-ray tube (ceramic tube with long fine focus), line focus, warm up time 0.5 h
- Tube at the track end on primary side if standard 40 mm Goebel mirror is installed (type 1),
  60 mm before track end if no Goebel mirror is installed,
  15 mm beyond track end if 60 mm Goebel mirror is installed (type 2),
- Detector end approx. flush with track end on secondary side (fig. 6-1), (negative 2theta must be possible and box window should not be touched during 2theta movement).
- Goebel mirror housing flush with tube flange (fig. 6-2)
- No Soller slits
- In case of a 3DOF mount, the vertical translation (third degree of freedom) should be reset to flush position (fig. 6-2). Remark: The vertical translation is actually realized by a small rotation and a relatively long arm. This degree of freedom is only needed if point focus shall be used.
- For alignment purposes it is helpful to use the X-ray interlock for factory alignment until the DOF is finally fixed (fig. 6-2). By this, the safety circuit is closed without fixed position of the tube. The DOF can be shifted along the track without shutting down the generator.

Danger: ⚠
Only Bruker service and users with special education in radiation safety are allowed to do this !

The standard interlock must be installed after alignment to get full radiation safety. Any manipulation to the interlock of the D8 can cause injury due to X-rays. If a forced or irregular interlock manipulation is detected, the licence for full radiation protection systems will be revoked.

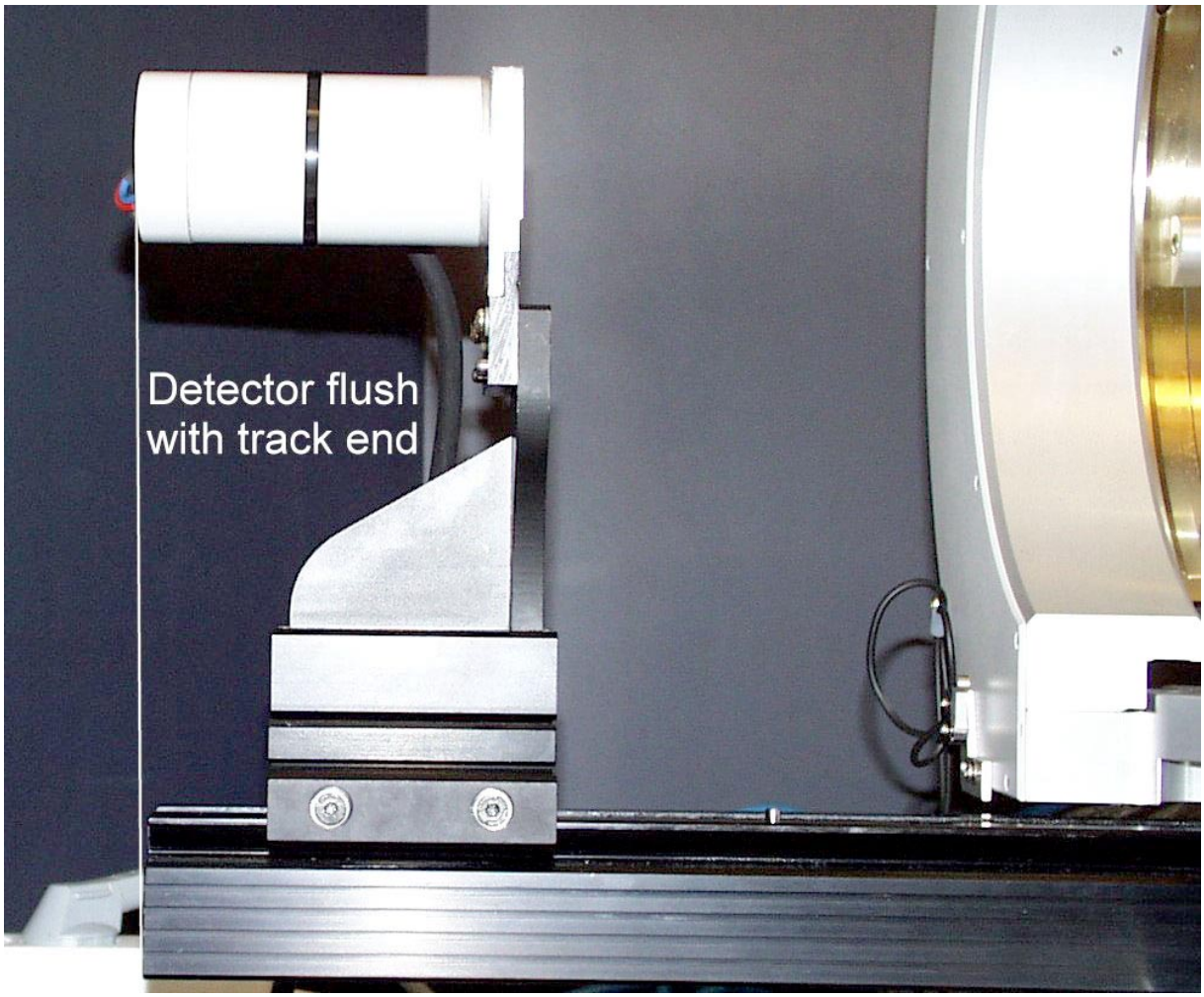


Fig. 6-1: Detector position for alignment of D8HR

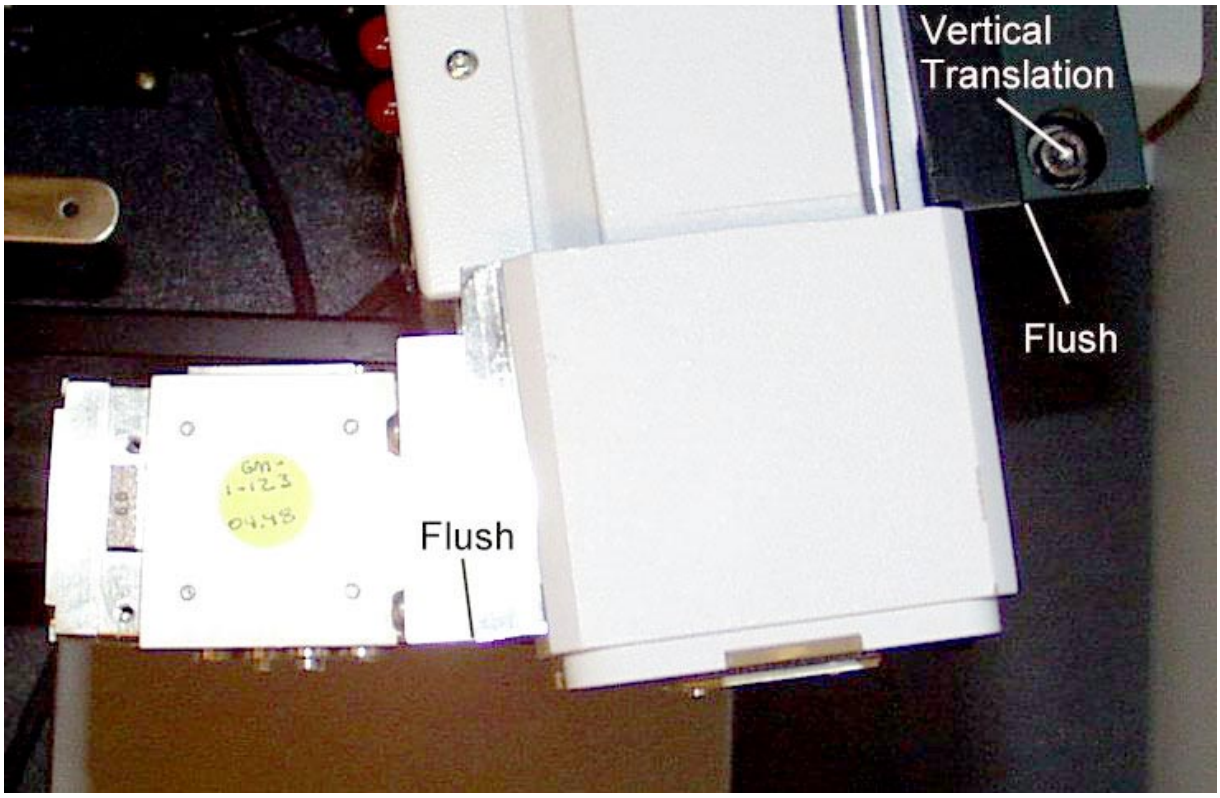


Fig. 6-2: Mounting the Goebel mirror

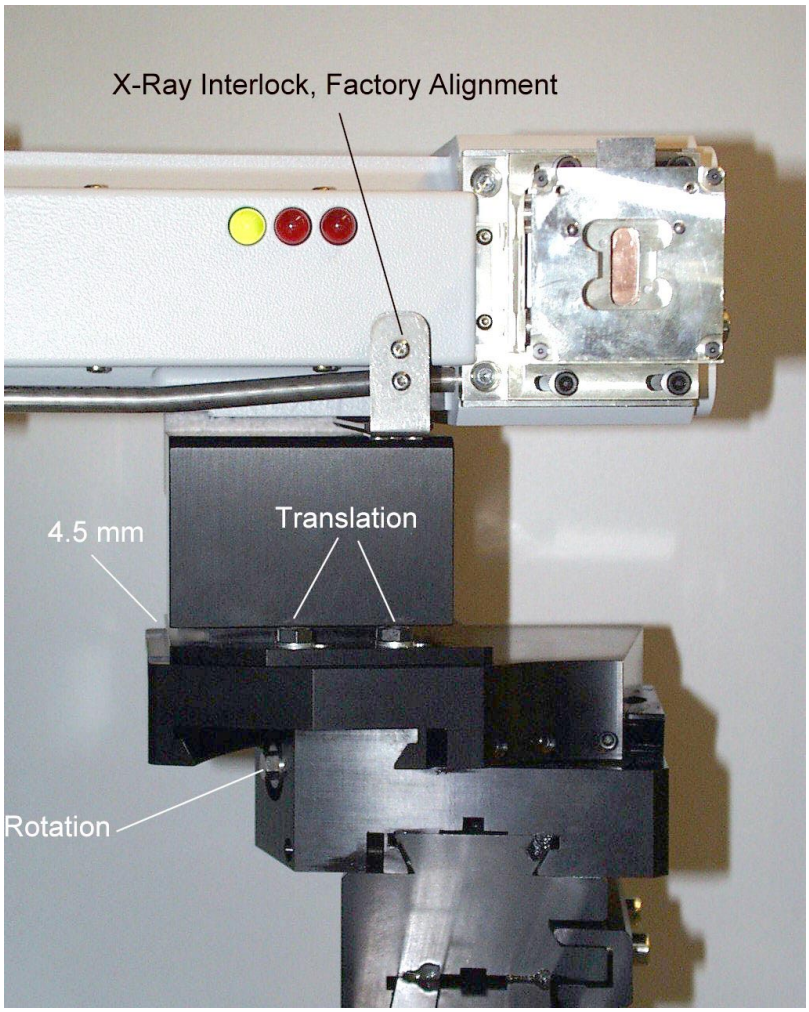


Fig. 6-3: X-ray tube with Goebel mirror on first generation 3DOF

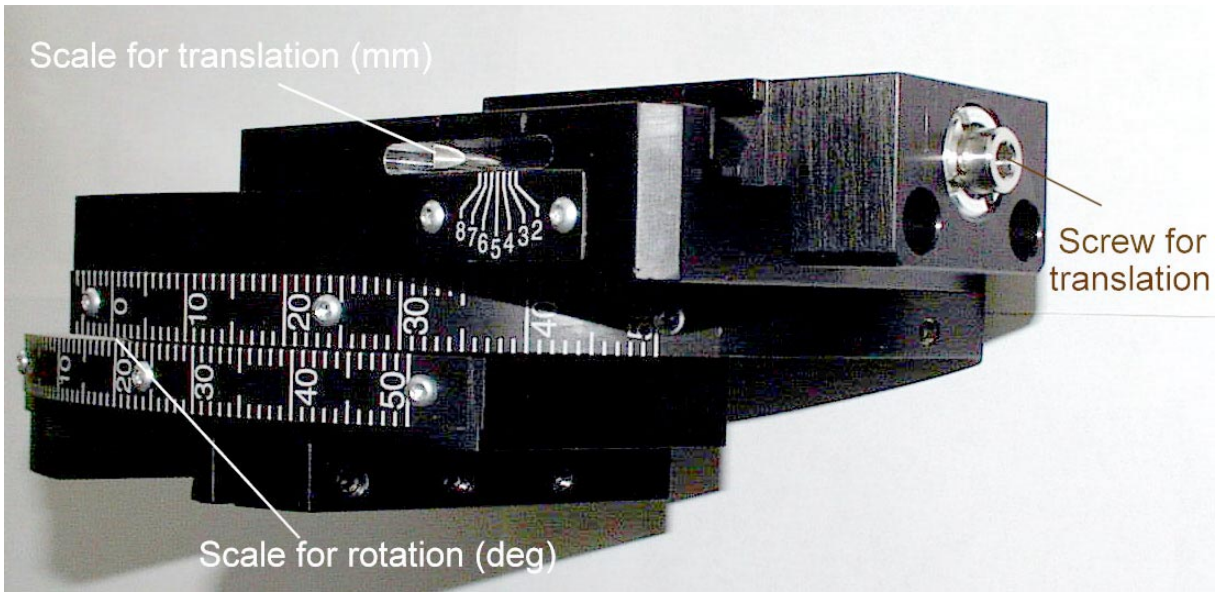


Fig. 6-4: 2nd generation 2DOF (1)

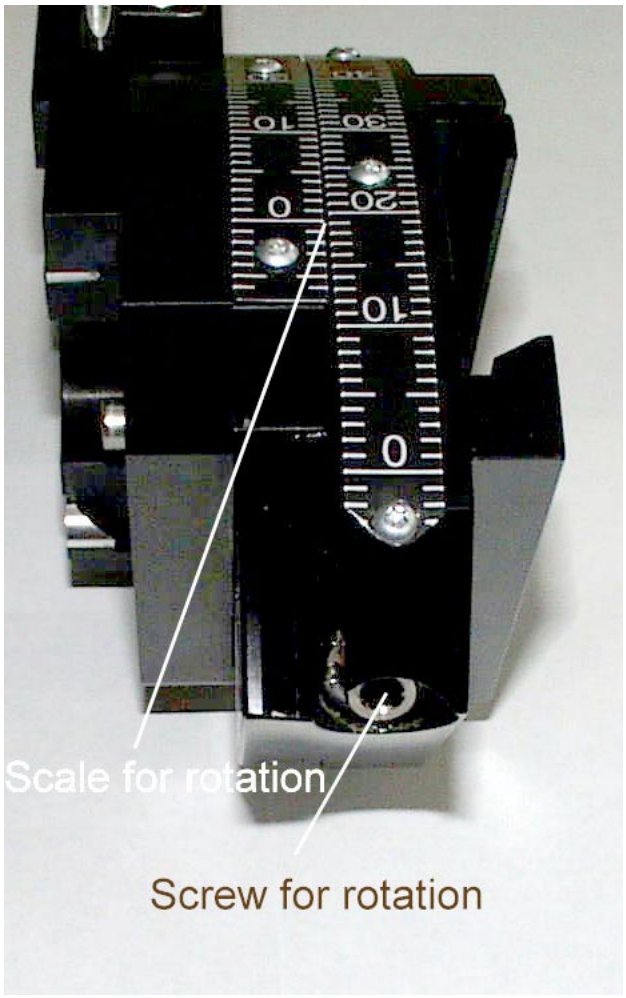


Fig. 6-5: 2nd generation 2DOF (2)

1. Centre cradle.
Mounting the cradle without play is performed in the identical way as with the standard sample carrier (chapter 3 users manual). The cables of the cradle should be connected in a way suggested by the standard quarter circle configuration (compare *.cnf files). According to user requests different motor driving channels of the D8 might be used. The motor driver boards must be switched to high current. The cables should be guided over the guiding spring (compare fig. 6-41 in the D8 DISCOVER reflectometry section).

Mount alignment pin and centre pin with telescope (cradle accessories). Therefore move to theta=90°.

⚠ **Warning:**
Be careful moving goniometer with telescope mounted. Danger of crash!

a) Centre Phi at Chi = 90° (horizontal position of Phi circle). Determine position of pin at Phi=0° and 180° with the crosshair. Move it to the average position with the x translation.

b) Move to the same position with y translation at Phi=90°. Check the position with Phi=270°.

c) Repeat a) and b) until the position of the pin is stable (within 0.2 smallest units = 8 µm).

d) The zero positions of the x and y translations have been determined and can be initialised to 0.000 mm: Inside configuration program set $Zi_{new} = Zi_{old}$ – Offset.

1. Move to Chi = 0°. If the tip is not on the Chi rotation axis the tip moves on a circle around this axis. Determine the radius of the circle by measuring the horizontal and vertical displacement of the tip with the crosshair (compared to Chi=90°). Move tip by the average of this amount with z translation towards the centre of the circle. Check with Chi=90° and iterate if necessary until the position of the tip is stable within 1.2 units = 50 µm. If this does not work the error sphere of the cradle is worse than 50 µm. Values up to 100 µm may be tolerated. Worse values require servicing of the cradle in factory.

a) Initialise z to zero: Inside configuration program set $Zi_{new} = Zi_{old}$ – Offset.

b) Check that the micrometer dial is displaying 0.00 mm if it is mounted and measuring the height of the alignment pin (x = y = z = 0.000 mm). If necessary redefine zero of dial.

2. Align Goebel mirror for maximum intensity.

a) Put fluorescent screen on cradle (accessories of cradle, fig. 6-6). Move to x= 0 mm, y=8.2 mm = thickness of fluorescent screen, z=0 mm, Phi=0°, do not use any absorbers, filters, or slits.

b) Screws #1 (Bragg angle), #4 (tilt), #5 of mirror must be flush with housing (fig. 6-8, fig. 6-19).

c) Move knife edge of mirror until 2 mm width of the white beam can be seen on the fluorescent screen.

d) Rotate DOF until white beam is in the left half of the fluorescent screen

e) Turn screw #1 clockwise in 1/8 turns until the white beam gets narrower. Total reflection can be seen close to this position of screw #1. This should not be mistaken for the Bragg reflection! This position is about 0° incident angle for the Goebel mirror.

f) Turn screw #1 clockwise in ¼ turns until the Bragg reflection appears.(approx. 1.5 revolutions). Take care that the white beam can always be seen on the fluorescent screen (width in the range 0.1 ... 2 mm), fig. 6-6. Move knife edge if the white beam vanishes!

g) Optimise the intensity with the help of the detector (move fluorescent screen out of the beam, put 0.2 mm Cu absorber and Ni filter, secondary slit system may be inserted temporarily for this purpose.). Iterate #1 (Bragg angle) and screw #4 (tilt). The intensity should be at least 100 kcps if the tube is at the end of the track (fig. 6-7).

h) Optimise knife edge position: Move until white beam just vanishes on the fluorescent screen.

Warning:
Alignment only by alternately optimising something and looking at the result through closed box window! Radiation level is hazardous if shutter is opened while box window is opened! (Only possible when overriding the safety circuit and not accidentally.)

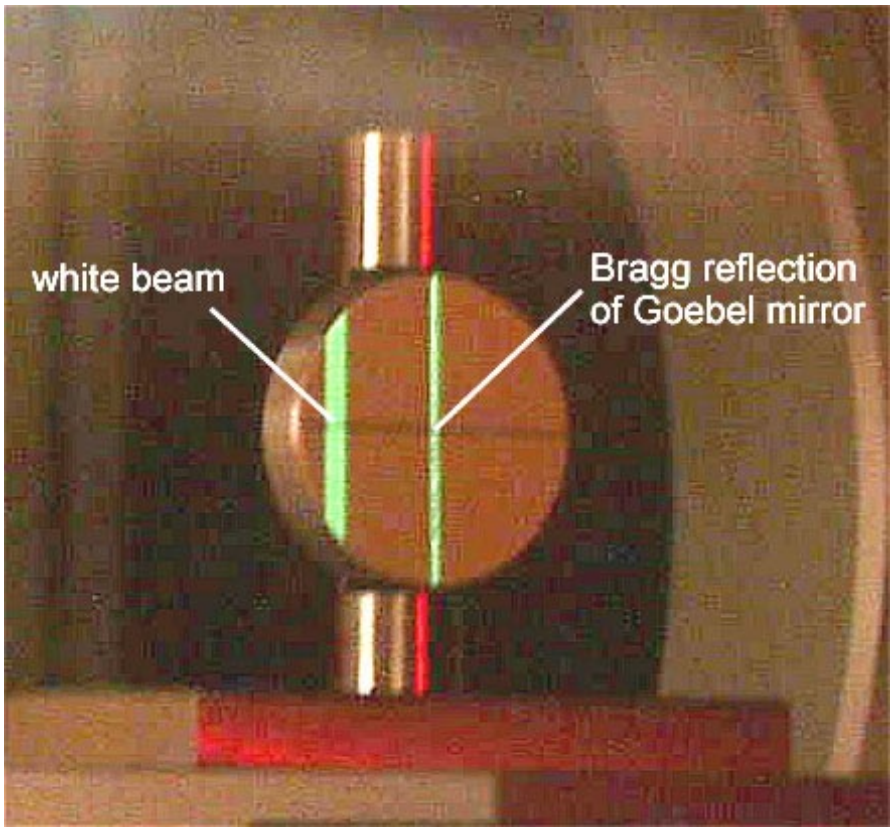


Fig. 6-6: Primary beam of Goebel mirror, view from tube side

The beam is parallel to the track and going through the centre of the goniometer. X=0 mm, y=8.2 mm, z=0 mm,Phi=0°, Chi=0°, Theta=0°. The knife edge of the Goebel mirror is not optimised. Due to this the white beam can be seen left from the monochromatic Bragg reflection. The brightness of both should be roughly the same.

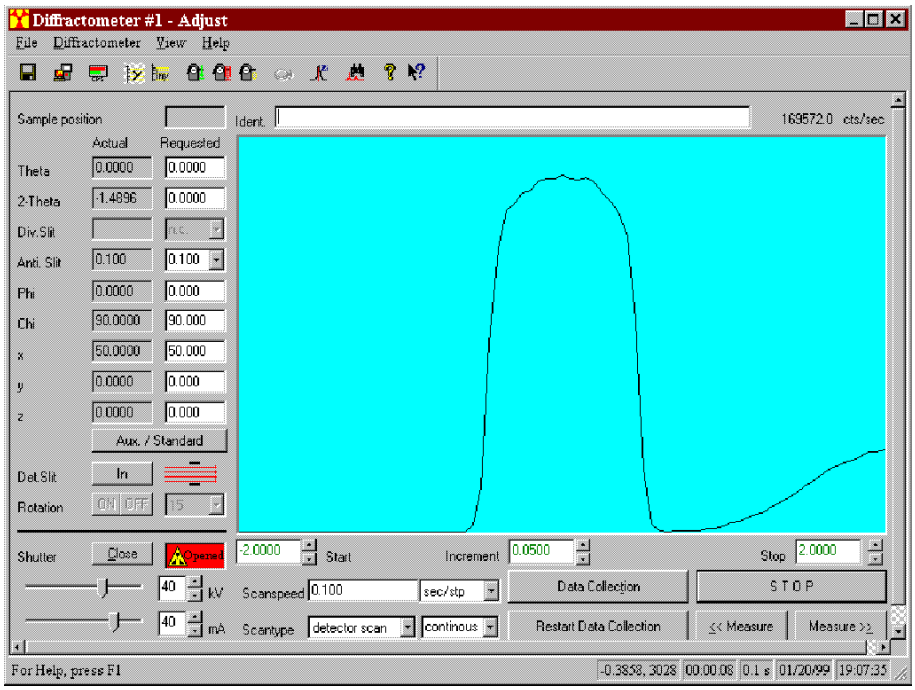


Fig. 6-7: Primary beam, Goebel mirror only at 40 kV, 40 mA with 0.2 mm Cu absorber and Ni filter

3. Align beam of Goebel mirror parallel to track and through centre of goniometer
(4bounce monochromator not mounted)

a. A coarse alignment should be done using the crosshair on the fluorescent screen (x=0 mm, y=8.2 mm = thickness of fluorescent screen, z=0 mm, Phi=0°, Chi=90°, theta=0°)

First, the beam must be put parallel to the track.

- Translate the DOF by 4.5 mm in direction to the box window (fig. 6-3).
- Compare the beam position of tube close to the sample with that of tube far away from the sample (fig. 6-6, 6-8, 6-9) and rotate DOF until the beam is at the same position in both cases.
- Optimise translation of DOF so that beam hits cross hair.

b. The precise parallel to track alignment of the DOF must be performed by doing scans and optimising the rotation of the 2DOF / 3DOF (fig. 6-3, fig. 6-4). According to figures 6-8 - 6-12 the beam position can be measured by a x-scan of the pinhole (cradle accessories) through the direct beam. This must be performed with close distance tube to goniometer (fig. 6-8) and large distance (fig. 6-9). If the peak positions differ in the two configurations, this must be reduced to within 0.2 mm by the rotation of the tube. See fig. 6-10 for an example of a beam which is parallel to the track but not going through the goniometer centre.

c. Next, the beam must be put through the goniometer axis by using the translation of the 2DOF / 3DOF (fig. 6-3, fig. 6-4). The absolute peak position of the x-scans must be reduced to 0 +/- 0.2 mm. The offset in the translation of the DOF will be about 4.5 mm. Measure this distance at both edges and perform a parallel translation of the tube. In the first step just reduce the peak position in the end of track position to about 0 mm. The result should look similar to figures 6-11, 6-12.

d. Third, the beam position must be put to x=0 +/- 0.05 mm at the standard position of the DOF on the track (i.e. normally at the end of the track) by using the horizontal rotation of the DOF.

e. Redefine the zero position of theta by using the glass slit, if necessary (Rocking curve with glass slit mounted in the goniometer centre, no detector slit).

f. Redefine the zero position of 2theta, if necessary (detector scan with 0.1 mm detector slit, without glass slit). Calculate base width of beam from peak width and peak intensity. It must be within 0.6 mm ... 1 mm. Otherwise alignment or mirror are not ok.

g. Align the position of the exit slit of the Goebel mirror. Put 0.6 mm slit and optimise intensity of direct beam (fig. 6-8, screw #9). Do not

use narrower slits: Beam position might differ when changing to different exit slits because of this. Put 1 mm slit as standard exit slit. (Intensity should be the same as without exit slit. 0.6 mm slits should cut down the intensity by 5 ... 20%.). For reflectometry measurements use 0.2 mm slit.

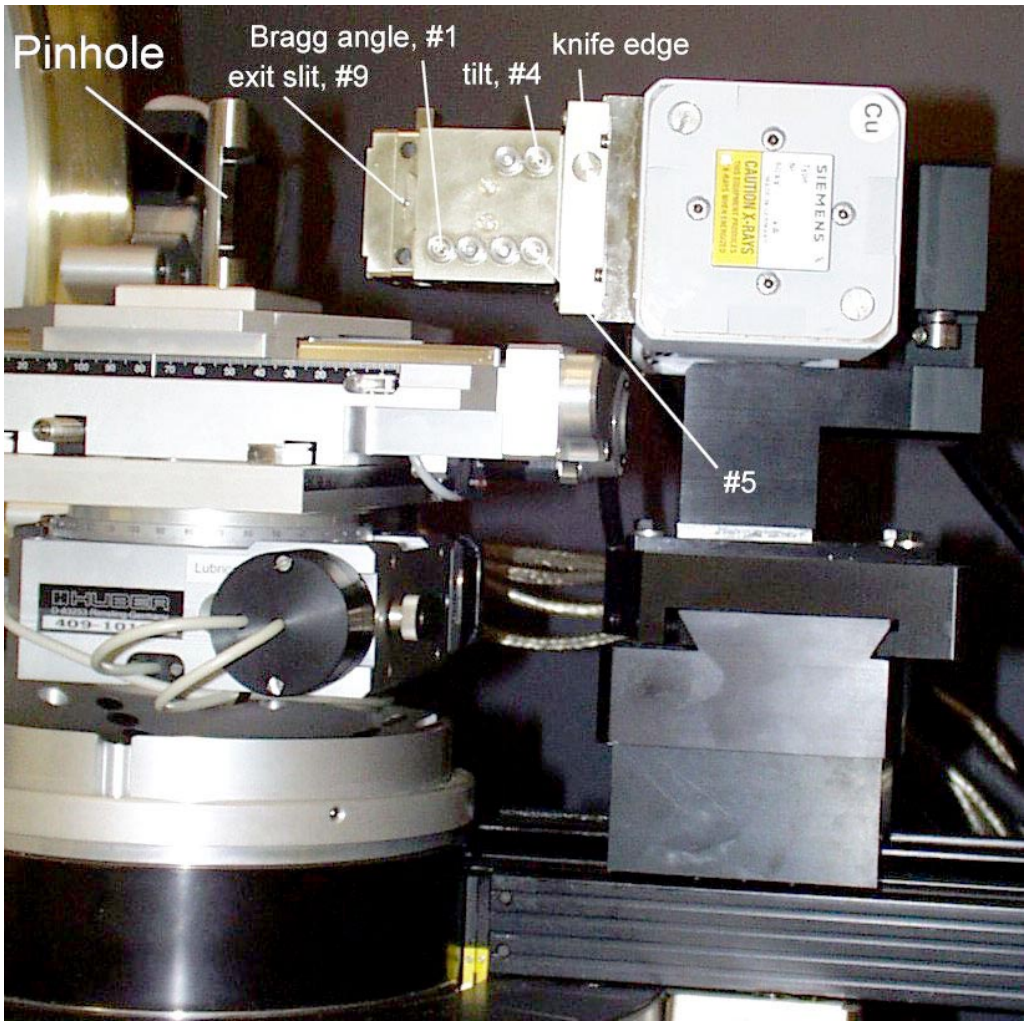


Fig. 6-8: Alignment of beam parallel to track, short distance

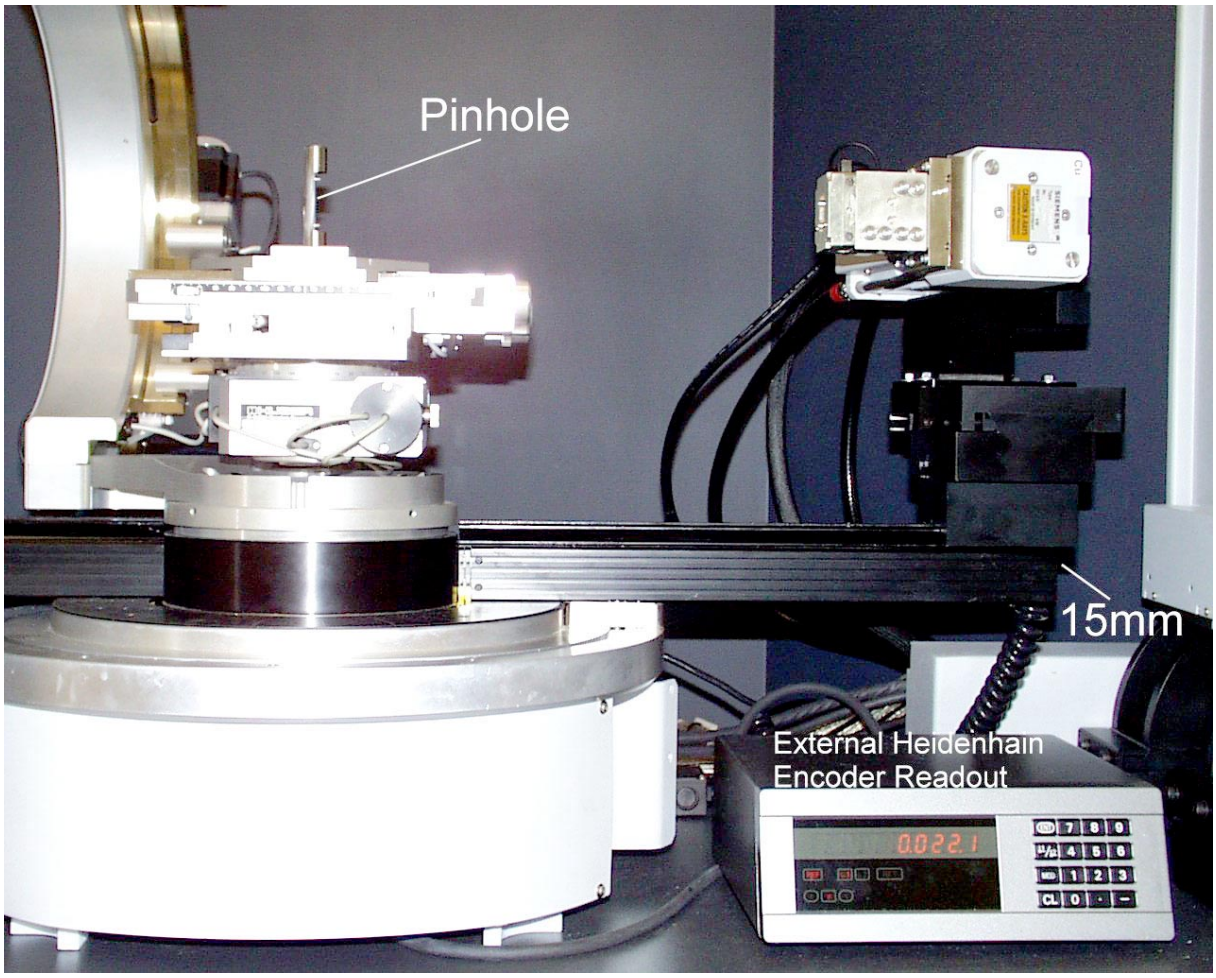


Fig. 6-9: Alignment of beam parallel to track, large distance

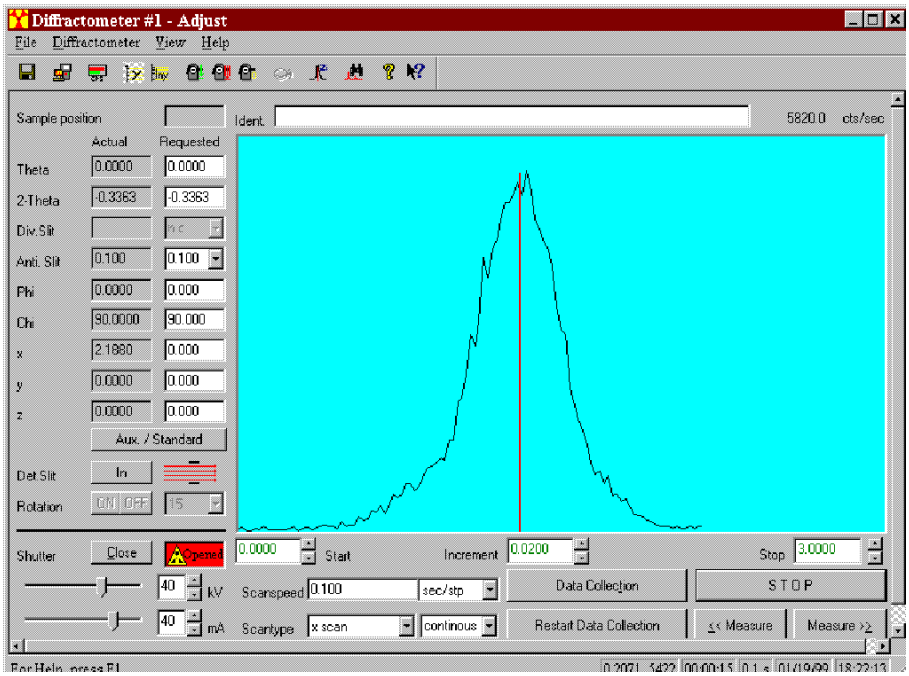


Fig. 6-10: Beam parallel to track with offset, large distance

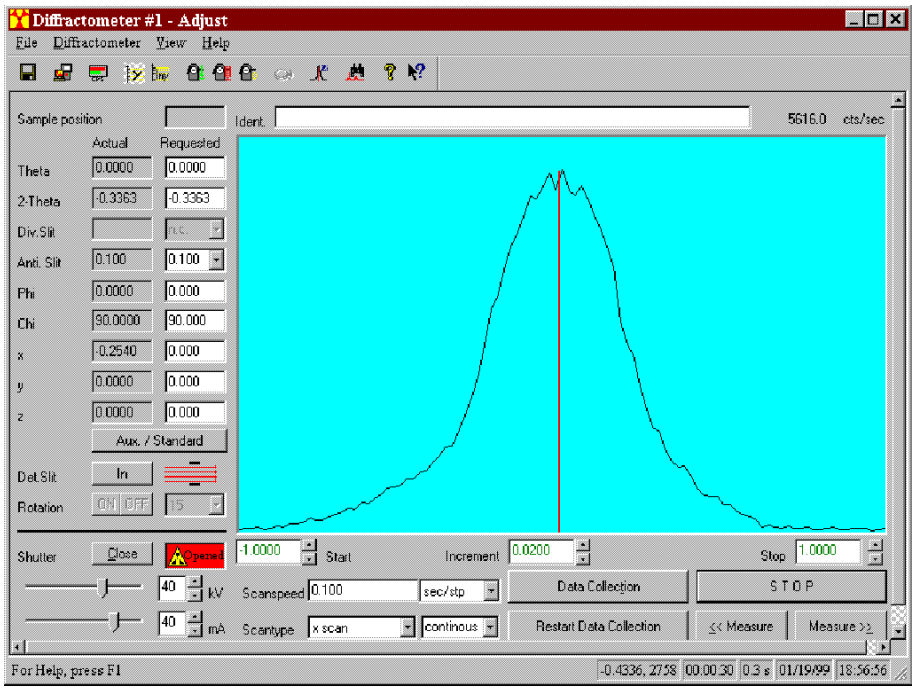


Fig. 6-11: Beam parallel track, large distance, after alignment

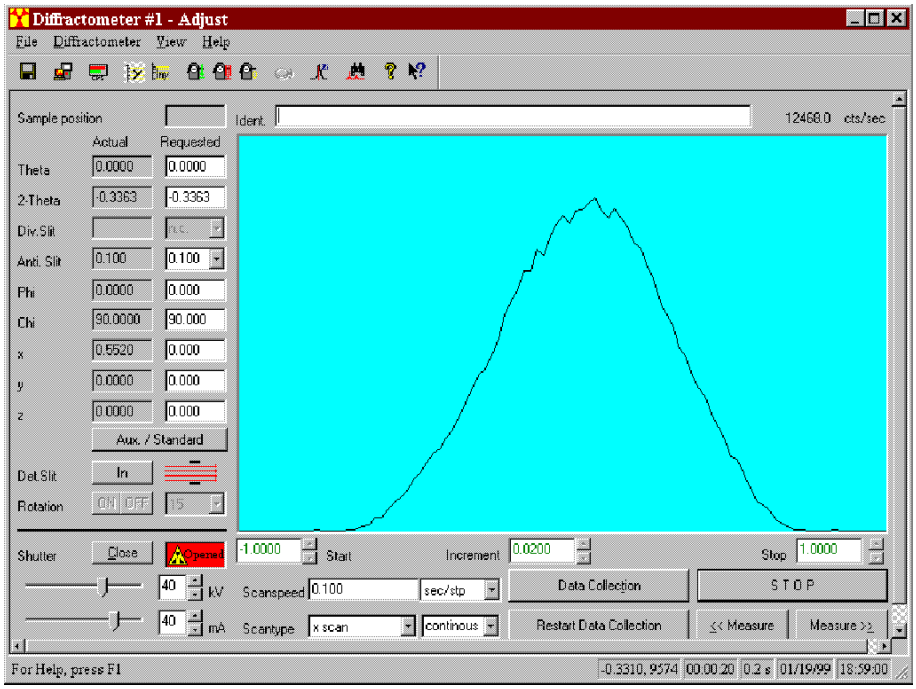


Fig. 6-12: Beam parallel to track, short distance, after alignment

4. Align beam through 1mm entrance slit of monochromator

No channelcut crystals mounted, centre slit open and translated to direct beam position. Use set screws in monochromator base underneath entrance slit (fig. 6-15, 6-16, 6-18, 6-19). Intensity of direct beam should be more than 100 kcps with 0.2 Cu and Ni filter (fig. 6-13, 6-14). Coarse alignment can be done by fluorescent screen but has to be optimised with the detector.

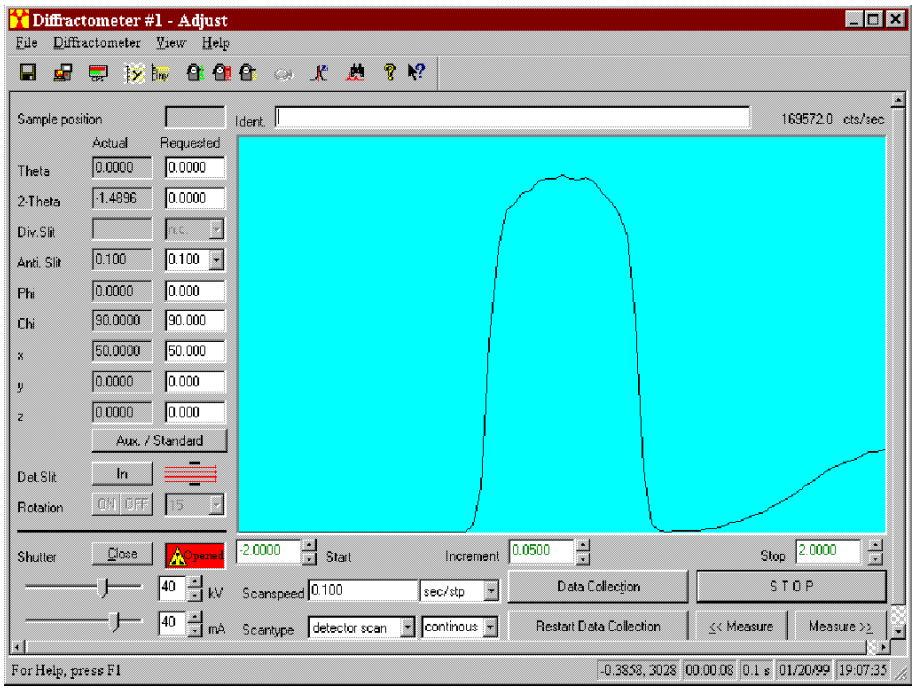


Fig. 6-13: Goebel mirror beam with monochromator base mounted, no crystals, 1 mm entrance slit, no centre slit, centre slit position as far as possible to the back (Goebel mirror beam must be able to pass centre slit)

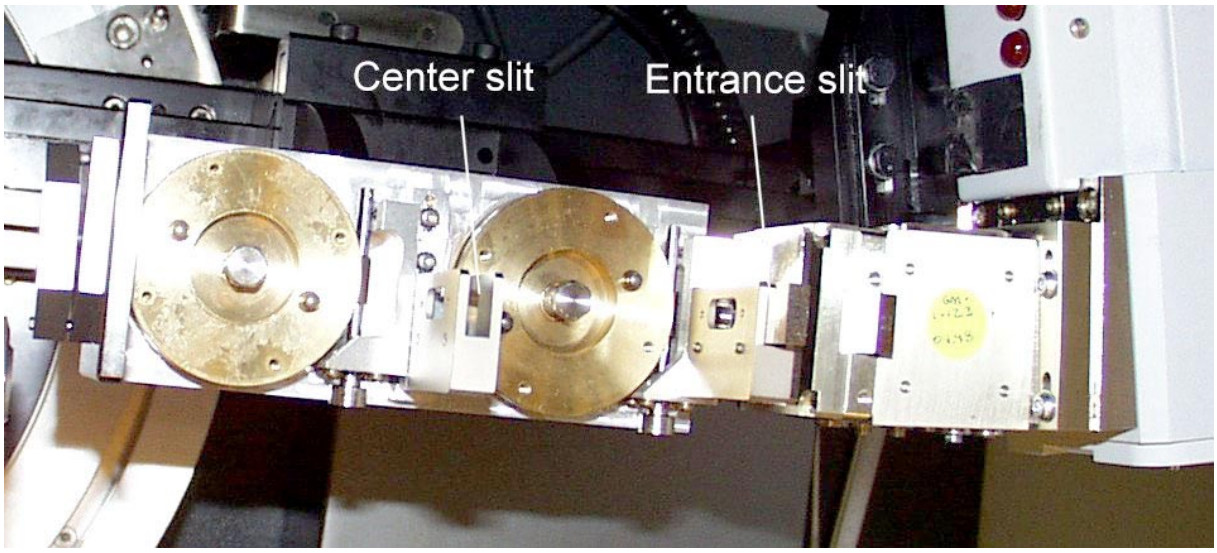


Fig. 6-14: Monochromator without channel cuts. Alignment of entrance slit

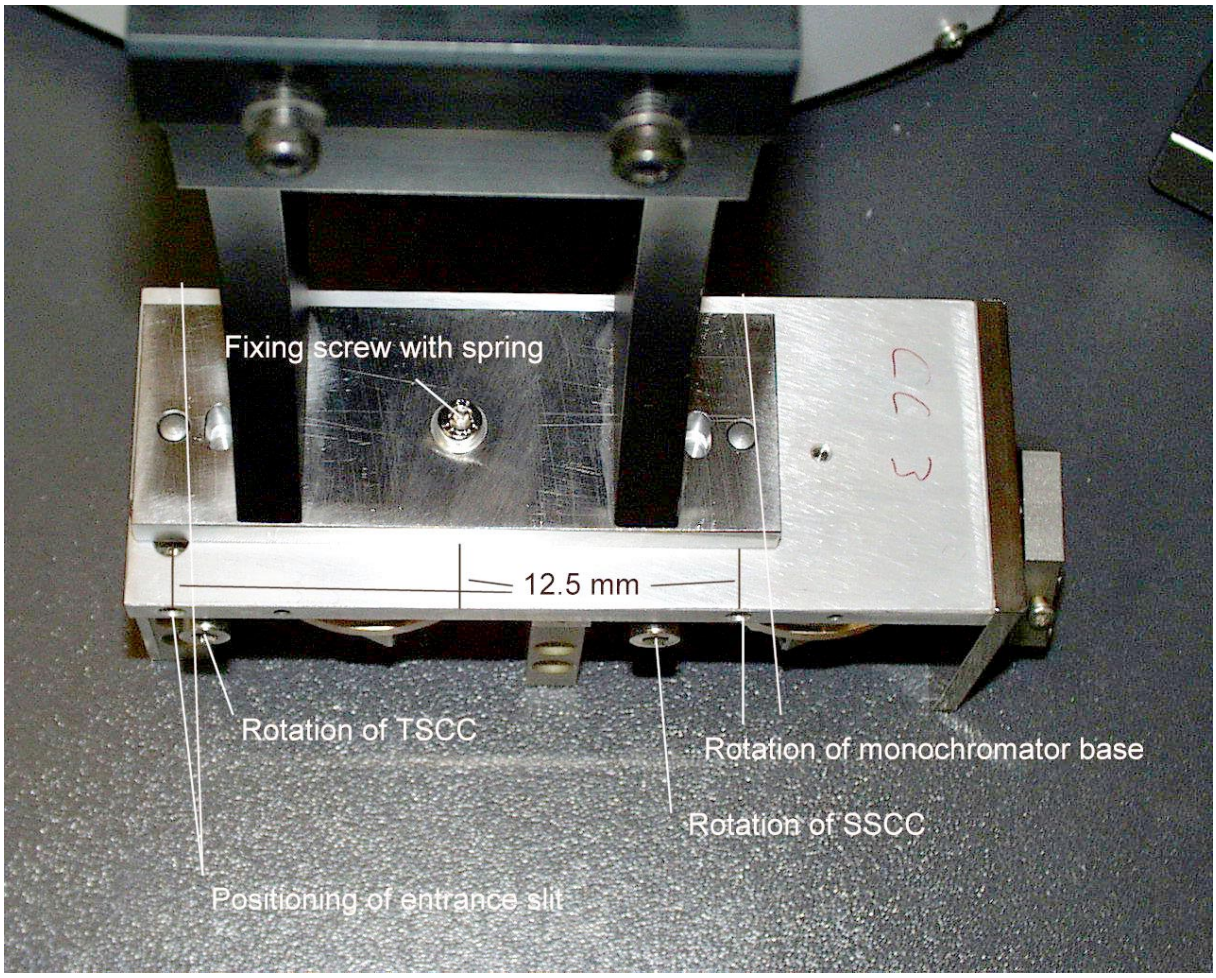


Fig. 6-15: Bottom view of monochromator . Distance base to edge should be about 12.5 mm

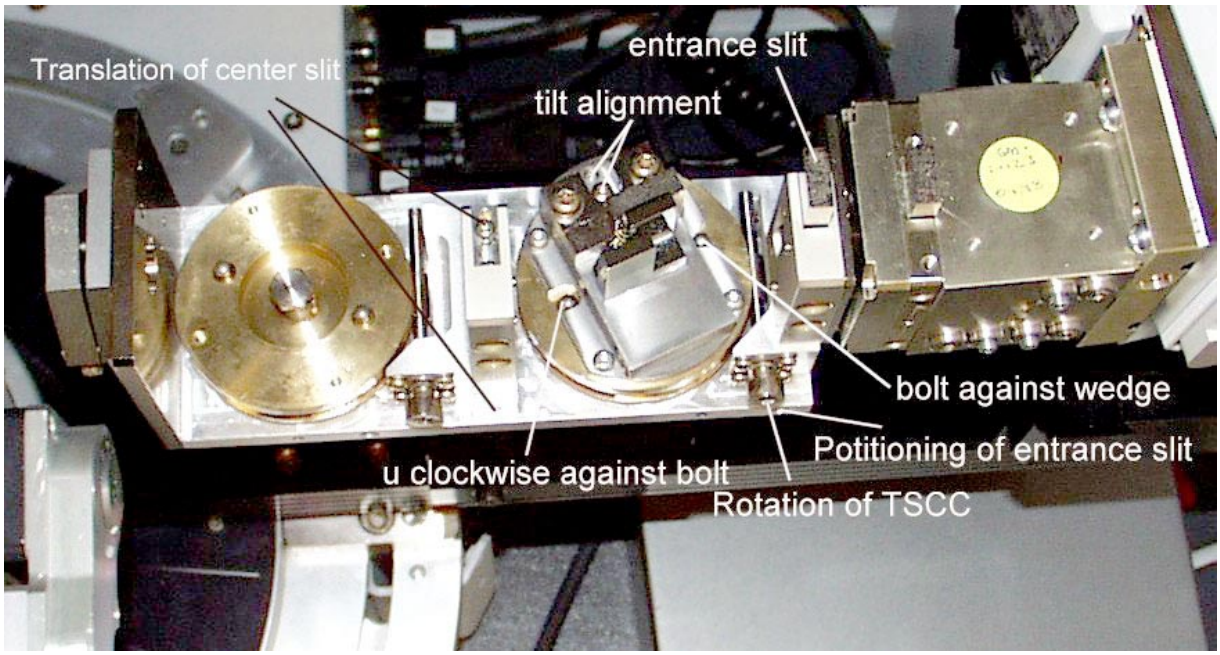


Fig. 6-16: Monochromator with one crystal mounted (TSCC)

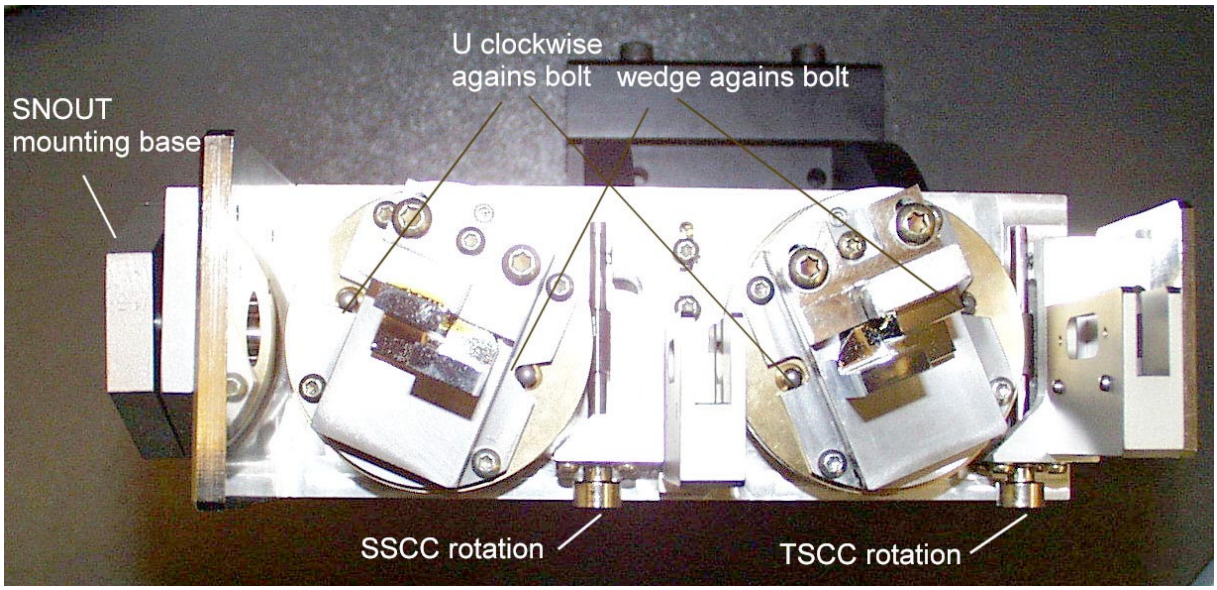


Fig. 6-17: Top view of monochromator after alignment

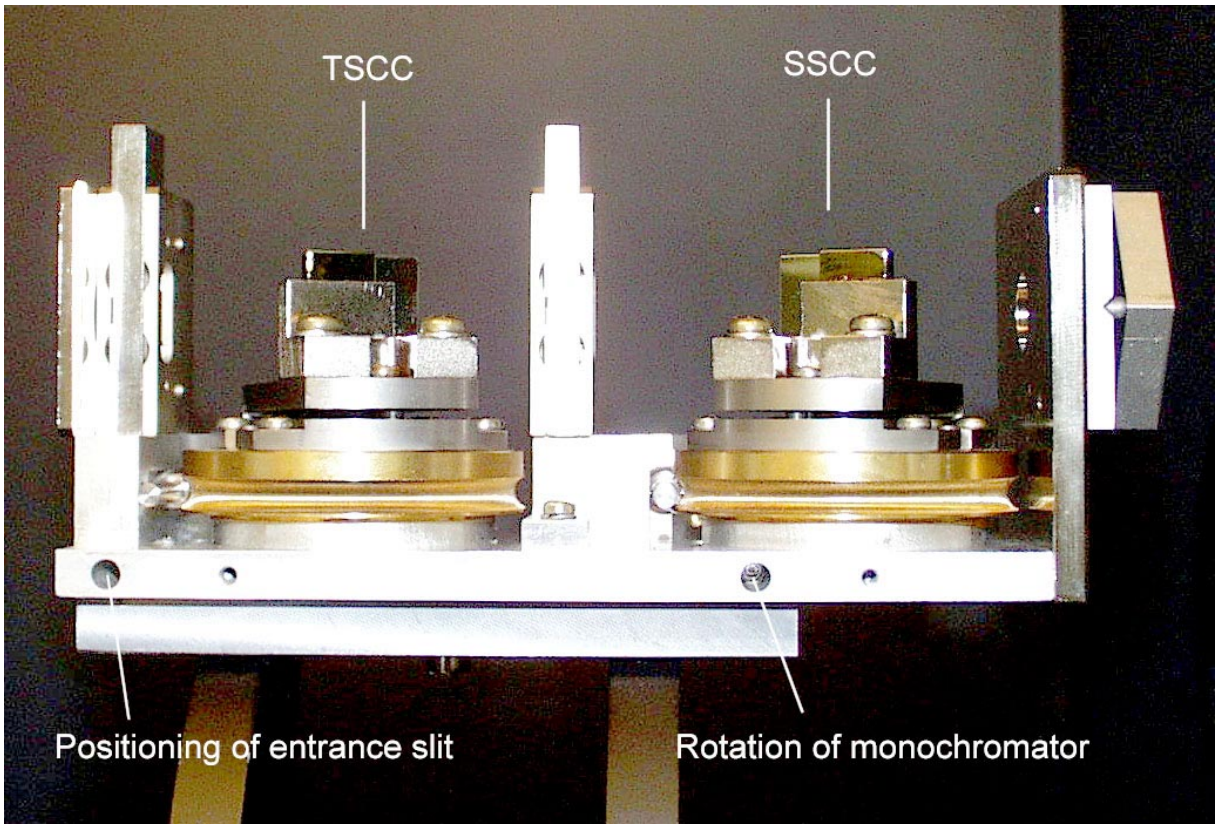


Fig. 6-18: Back view of monochromator after alignment

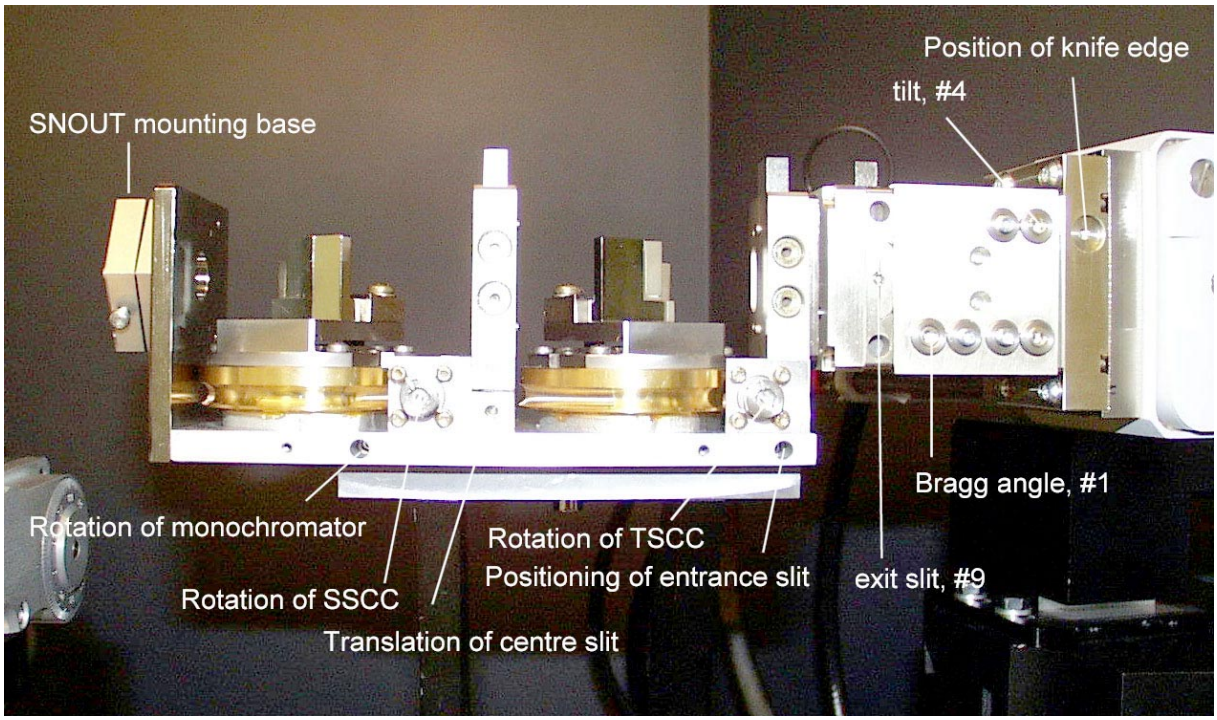


Fig. 6-19: Front view of monochromator after alignment

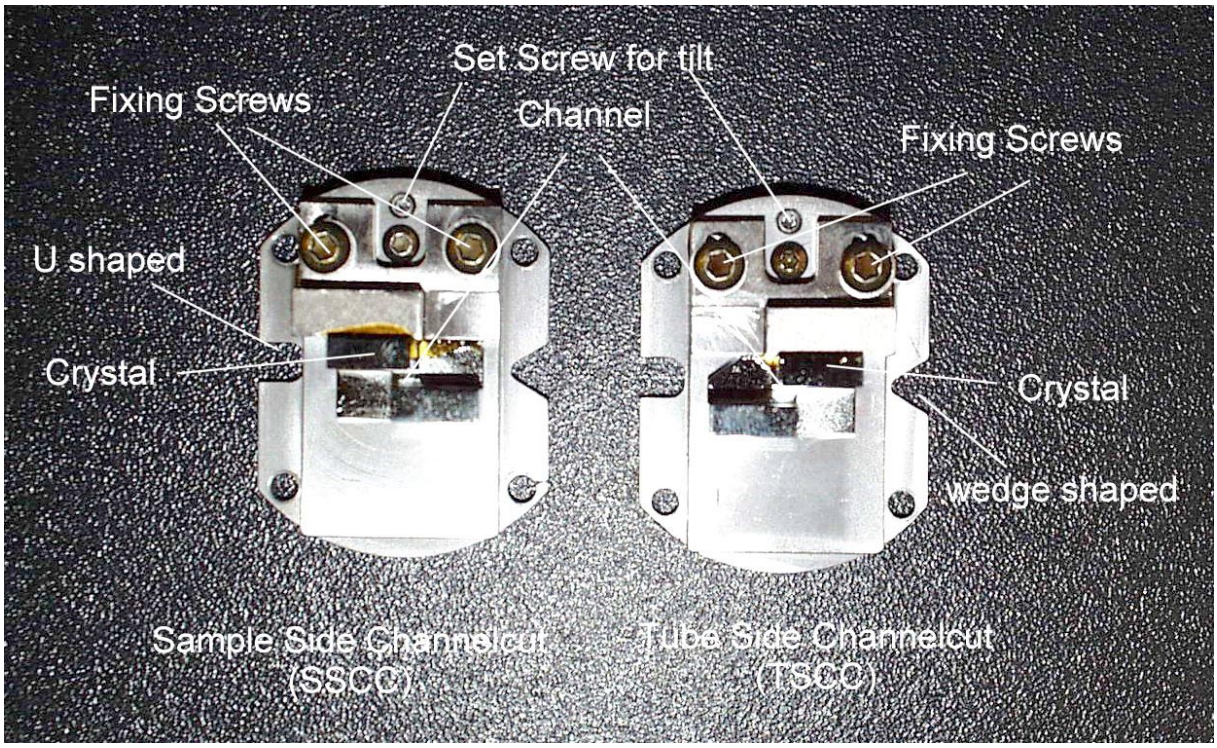


Fig. 6-20: Channel cut crystals

5. Put monochromator housing parallel to track.
Measure at position of screws and rotate the whole monochromator until the base is parallel to the spacer within 0.1 mm

Hint:

Distance is typical approximately 12.5 mm (fig. 6-15).

6. Remount the second channel cut crystal for checking the translation (SSCC = sample side channel cut, fig. 6-20)**.**
Therefore, the wedge shaped opening of the tilt table must be pushed against the bolt on the centre slit side of the goniometer base. Then, the tilt table must be rotated clockwise so that the u shaped opening is pushed against the other bolt (fig.6-17, 6-20). The primary beam intensity must be half cut by the second face of the second channel cut crystal within +/- 10% after optimisation of the SSCC rotation. If this is not the case, translate SSCC by using fixing screws (fig. 6-20). Take SSCC out again (including tilt table!).

Remarks: Mark the zero position and choose this zero of the goniometer of SSCC in a way that the 044 reflection is possible.

7. Remount the first channel cut crystal (TSCC = tube side channel cut, fig. 6-20).
Therefore, the wedge shaped opening of the tilt table must be pushed against the bolt on the entrance slit side of the goniometer base. Then, the tilt table must be rotated clockwise so that the u shaped opening is pushed against the other bolt (fig. 6-16, 6-20). The primary beam intensity must be half cut by the first face of the channel cut crystal within +/- 10% after optimisation of the SSCC rotation. If this is not the case, translate TSCC by using fixing screws (fig. 6-20).

Remarks: Mark the zero position and choose this zero of the goniometer of TSCC in a way that the 044 reflection is possible.

8. Find the reflection of TSCC (first crystal):
Loosen adjusting screws of the TSCC tilt table. Turn by 44 revolutions counter clockwise and mark this position (1 revolution is approximately 0.5°, Ge022 reflection, asymmetrical channel cut 10 turns, 044 channel cut 106 turns), translate centre slit so that the mirror image of a 1mm entrance slit can be seen in the crystal if a 2 mm centre slit is mounted (without centre slit roughly aligned only background and/or no Bragg reflection will be seen). The slit translation base should be roughly 6 mm from the back of the monochromator base (symmetrical Ge022). Be aware that the beam is shifted by the TSCC towards the box window by about 4.6 mm (symmetrical Ge022) with respect to the Goebel mirror beam. Therefore the TSCC beam is not going through the centre of the exit opening of the monochromator but 4.6 mm closer to the box window! Perform a detector scan –2° ... 2°, step size 0.05°, without absorber, 2 mm centre slit of monochromator. There should already be a reflection at about 1° (0.5° for asymmetric Ge022).

Optimise by rotating TSCC. Intensity should be approx. 5000 cps with 0.2 mm Cu (symmetric Ge022), compare table 6-2, fig. 6-21.

9. Optimise position of centre slit.
No intensity loss should be visible with 1 mm centre slit.

Optimisation is easier by using a 0.6 mm slit. Approximate position is about 6 mm from the edge of the base.

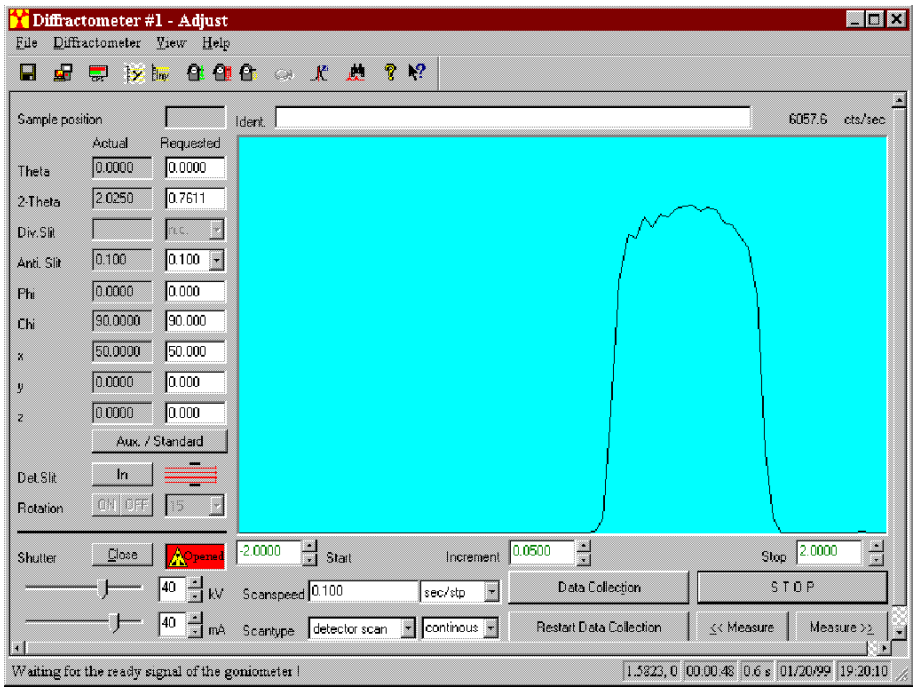


Fig. 6-21: Primary beam behind TSCC with 0.2 mm Cu absorber

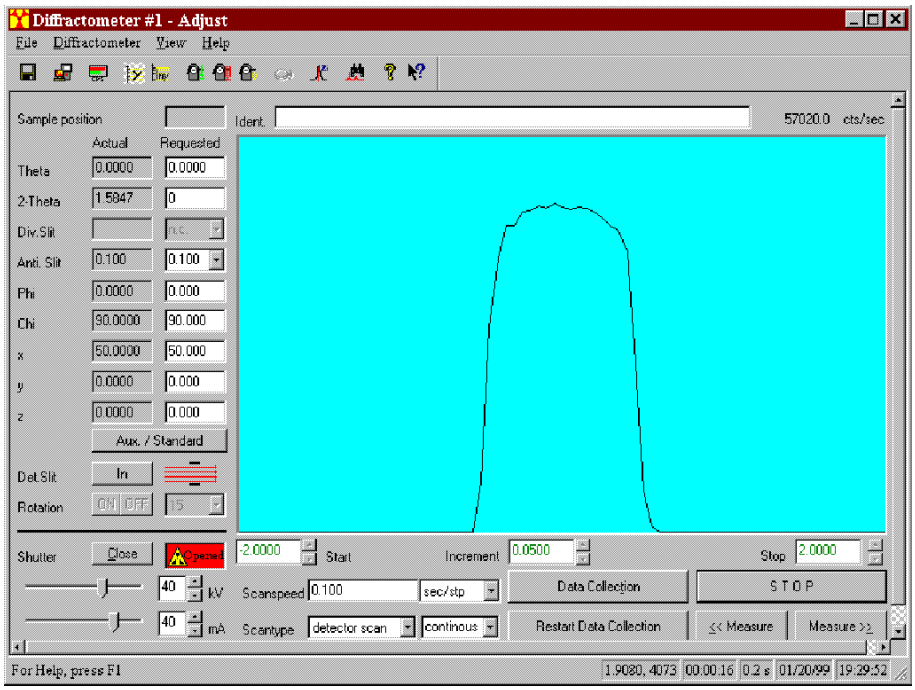


Fig. 6-22: Primary beam behind SSCC with 0.1 mm Cu absorber (4bounce monochromator complete)

10. Mount second channelcut crystal (SSCC = sample side channelcut)**.**
Follow the same procedure as for TSCC. Loosen adjusting screws of tilt table.

Rotate SSCC by 44 revolutions clockwise (symmetrical Ge022).

Perform a detector scan –2° ... 2°, step 0.05° without absorber. There should be a peak at 0°. Optimise intensity. Intensity should be at least 45 000 cps with 0.1 mm Cu (symmetric Ge022). Compare table 6-2.

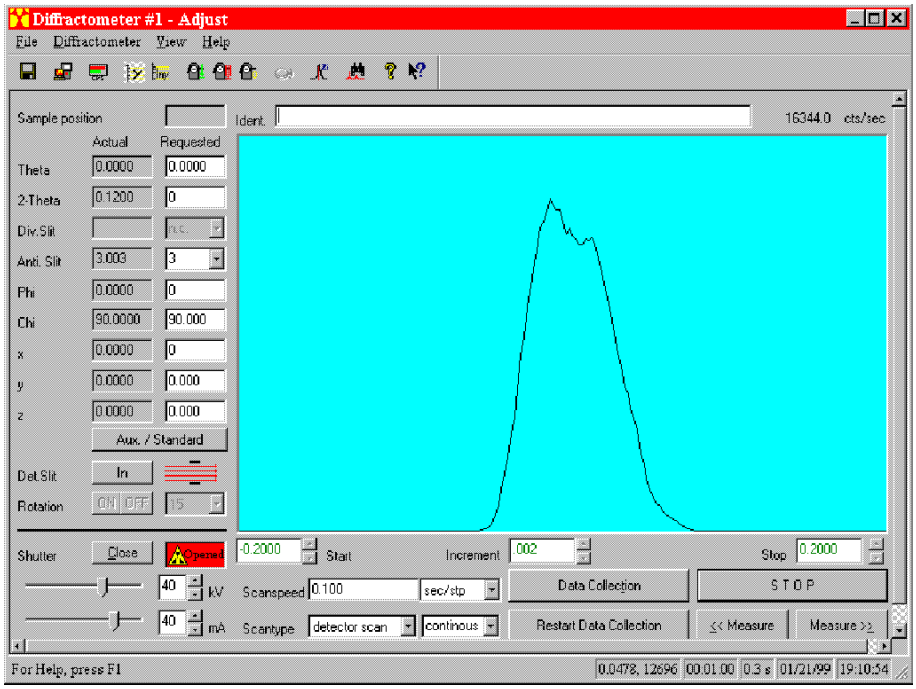


Fig. 6-23: 2theta scan of primary beam with 0.1 mm detector slit and 0.1 mm Cu absorber

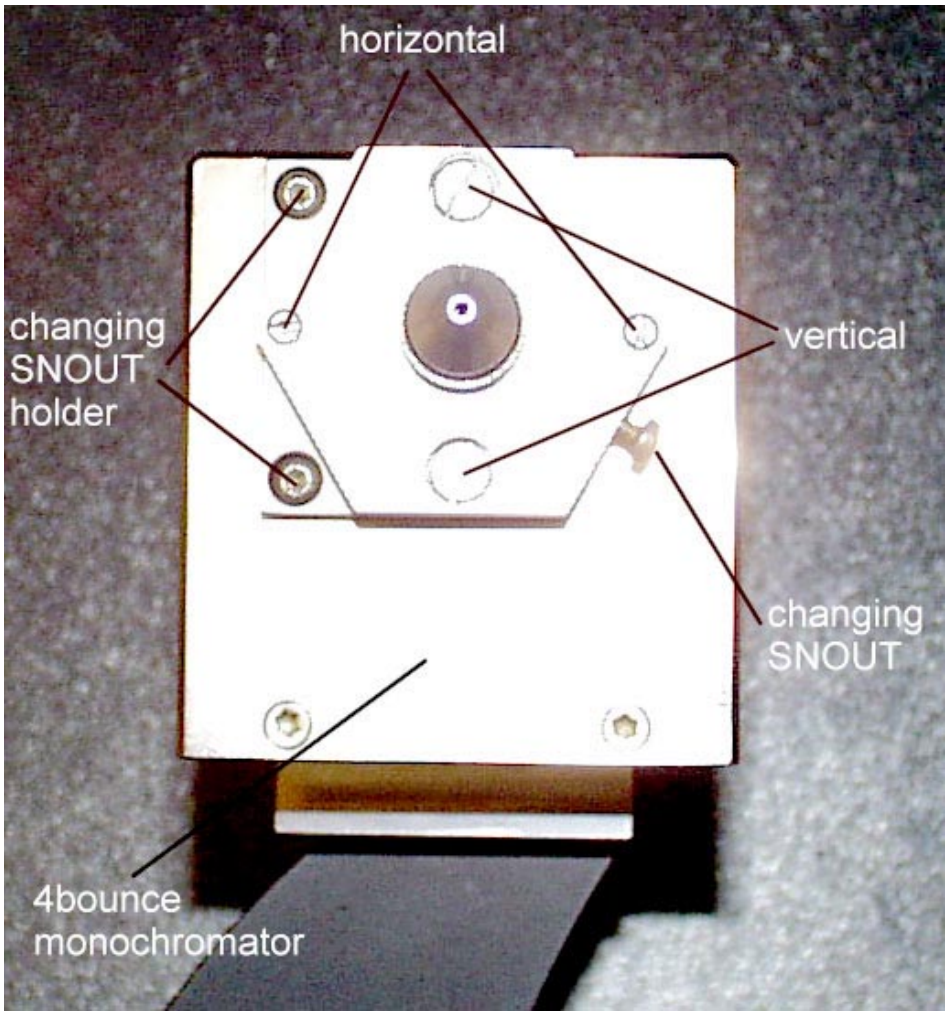


Fig. 6-24: Front view of short 1mm pinhole mounted to monochromator

11. Check the resolution with Si 220 reference sample.
At chi=90° mount sample on vacuum stage (start pump and open adequate valves – compare D8 DISCOVER reflectometry section) and determine correct z value with the help of the micrometer dial (it should show 0.00 mm). Measure 022 reflection (Rocking curve, 2theta= 47.3°, theta = 23.65°, equivalent to plus set up) and optimise chi of the sample to highest intensity. Intensity should be at least 20 000 cps with 0.1 Cu. More important: FWHM of the 022 reflection must be 0.0033° = 12" (<0.0035°) (for the symmetric Ge022 monochromator). Instead of Si 022 Si 111 can be used. The thickness of the sample must be larger than 2 mm to avoid bending effects, fig. 6-25.

12. Remount radiation safety interlock!
By this define the standard operation position of tube and monochromator on the track. Fix the limit switch positions and check that they are working correctly! Standard settings are listed in table 6-1. Change the software limits to slightly harder conditions (approx. 0.1°...1° or 0.1 ... 1mm). Check all drives for collision possibilities!

Tab. 6-1: Drive limits.

Drive	Lower Limit	Upper limit
Theta	-5°	185°
2Theta	-95°	165°
Chi	-4°	94°
Phi	Standard none, -10° if knife edge mounted	Standard none, 10° if knife edge mounted
X	-75.5 mm	75.5 mm
Y	-75.5 mm	75.5 mm
Z	-1.2 mm	12 mm

13. Check parallelism of goniometer axis with the line focus.
Measure the same 022 Si reflection at 2theta = -47.3° (theta = 156.35°, equivalent to minus set up). Determine again the sample chi with highest intensity and smallest width. If the optimum chi setting is the same for both reflections, the alignment is okay, fig. 6-26.

Otherwise the tilt of channel cuts has to be changed. Chi should be equal within < 0.1°.

Note:
Normally the absolute angle of chi will not be correct and the sample will have a miss cut. Therefore the optimum chi is not necessarily 0°!

If the chi settings are not equal, the tilt of the beam can be optimised according the following cases until the optimum chi setting of the reference sample is the same within 0.1° for 2theta=+47.3° and 2theta=-47.3°.

Possible cases:

A. Clockwise rotation of set screw of the SSCC tilt table leads to increase of optimum value of chi(-47.3°) of the reference sample.

B. Counter clockwise rotation of set screw of the SSCC tilt table leads to decrease of optimum value of chi(-47.3°) of the reference sample.

C. Clockwise rotation of set screw of the TSCC leads to decrease of optimum value of chi(+47.3°) of the reference sample.

D. Counter clockwise rotation of set screw of the TSCC leads to increase of optimum value of chi(+47.3°) of the reference sample.

☞ **Note:**

Of course the second tilt screw (torx) must be rotated accordingly! The set screw is just listed in the cases A to D to define the rotation sense in a unique way.

A 1/4 turn of a tilt table screw leads to about 0.15° change in optimum chi setting of the sample.

If a 011 Si wafer is not available, a Si 111 wafer, thickness larger than 2 mm can be used. Instead of 022 and 044 the 111, 222 (very weak), 333, and 444 reflections must then be used.

14. Optimise the intensity

Follow description in "Standard operation of the optics". Recheck that the chi setting is the same for Si 022 (plus and minus set up) and Si 044 (plus set up).

15. Absolute setting of Chi

The absolute setting of Chi can be determined by aligning the Chi setting of the 022 Si reflection at 2theta=+47.3° for Phi=0° and Phi=180°. The average for both Phi positions must be 0.0° within 0.05°. Otherwise, the Zi value of Chi must be redefined accordingly: $Zi_{new} = Zi_{old} – 0.5*(chi(Phi=0°) + Chi(Phi=180°))$. Therefore, start direct command mode and type: SU, $Zi4,Zi_{new}$, QU

Example:

At Phi = 0° Chi = -0.47°, at Phi=180° Chi = -0.22°, and Zi_{old} = 91°. The absolute zero of chi is then (-0.47° - 0.22°)/2 = -0.345°. For resetting Chi = -0.345° to zero, start direct command mode and type: SU Enter Zi4,91.345 Enter QU Enter

Recheck absolute setting of chi and change Zi value of chi inside configuration program.

16. Check the wavelength of the monochromator settings,
especially confirm that Cu K α_1 has been aligned. Therefore take the theta positions of a reflection which you have measured in plus and minus setup.

$\lambda = 2*d*\sin(0.5*(\theta_{plus} + 180 - \theta_{minus})) / \sqrt{(h^2+k^2+l^2)}$, d_{Si}= 0.5431 nm

I.e. for Si 022: $\lambda = 2*0.5431*\sin(0.5*(23.65+180-156.35)) /\sqrt{8}$ nm

The result should be λ(Cu K α_1) = 0.15406 +/- 0.00005 nm at 22°C. Otherwise the theta alignment of TSCC and RSCC has to be repeated.

17. For documentation purposes safe:

- Si 022 rocking curve reflection +/- 0.02 °, step size 0.0001 °, 0.1mm Cu absorber, alternatively Si 111 is possible (220-Resolution-xxxx.raw, xxxx: Fabrication Number)

- Detector scan through primary beam, standard slit system without slits, 600mm circle, 0.1 mm Cu absorber, without sample (e.g. x=-70), 0 ° +/- 1 °, step size 0.05 °. (220-Intensity-xxxx.raw)

- Detector scan through primary beam, standard slit system with 0.1 mm detector slit, 600mm circle, 0.1 mm Cu absorber, without sample (e.g. x=-70), 0 ° +/- 0.3 °, step size 0.004 °. (220-Width-xxxx.raw)

- Repeat the three scans without channelcut monochromator: Goebel-Resolution-xxxx.raw, Goebel-Intensity-xxxx.raw, Goebel-Width-xxxx.raw

- In case of additional channelcut monchromators perform additional scans.

- The zero beam position of the different primary beams (Goebel mirror, different channelcut monochromators) must be equal within 0.05°. If this is not the case, check if the channel width of the crystal pairs is equal within 0.1 mm. If the difference is larger, one of the crystals should be replaced by a better fitting one. A little improvement (up to 0.01° ... 0.02°) is possible by not adjusting the TSCC to the centre part of the Goebel mirror beam.

18. Specification values
40 kV, 40 mA, KFLCu2K, less than 500 h in use, detector slit at 600mm diameter measurement circle and tube at end of track, warm up time 0.5 h.

Tab. 6-2: Specification values

	Base width (mm)	**Minimum I0 (cps)**	**Maximum FWHM Si022 (°)**	**Minimum I0 TSCC (cps)**
Goebel mirror type 1	0.6 ... 0.8	$1.7*10^9$	0.07	-
Goebel mirror type 1+ Ge022 symmetric	0.6 ... 0.8	$4.50*10^6$	0.0035	50*106
Goebel mirror type 1+ Ge044 symmetric	0.6 ... 0.8	$150*10^3$	0.0015	10*106
Goebel mirror type 1+ Ge022 asymmetric	0.6 ... 0.8	$18*10^6$	0.008	150*106
Ge022symmetric Point focus	1.2 +/- 0.1	$0.75*10^6$	0.0035	7.5*106
Ge044 symmetric Point focus	1.2 +/- 0.1	$25*10^3$	0.0015	0.6*106
Ge022 asymmetric Point focus	1.2 +/- 0.1	$3*10^6$	0.008	20*106

Tab. 6-3: Influence of vertical divergence (dominant!) and air absorption on intensity change with distance.

	Minimum I_0 (cps) at optic exit
Goebel mirror (type 1) + Ge022 symmetric	$10*10^6$

19. Reproducibility test for theta circle.
Use Si 011 or Si 111 wafer with thickness larger than 2 mm. Vacuum sample stage must be used. In case of Si 011 determine the optimum chi and omega settings for 022, 044 , 022 in minus setup. Define the average value. Measure these three reflections and repeat this 6 times, Bragg position +/- 0.02 °, step size 0.0001 °, time per step 0.1 sec. Take only the 5 last ones for evaluation because of warming up effects in the goniometer gear. The room temperature must be stable within 1°C. The system must be at operating power for at least 30 min. For all three reflections the peak positions is not allowed to differ more than 0.0004°.

(Filenames: Repro-theta-022plus-xxx-z, Repro-theta-022minus-xxx-z, Repro-theta-044plus-xxx-z, xxxx = Fab. No., z = number of repetition, fig. 6-31, 6-32, 6-33)

20. Reproducibility test for 2theta circle,
only in case of systems with analyser crystal.

Insert the analyser crystal. Perform detector scans through Si 022 (plus set up) and Si 044 (plus setup). Repeat the measurement of the two reflection 6 times. Take only the 5 last ones for evaluation because of warming up effects in the goniometer gear. For all three reflections the peak positions is not allowed to differ more than 0.0004°.

(Filenames: Repro-2theta-022plus-xxx-z, Repro-2theta-022minus-xxx-z, Repro-2theta-044plus-xxx-z, xxxx = Fab. No., z = number of repetition, fig. 6-34, 6-35)

21. Check the long term stability of the intensity:
The intensity of the primary beam of each channelcut monochromator after running at operating power at least for 30 min must be the same within 10% after 24 h without optimisation. Repeat after 24 h:

Detector scan through primary beam, standard slit system without slits, 600mm circle, 0.1 mm Cu absorber, without sample (e.g. x=-70), 0 ° +/- 1 °, step size 0.05 °. (220-Intensity-after24h-xxxx.raw).

22. Changing of optics.
The intensity should not differ by more than 10% after remounting a monochromator. This should be checked by reinserting each monochromator 3 times.

23. User documentation.
With each monochromator the test field values of resolution, beam position and integral intensity must be delivered.

24. Alignment of pinholes

- Activate the ratemeter in the primary beam of the monochromator
- After mounting the pinhole, perform the horizontal alignment. Loosen one of the horizontal alignment screws and fasten the

other one in the direction of higher intensity, fig. 6-17, 6-19, fig. 6-24. Optimise until more than 10% of the intensity without pinhole is reached (standard accessory C79298-A3158-B153: short type and 1mm diameter).

- At Chi = 0 ° (vertical) perform x-scan with pinhole to determine the vertical position of the peak. Loosen and fasten the vertical pair of alignment screws until the peak maximum is at 0 +/- 0.1 mm, fig. 6-17, 6-19, 6-24.

- Following modifications of the pinholes are available:
  short pinholes (20 mm): 0.3 mm (C79298-A3158-B151), 0.5 mm (C79298-A3158-B152), 1.0 mm (C79298-A3158-B153)
  long pinholes (60 mm): 0.3 mm (C79298-A3158-B154), 0.5 mm(C79298-A3158-B155), 1.0 mm (C79298-A3158-B156)
  short slit (20 mm), free rotation, height 8 mm, width adjustable 0.1 ... 1.0 mm: (C79298-A3158-B157)
  long slit (60 mm), free rotation, height 8 mm, width adjustable 0.1 ... 1.0 mm: (C79298-A3158-B158)
  illumination slit, height 2 ... 8 mm, height approx. 1 ... 6 mm: (C79298-A3161-B35)

  All of these slits and pinholes might be fitted directly to the holder. Precise manufacturing of the hole position can only be achieved within 0.1 mm. Therefore either realignment of the base might be necessary when switching between different pinholes and slits or an alignment base should be ordered for each slit and pinhole (recommended).

25. Finally all critical screws must be coloured:

- fixing screws of crystals on tilt tables (fig. 6-20)
- tilt screws of tilt table (fig. 6-20)
- translation of centre slit (fig. 6-17, 6-19)
- screw on back side of monochromator (fig. 6-15)
- Rotation of TSCC (fig. 6-16)
- pinhole alignment screws (fig. 6-24)
- translation of tube (DOF), fig.6-3, 6-4
- rotation of tube (DOF) fig. 6-3, 6-5
- out of scattering plane translation of tube (vertical translation) (in case of 3DOF), fig. 6-2
- set screws for translation of entrance slit
- mounting the tube on spacer
- mounting the channel cut monochromator on spacer

26. Examples for the other channelcut types.

Fig. 6-27 and 6-28 show examples for the Ge 044 4bounce monochromator. Fig. 6-27 shows the integral intensity without any slits and absorbers in front of the detector. Fig. 6-28 shows an example of a Goebel mirror with bad bending: The integral intensity and resolution is still okay, but not perfect parabolic bending leads to dark spots in the beam if the beam is scanned with a 0.1 mm detector slit (two minima in the scan). This cannot be seen in Rocking Curve measurements of the samples but will have negative effects in surface mapping.

Fig. 6-29 shows the primary intensity of the asymmetric 4bounce monochromator (0.008° resolution): detector scan with 0.1 mm Cu absorber but without detector slit. Fig. 6-30 shows the typical shape of the asymmetric 4bounce monochromator: detector scan with 0.1 mm detector slit and 0.1 mm Cu absorber.

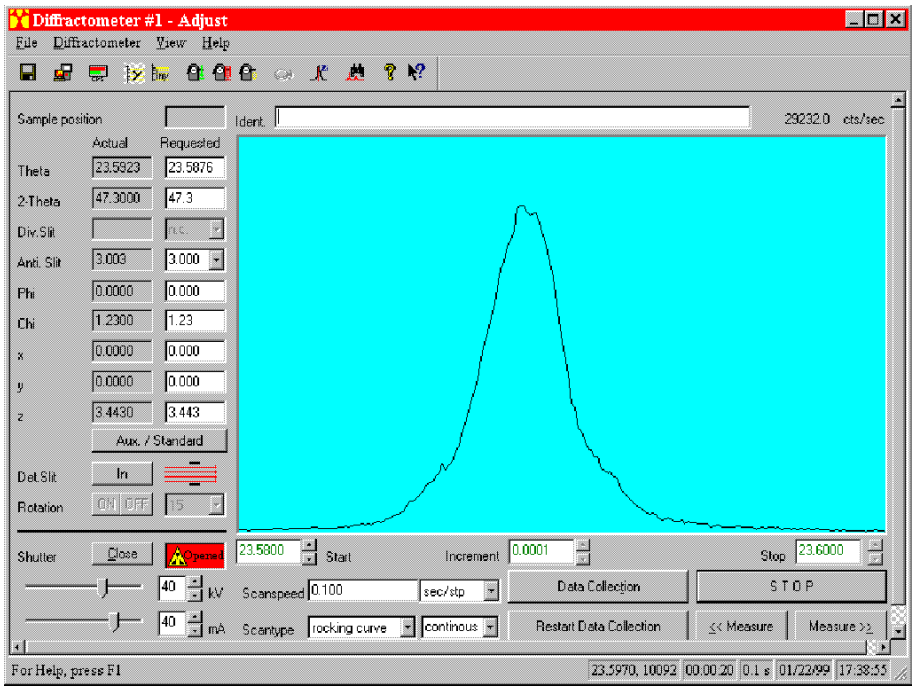


Fig. 6-25: Checking the resolution with Si 220 reference sample

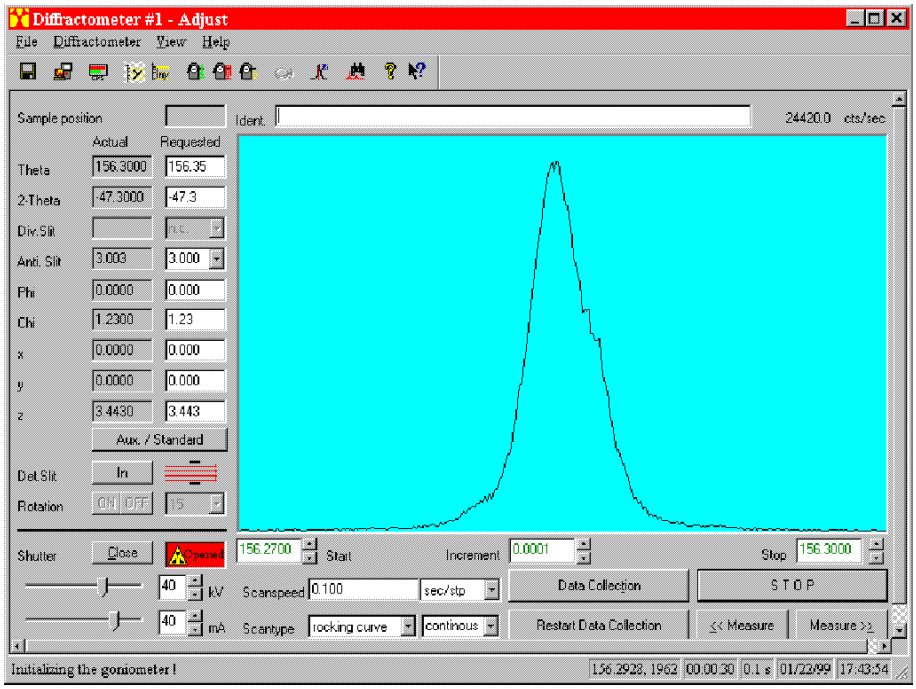


Fig. 6-26: Checking the parallelism of the goniometer axis with the line focus

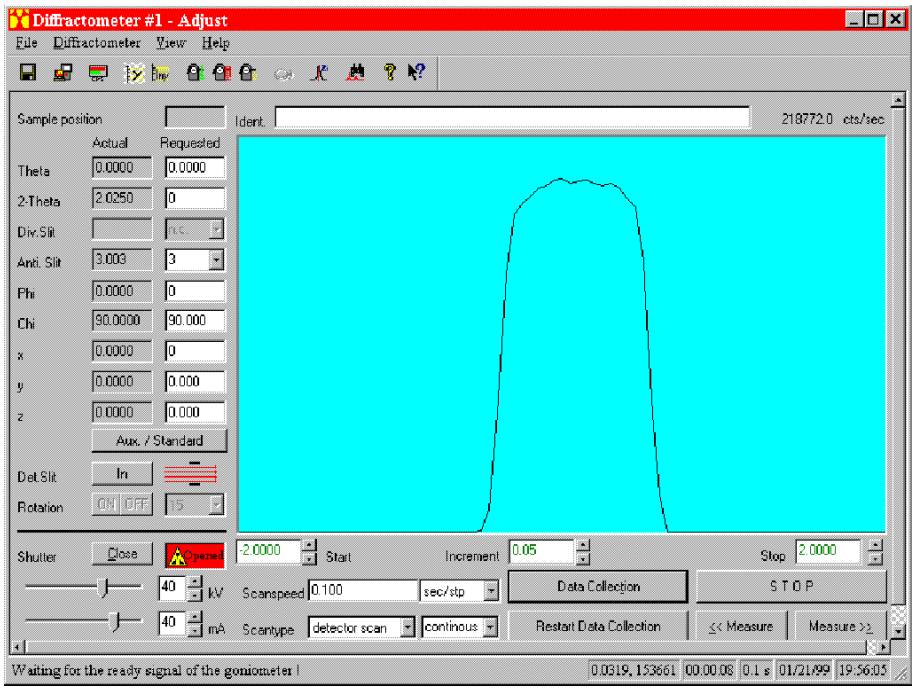


Fig. 6-27: Primary beam of Ge044 monochromator

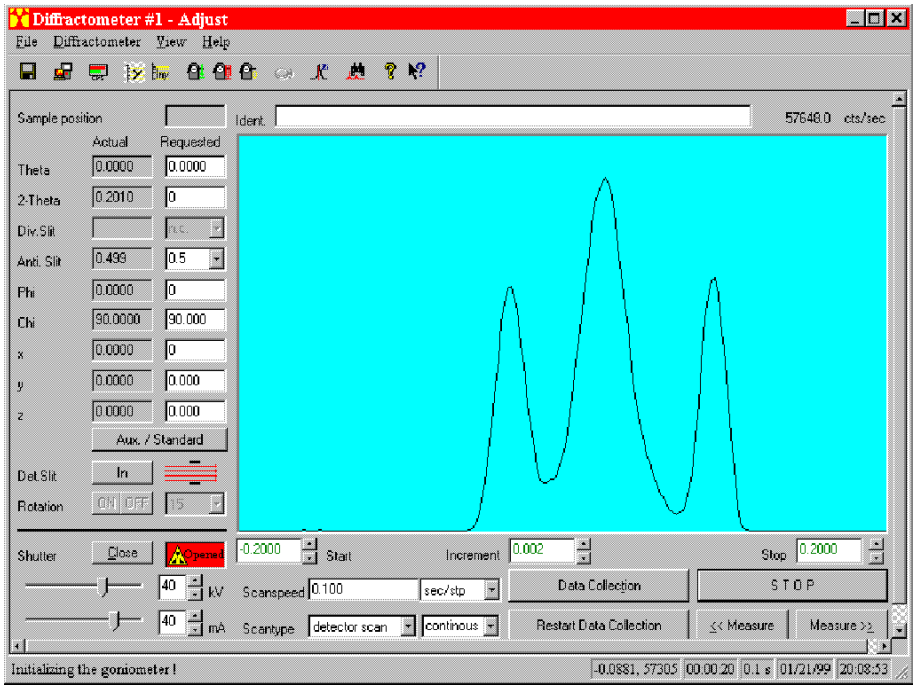


Fig. 6-28: Primary beam of Ge044 monochromator and Goebel mirror with 0.1 mm detector slit (300 mm radius measuring circle)

In this case, there are 3 peaks which stem from incorrect bending of the Goebel mirror. As a result the intensity of the Goebel mirror beam is not constant over its cross section. It does not have an effect on Rocking curve measurements.

Exit slits of the Goebel mirror (0.6 mm or 0.2 mm) can remove the satellite peaks. Such Goebel mirrors should not be delivered!

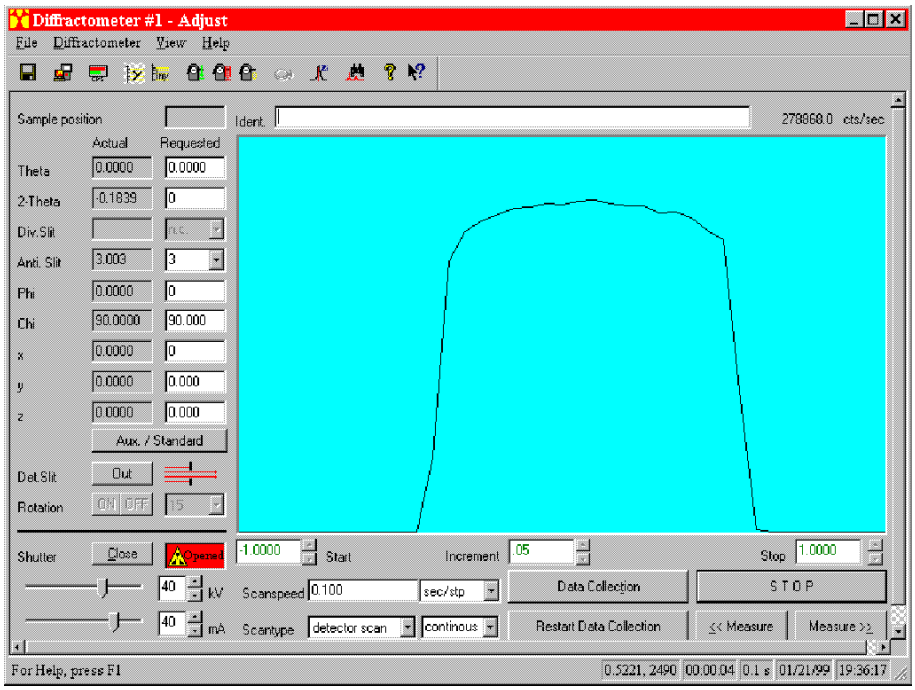


Fig. 6-29: 2theta scan of primary beam of Ge022 asymmetric monochromator with Goebel mirror with 0.1 mm Cu absorber

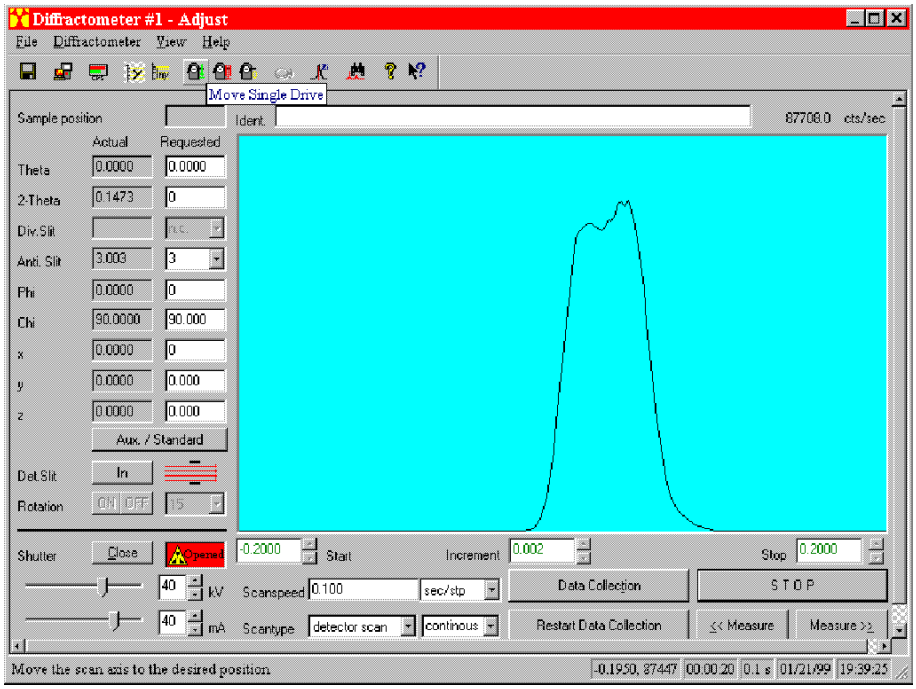


Fig. 6-30: 2theta scan of primary beam of Ge022 asymmetric monochromator and 0.1 mm Cu absorber

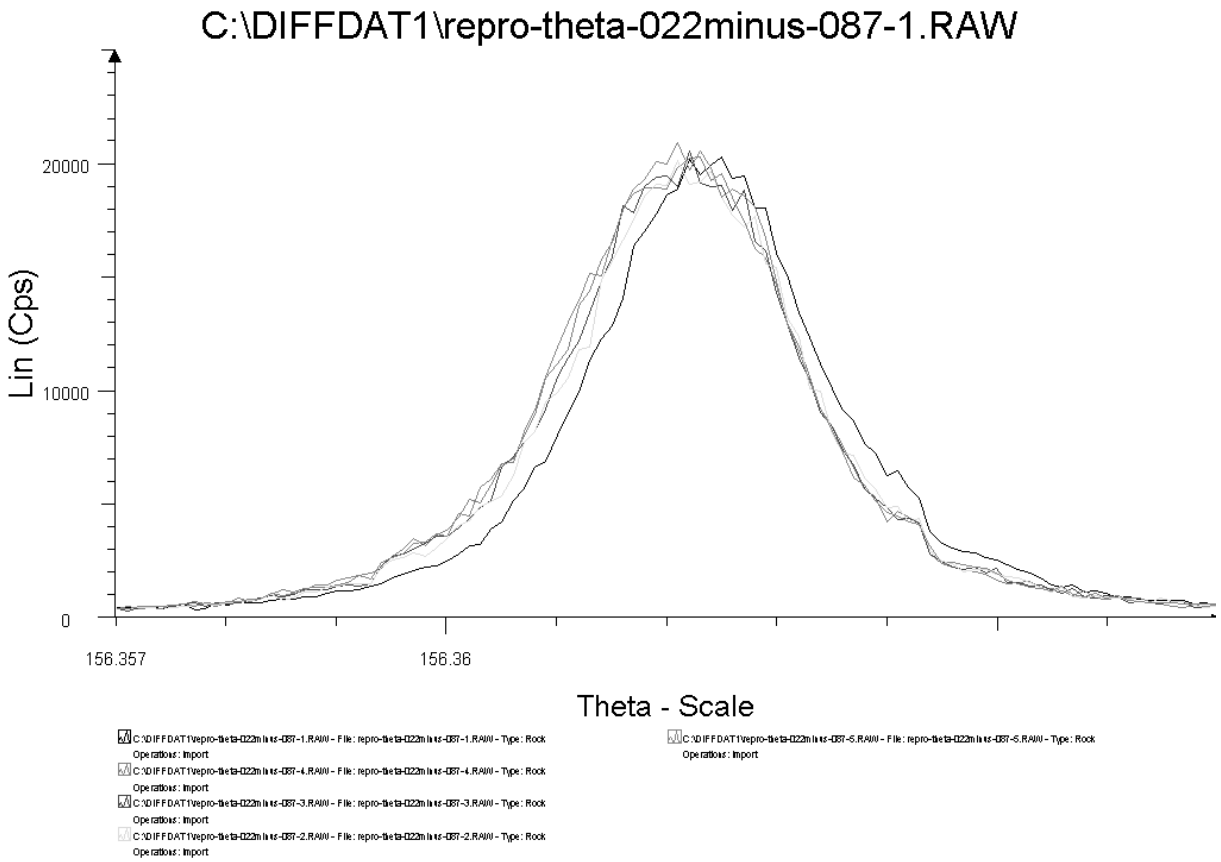


Fig. 6-31: Reproducibility test of theta circle, alternative rocking curves of 022 minus set up

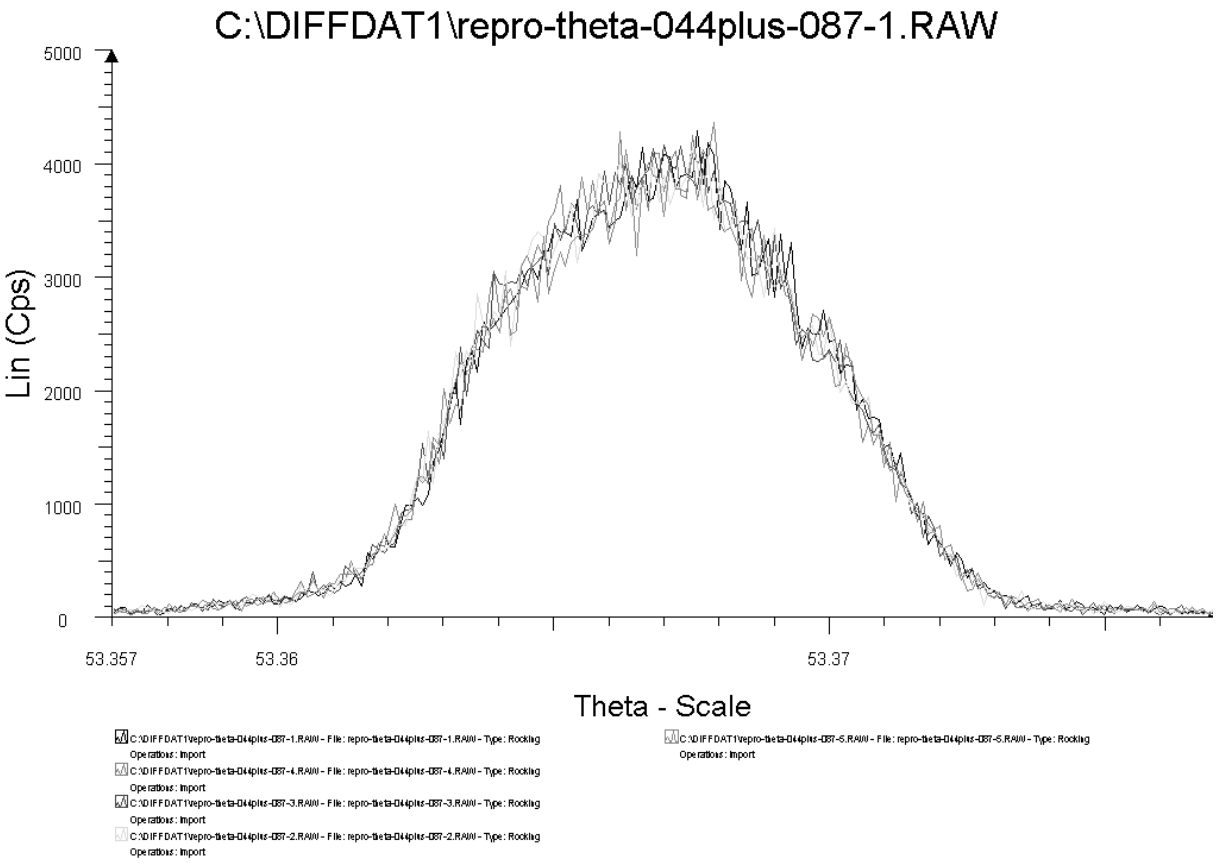


Fig. 6-32: Reproducibility test of theta circle, alternative rocking curves of 044 plus set up (2)

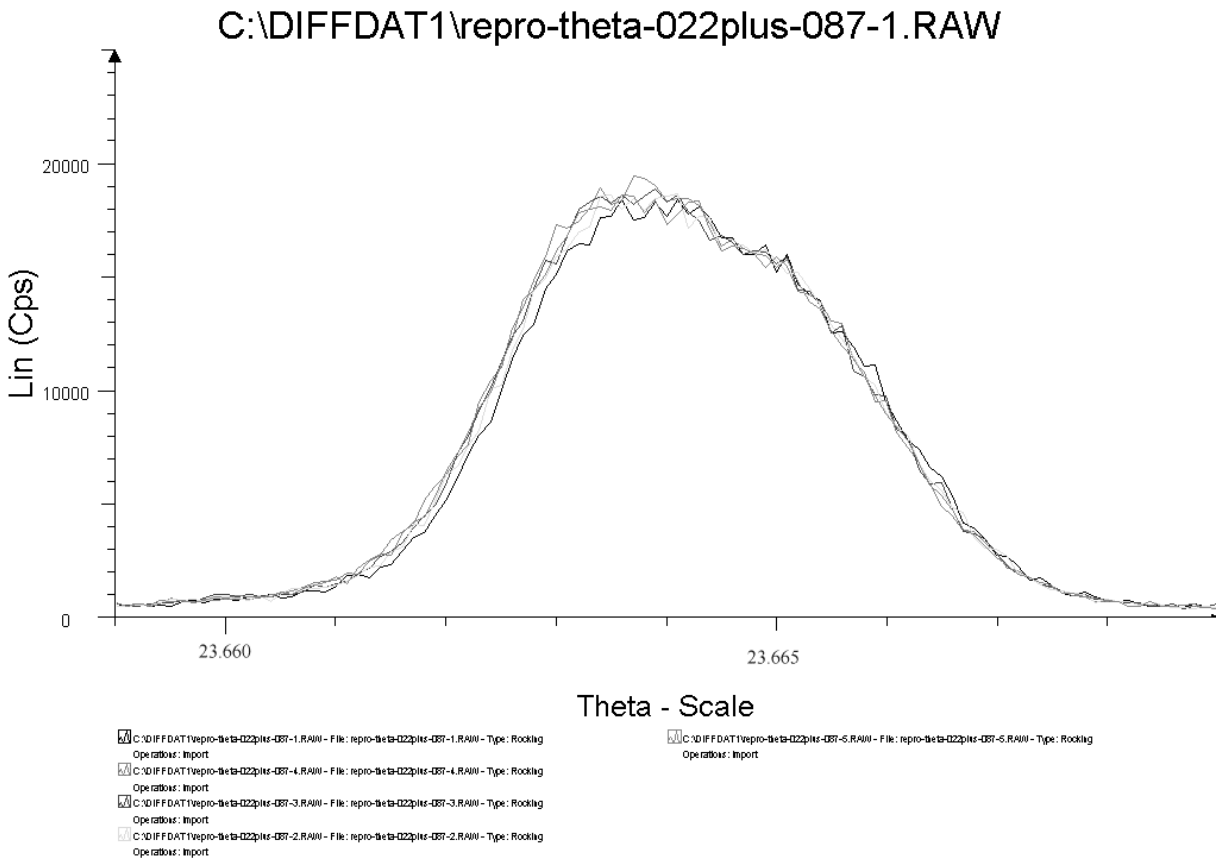


Fig. 6-33: Reproducibility test of theta circle, alternative rocking curves 022 plus set up

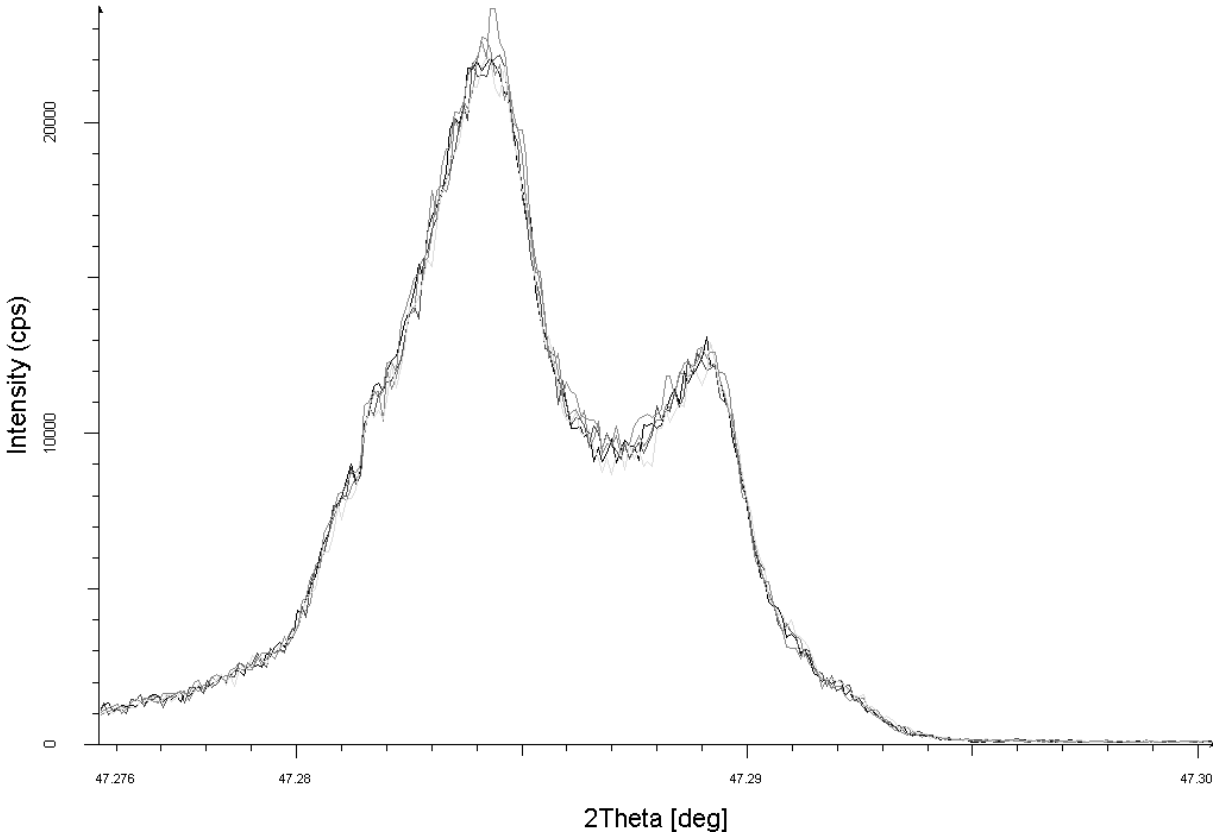


Fig. 6-34: Reproducibility test of 2theta circle, detector scans with analyser crystal, 022 reflection

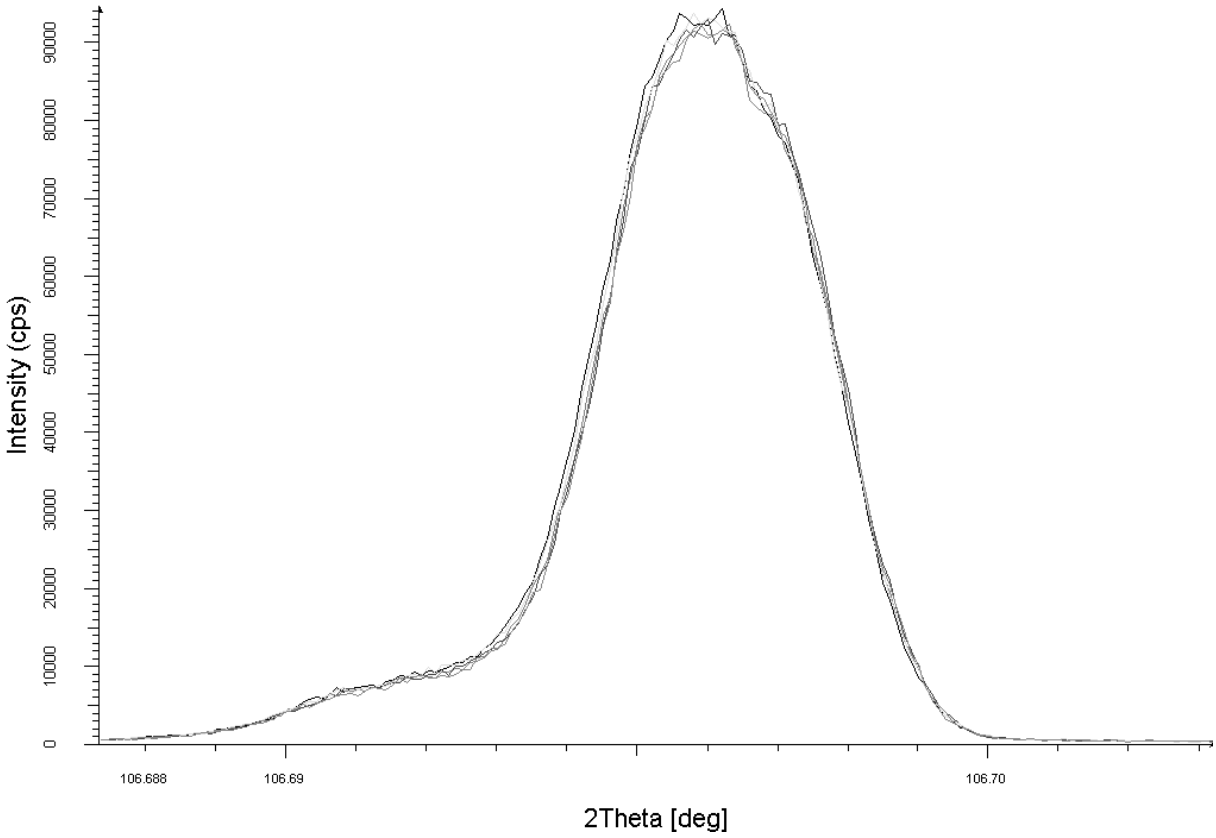


Fig. 6-35: Reproducibility test of 2theta circle, detector scans with analyser crystal, 044 reflection

Standard Operation of the Optics

General Hints

The primary intensity of the different optics should be compared with the factory values regularly (e.g. once a month). For optimum operation of the system the room temperature should be stable within +/-1°C. If high resolution monochromators are used, primary intensities, intensities and positions of sample reflections vary significantly due to changes of the lattice constants, if the temperature changes more than 1°C.

Checking and Optimising

1. All checks must be done at 40 kV and 40 mA and after minimum operation time of 30 min.

2. Before installation of channelcut measure the primary intensity from the Goebel mirror, no tuning!

3. After installation of the monochromator determine the primary intensity, no tuning!

4. If the intensities are equal within 10% compared to the last check, no optimisation is necessary. For factory settings compare table 6-2. Of course some intensity loss is normal due to ageing of the X-ray tube (something in the order of 5% per 1000 h of operation).

5. A. Optimise the parallelism of TSCC and SSCC by tuning SSCC (fig. 6-19, rotation of SSCC).
Use the ratemeter inside the ”Immediate Adjust” program. Move to the direct beam (2theta=0 ° , no sample mounted, do not forget to put an adequate absorber to avoid detector dead time or saturation effects: e.g. 0.1 mm Cu for standard symmetrical 4bounce monochromator, save the actual intensity values with the lower bar). Be careful: one full turn of SSCC knob means about 0.5 °. The reflection width of the SSCC reflection is only 0.0033 °. Be aware of K α_2 which is about 1/8 turn away from K α_1 and has about ½ the intensity of K α_1. Be aware of the mechanics backlash: Always optimise in the same rotation direction. If you have moved too far, turn a full revolution backwards and start again 1/16 turn before the reflection from the same side as before. The precision needed is in the order of the smallest digits of the scale on the monochromator housing. If the intensity is within 10 % of the last known value, you are finished.

B. Optimise parallelism of channelcut monochromator and Goebel mirror beam (fig. 6-19, rotation of monochromator)**.** Therefore the pair of set screws of the monochromator base underneath the SSCC crystal is used (fig. 6-18, 6-19). The function

is analogue to the set screws underneath the slit systems of the D8 - compare ”Prüfung-D8.doc” or according chapter in user’s manual (chapter 3, Alignment of diffractometer). You are still using the rate-meter. Start with fastening the front set screw. Either the intensity has increased or decreased.

In case of increase: Stop if you have reached +/- 10% of the old value. Otherwise loosen the back set screw half a turn and fasten the front set screw in small turns until +/- 10% of the old intensity is reached. Lock the setting with the back set screw.

In case of decrease: Loosen the front set screw half a turn and fasten the back set screw in small turns until +/- 10% of the old intensity is reached. Lock the setting with the front set screw.

C. Optimise Goebel mirror intensity (fig. 6-19: Bragg angle of Goebel mirror, screw #1). With Channelcut monochromator mounted optimise the intensity (rateme-ter!) with tuning screw 1 of the Goebel mirror (Bragg angle of the Goebel mirror).

6. If these optimisation has not been done for some time some iterations of step 8A ... 8C might be necessary.

7. Document the obtained values for future reference.

8. The rotation of TSCC is only needed during factory alignment.

Reflectometry Stage for Eulerian Cradles

The purpose of this modular sample stage is to achieve simple sample fixing without uncontrollable bending and without contamination. It can be used without knife edge for high resolution, texture and stress measurement (fig. 6-36). For reflectivity measurements a knife edge collimator can be easily mounted to control the illuminated sample area at grazing incident angles (fig. 6-45). Without breaking the vacuum the Phi circle of the cradle can rotate infinitely in either direction.

Mounting

First the base plate has to be mounted on the cradle with four screws (fig. 6-38). The stage was mainly designed for the ¼ circle cradle but can also be used on the open cradle with one translation only. Important is the thickness of the whole stage: 30 mm. The distance between mounting base on the cradle and the beam must be larger. Two spacer plates are delivered with the stage to increase the thickness of the stage (fig. 6-42). For the ¼ circle cradle these are not needed. The ring has to be connected with the vacuum pipe (Fig. 6-39) and the vacuum pump (fig. 6-40). The vacuum pump must be connected to the multiple socket which should be placed inside the D8 box (fig. 6-40). The switch on this socket can be used to switch off the vacuum pump (e.g. for changing the samples). An O-ring has to be placed on top and below the ring (fig. 6-39). The pipe has to be fixed with a guiding clamp to the chi circle (fig. 6-36, 6-37) and must be lead parallel to the motor cables of the cradle above the guiding spring (fig. 6-41).

The chuck (3” – fig. 6-37 or 5” – 6-36) is fixed with three 3mm allen screws to the base (fig. 6-44). Be sure that the chuck is fixed securely.

If no reflectivity measurements are planned, the mounting base of the knife edge should not be mounted (fig. 6-42, fig. 6-37). This mounting base restricts the allowed range of the translations. Due to the planned application the limits (hardware and software) of phi or x and y must be restricted to avoid collisions. The user must determine the restriction which suits best to his requests.

The knife edge should be mounted with the mounting base pointing to the ceiling of the box at Phi=0°, Chi=0° (6-37, 6-45). Phi should be restricted to +/- 10° with hardware limits and software limits. This is no restriction because the knife edge must be normal to the incident beam to meet it’s purpose.

The protective cover should be mounted on the knife edge if it is not in use ! (fig. 6-44)

Alignment of samples

For further reference on reflectometry please also read chapter 7 of the D8 manual.

Conditions for alignment

- System at operating power for at least 0.5 h (normally 40 kV, 40 mA) to stabilise position of tube focus
- Alignment with „adjust“ program
- Primary beam is at 2theta = 0°
- Primary beam is going through centre of goniometer (glass slit, chapter 3 of D8 manual)
- Primary beam is reduced to 0.2 mm width with 0.2 mm slit (e.g. as exit slit of Goebel mirror)
- Primary intensity reduced to below 300 kcps with adequate number of 0.1 mm Cu absorbers
- 0.1 mm detector slit
- 0.2 mm antiscatter slit
- knife edge moved out of beam (e.g. top most position, compare fig. 6-44)
- sample mounted symmetrical underneath knife edge. The air holes are placed on circles around the centre of the chuck. The holes belonging to different diameters can be opened by independent valves. The diameter is marked near to the valves. The open position is defined on the chuck. Open the valves which fit best to the sample to be measured. Switch on pump. (compare fig. 6-43, 6-40).
- x = y = 0 mm
- Phi=0°

Alignment of sample

1. Perform z scan –1 mm to 12 mm in 0.1 mm steps (can be reduced if thickness of sample is known) at theta =0°, 2theta=0°, Chi=0°. Repeat scan with 0.01 mm step size in a range of the z position with 50% level of primary beam intensity. Double click on this z position to update the requested z position. To first approximation the surface of the sample is now in the middle of the primary beam and in the goniometer centre.
2. Perform theta scan –2° ... 2° step size 0.02°. Double click at the centre of the peak to get a new requested value of theta. At this position the sample surface is parallel to the beam. If the peak has triangle shape and the peak maximum has 50% of the primary beam intensity go on with step 3 otherwise repeat step 1 and 2.
3. Move knife edge towards sample surface until it is optical dense. White paper is

helpful to have good contrast. If the knife edge is tilted against the sample surface tilt the knife edge with an allen wrench. The knife edge reduces the footprint of the beam to a flat area completely on the sample (fig. 6-44, 6-45).

4. Perform theta scan 0° ... 0.4°, step size 0.002° with 2theta=0.4° (the range of theta must be larger if the peak maximum in step 2 differed much from theta=0°). Double click on the maximum to get a new requested value of theta.

5. Perform a Chi scan from –2° ... 2°, step size 0.05° at theta=0.2°, 2theta=0.4°. Double click on peak maximum to change the requested value of Chi. By this the sample surface is moved parallel to the line focus of the X-ray beam.

6. Repeat step 4. This time the maximum of theta must be 0.2° because incident angle must be equal to exit angle. Perform a Zi determination with theta=0.2° as new value.

7. Perform a coupled scan 0° ... 2° to check the position of the critical angle of total external reflection, the quality of the alignment (flatness of plateau).

8. Determine a good measurement position for the knife edge (typically 0 ... 50µm on the micrometer scale, fig. 6-44, 6-45). The intensity increases with gap size and it should therefore be increased as long the total reflection plateau is sufficient.

9. Determine good positions for absorber switching. Keep in mind that you need about 1500 counts for sufficient statistics in the overlap point of the ranges.

10. Set up a dql file for the reflectivity measurement. Use the chi, z position determined above.

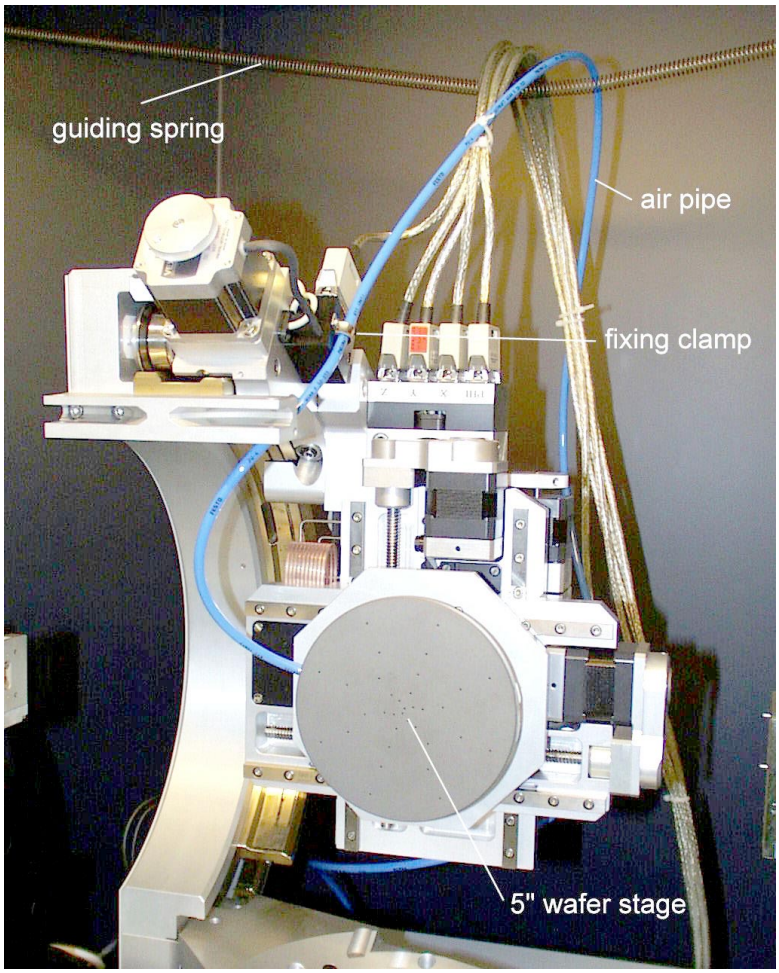


Fig. 6-36: 5” vacuum stage without knife edge and knife edge base mounted to ¼ circle eulerian cradle (e.g. HRXRD, Stress, Texture, Thin Film, Powder)

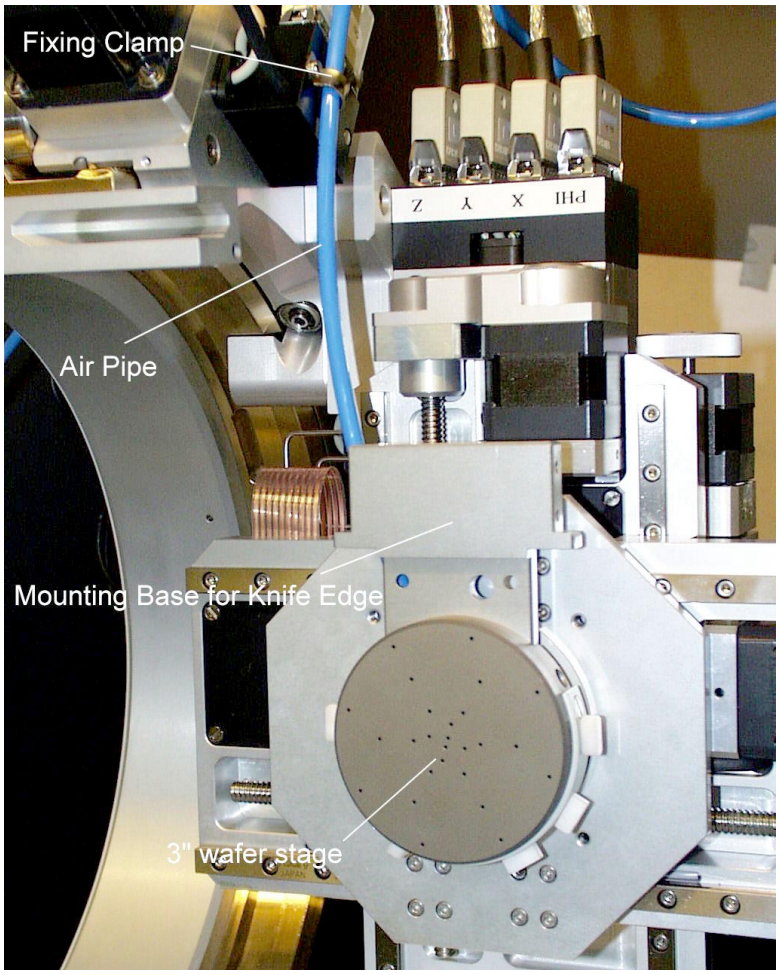


Fig. 6-37: 3“ vacuum stage mounted on ¼ circle eulerian cradle with mounting base for knife edge (e.g. HRXRD prepared for fast switching to reflectometry)

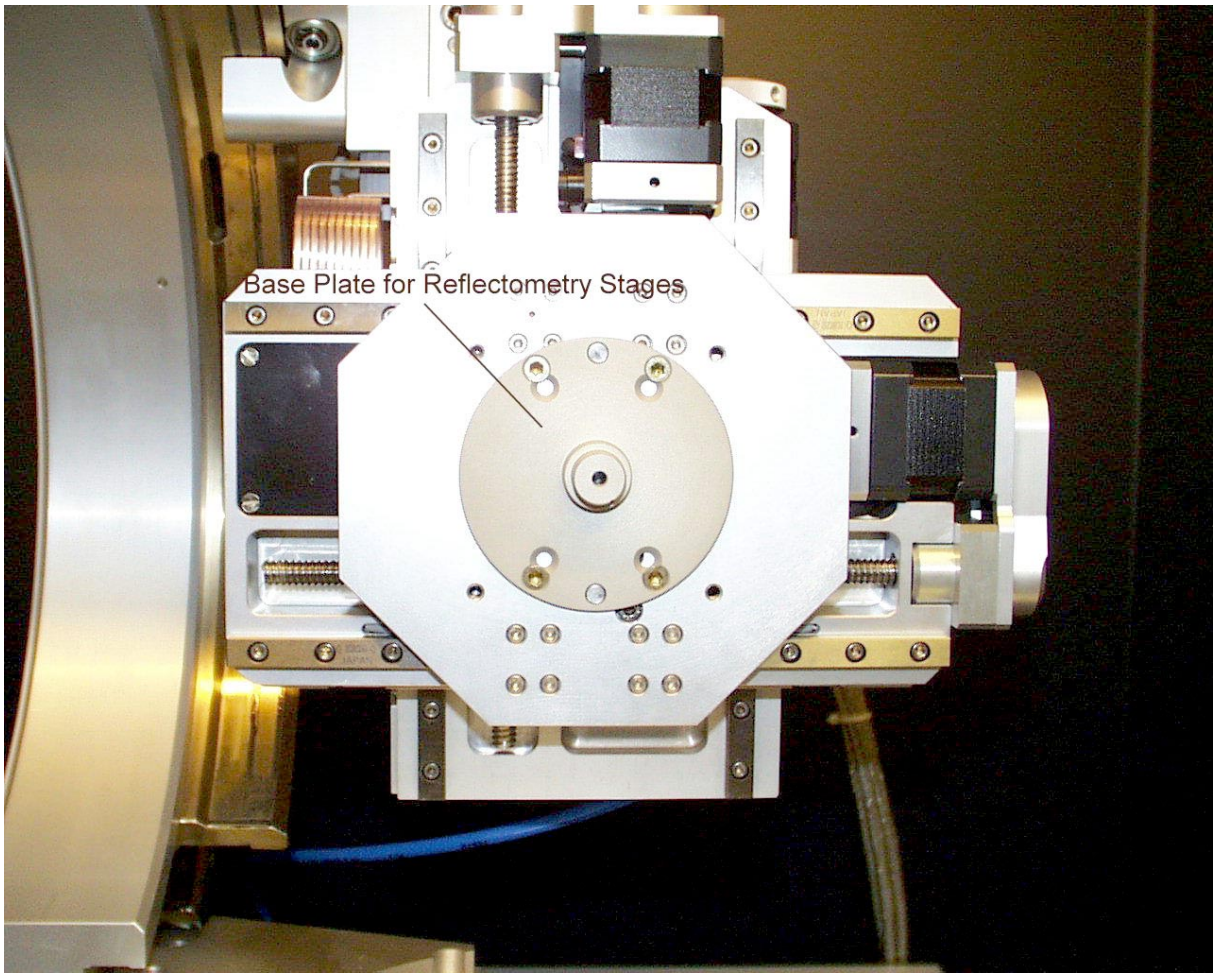


Fig. 6-38: Mounting base of vacuum stage on cradle

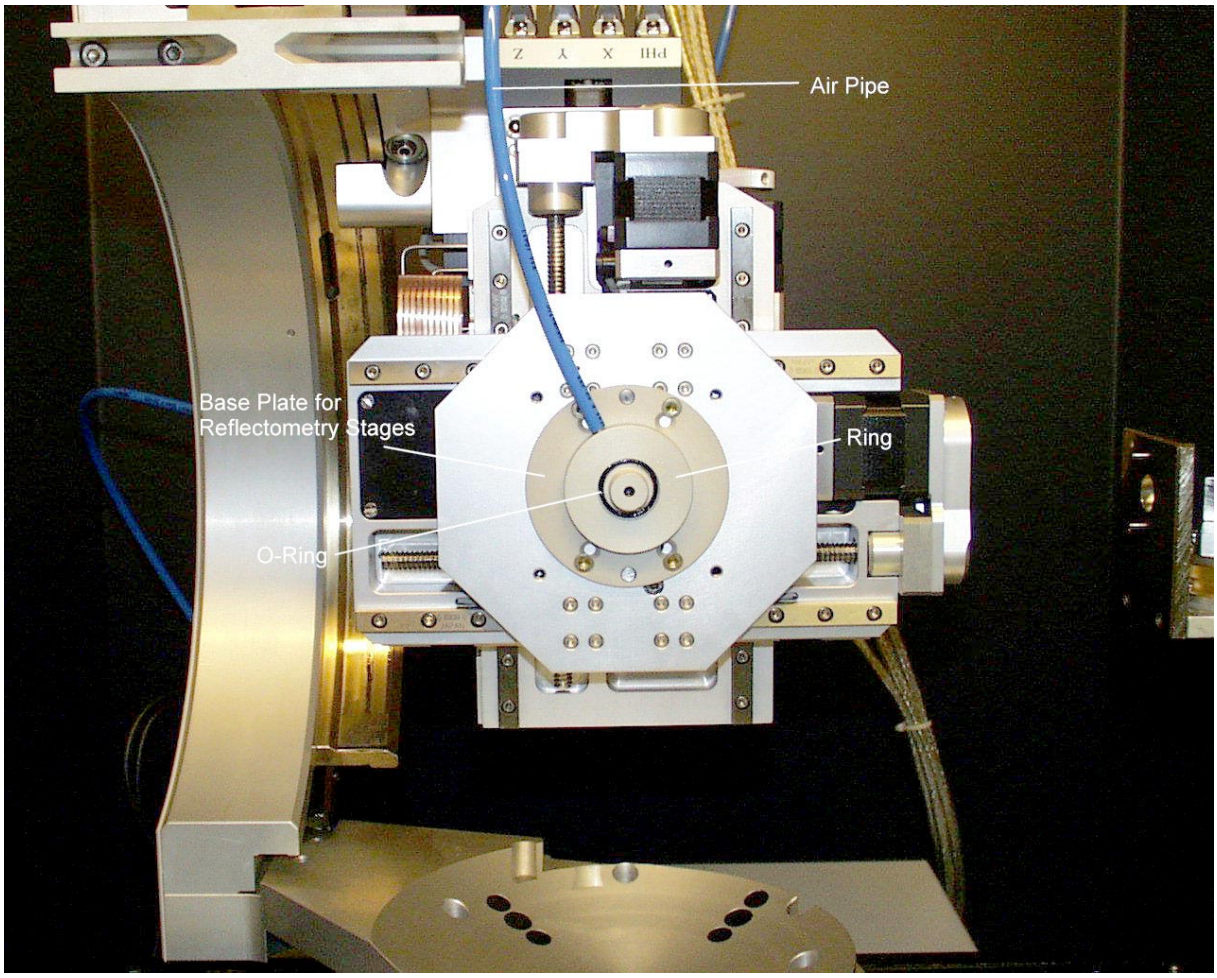


Fig. 6-39: Mounting the free rotation ring

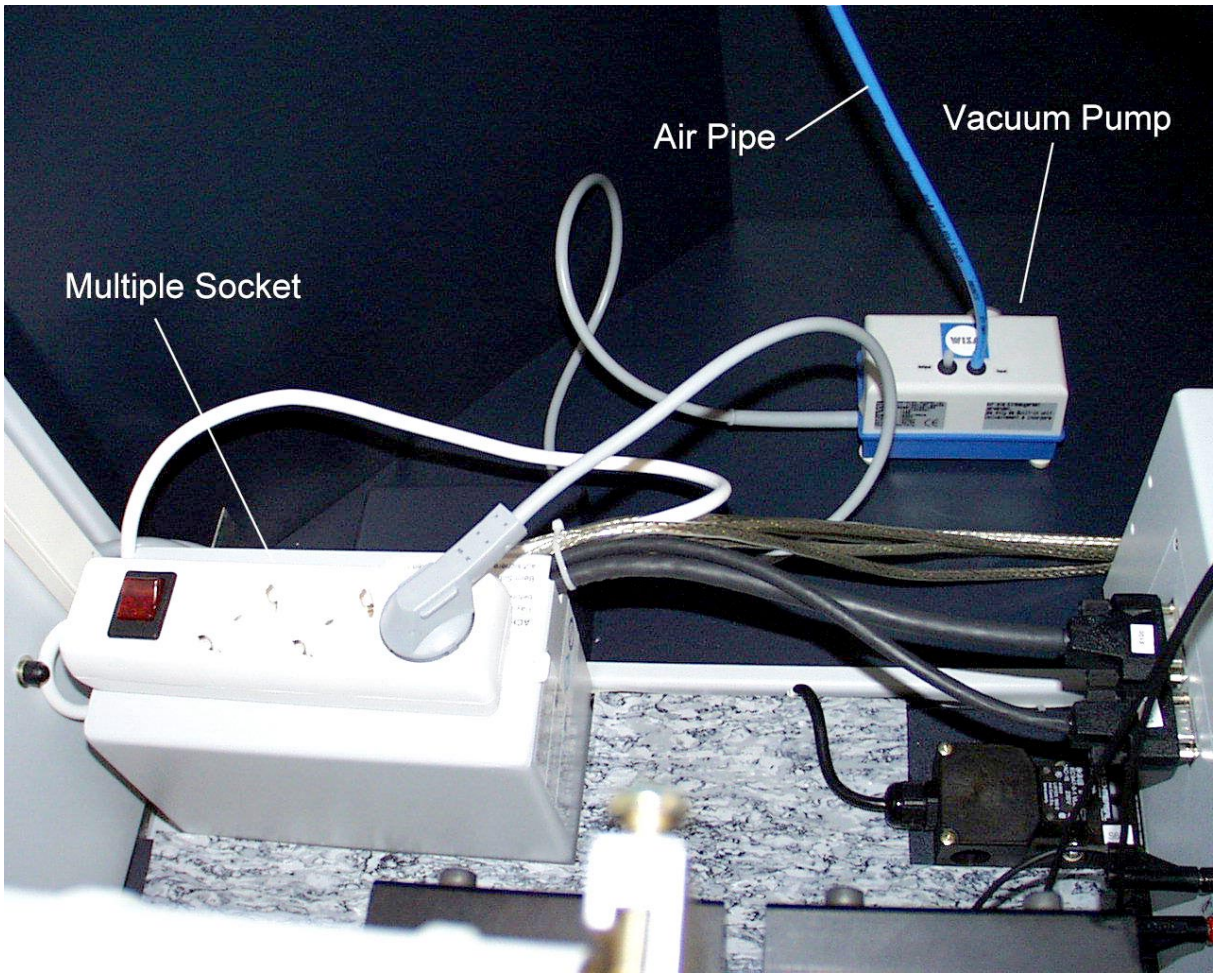


Fig. 6-40: Vacuum pump and socket

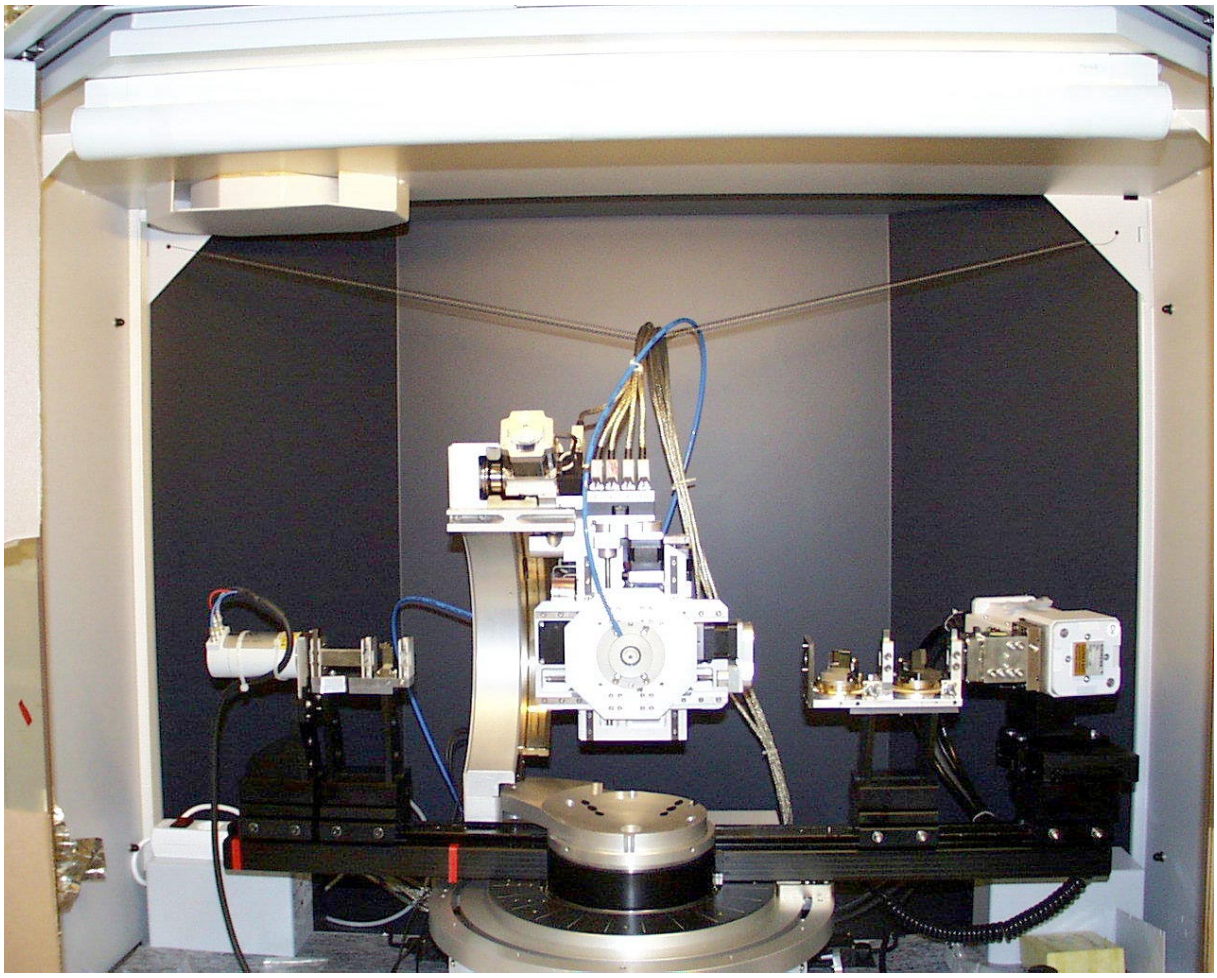


Fig. 6-41 Mounting the guiding spring

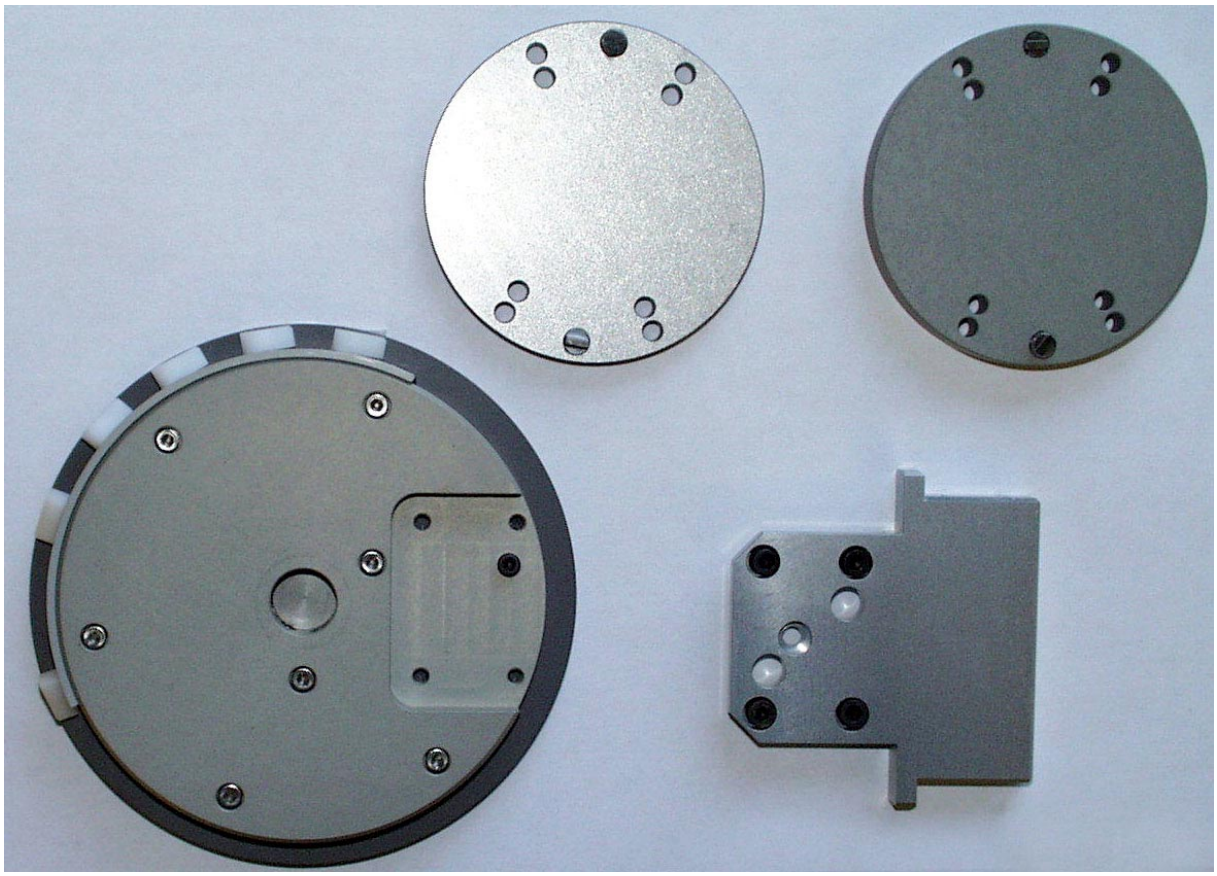


Fig. 6-42: Mounting the knife edge base, Spacers to increase thickness of vacuum stage

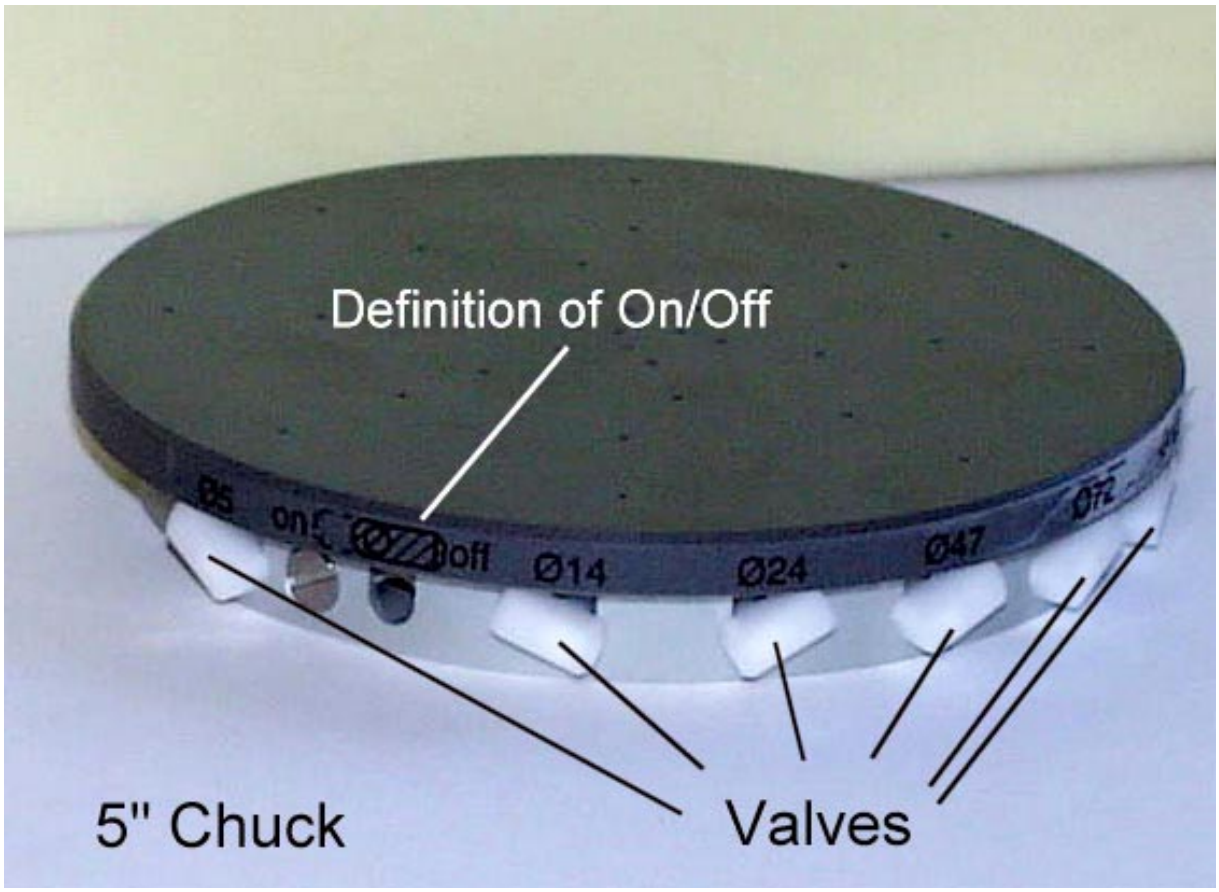


Fig. 6-43: Valves of 5” vacuum stage

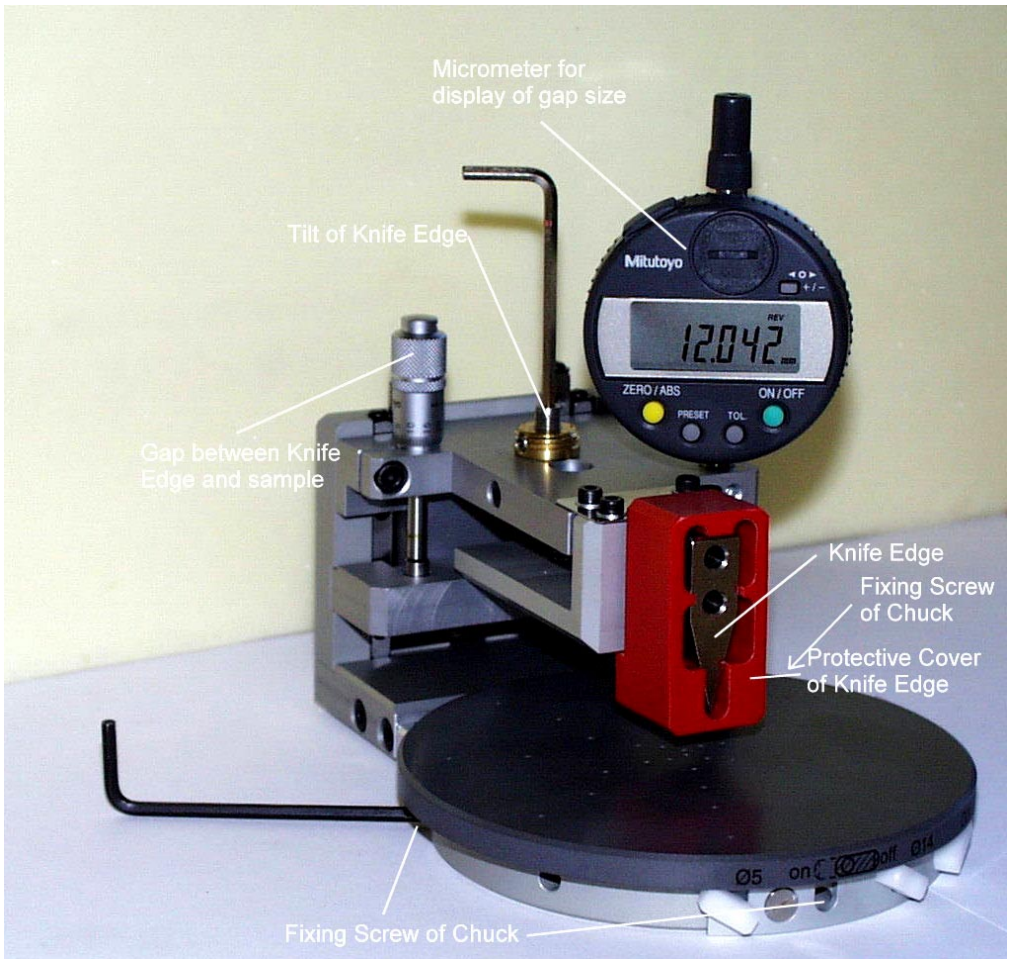


Fig. 6-44: Close view of 5” stage with knife edge mounted. The position of the three fixing screws is marked. It is indicated where to change gap and angle between knife edge and sample surface

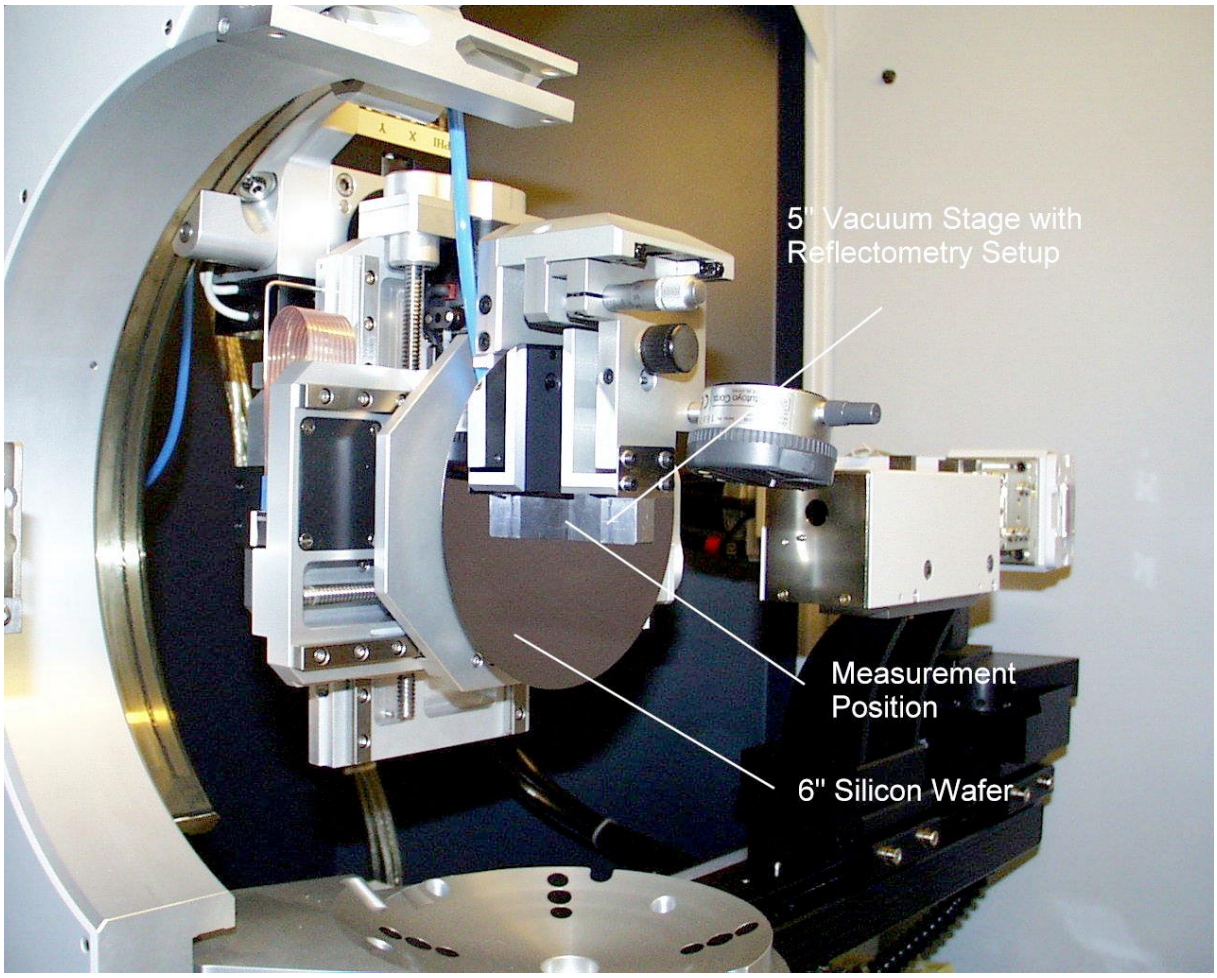


Fig. 6-45: 5” stage with 6” wafer in a typical measurement condition for reflectometry

Alignment Procedure for the D8 DISCOVER Channel-Cut Analyzer Crystal

1. Remove the analyser housing. With the analyser crystal at 0° rotation (fig. 6-46) determine the correct translation position in front of the detector as follows. With a 0.1 mm detector slit execute detector scans through the primary beam. The diffractogram should look similar to that obtained without the analyser crystal. Translate the analyser crystal (fig. 6-46) so that the front block of the channel cut crystal (that nearest to the sample) moves into the beam. Check the results of the translation with a detector scan and continue translating the crystal until the primary beam is completely shadowed.

2. Loosen the two screws on the bottom of the analyser unit (fig. 6-46) and offset the detector unit in its mount approximately 4.6 mm in the negative angular direction ($\Delta x = d * \sin(2*\Theta)/\sin(\Theta)$, d: channel width =2.5mm, Θ: Bragg angle of Ge 022 = 22.65deg). Retighten the screws.

   Rotate the knob 45 turns counter clockwise to obtain approximately the correct Bragg angle for the crystal (fig. 6-47). Remove the slit in front of the analyser crystal, and execute a detector scan through the primary beam (-2° < 2theta < +2°): A very broad peak will appear (position not around 0° 2theta, intensity approx. 80% of primary intensity without analyser mounted). This is not the reflected primary beam passing through the channel! The aim is to locate the narrow (approx. 0.0035° FWHM after final alignment, less than 0.01° with poor alignment) reflection with roughly 40% the intensity without analyser mounted respectively 50% of the broad peak mentioned above. This narrow reflection should appear on the negative side of the broad peak (about 0.5° +/- 0.2°). In case of problems to find the narrow peak the translation of channel cut or the detector holder is probably not correct. Repeat item 1 ! If available, the search can be made simpler by using a detector holder with a larger opening as in the grazing incident package (see note below).

   If this reflection is found, reduce the angular range of the detector scan and use a step size of 0.0004°. The intensity and width of the narrow peak must be optimised by aligning the tilt of the channelcut by turning the corresponding alignment screws by 1/8 turns until the highest intensity is achieved (fig. 6-47, compare alignment of four bounce monochromator).

   To get the peak to 0° the rotation angle (fig. 6-46) must be changed in steps of 1/16 turns counter clockwise and the detector

scan must be repeated. This must be repeated until the narrow peak is positioned around 0° +/- 0.005°. Better precision is possible, if necessary.

☜ **Note:**
In case of problems use a detector holder with a larger opening, if available (like that used in the grazing incidence attachment). In this case the translation of the detector holder is of no influence. Cover the parts of the detector opening left, right, and above the channelcut by e.g. lead tape to suppress the background. Now, it should be easy to find the narrow peak. The intensity can be optimised by translating the channelcut. Afterwards, the original detector holder must be remounted and the translation of the detector holder can be optimised independently.

The final result can be seen in fig. 6-48.

1. If the intensity is much less than 50% of the broad peak, the translation of the crystal requires adjustment (item 1 was not performed well enough). One half turn counter clockwise of the translation screw closer to the detector should be enough. Fasten the translation by turning the second translation screw clockwise. Check the result by repeating the detector scan. If the peak is not at 0°, the rotation must be improved to position the peak around 0° 2theta. If the angular position of the peak is above 0° turn the rotation 1/16 turn clockwise.

2. Mount a 1 mm slit in front of the analyser and scan the detector through 0° 2theta. The primary beam going directly through the channel must be fully suppressed without large intensity changes for the analyser reflection. If not, the translation of the detector and/or the analyser crystal require adjustment (see above) or the position of the detector slit holder must be optimised by loosening its fixing screws (fig. 6-46) and shifting it slightly.

3. After the alignment is finished, check that following positions are coloured to avoid accidental misalignment:

   - Mounting screws of the channel cut
   - Tilt screws

     Translation screws (channel cut)
   - Translation screws of detector holder

1. Replace the housing and tighten the three screws.

2. If the analyser crystal is used, the sample alignment will be more time consuming. The following procedure can be used to speed up the process (remark: automatic alignment must be done by specific alignment scripts which take into account the analyser):
   Remove the 1 mm slit in front of the ana-

lyser and execute a detector scan from –0.5° or 1.5° with a 0.01° step size. The analyser peak and the broad direct beam peak will appear. Determine the angular difference between the peaks.
If the Bragg reflection of a sample is to be aligned, the 1 mm can be removed. The detector should be positioned at the nominal angular value (two times the Bragg angle) plus the angular offset determined above. The sample rotation angle omega, the tilt angle chi, and the normal rotation phi can then be aligned as usual. Afterwards the slit is remounted. A detector scan around the nominal angular value is then executed. The angular position of the peak appearing now, is the starting point of the exact alignment of omega and 2theta of the substrate.

To find the optimum settings of omega and 2theta, a set of rocking curves with varying 2theta should be executed. The step size for the rocking curves is typically 0.0004° or less whereas the angular position of 2theta has to be changed in 0.001° steps. The perfect combination of omega and 2theta is found if the peak measured has its highest intensity (and smallest width).

3. Measuring without detector slit can also be used to combine reciprocal space mapping and ordinary rocking curves without any manual changes of the D8 DISCOVER (changing slits, taking out the analyser and inserting the slit system, ...). Just take into account the offset between the narrow analyser beam and the broad direct beam within your measurement set up.

Specification Values

Specification	Value
Intensity	>40% of primary beam without analyser mounted
Width of analyser peak (detector scan)	< 0.004°
Position of analyser peak	0° +/- 0.005°
Entrance slit of analyser (1mm)	Complete suppression of broad peak No shift and intensity loss of narrow peak

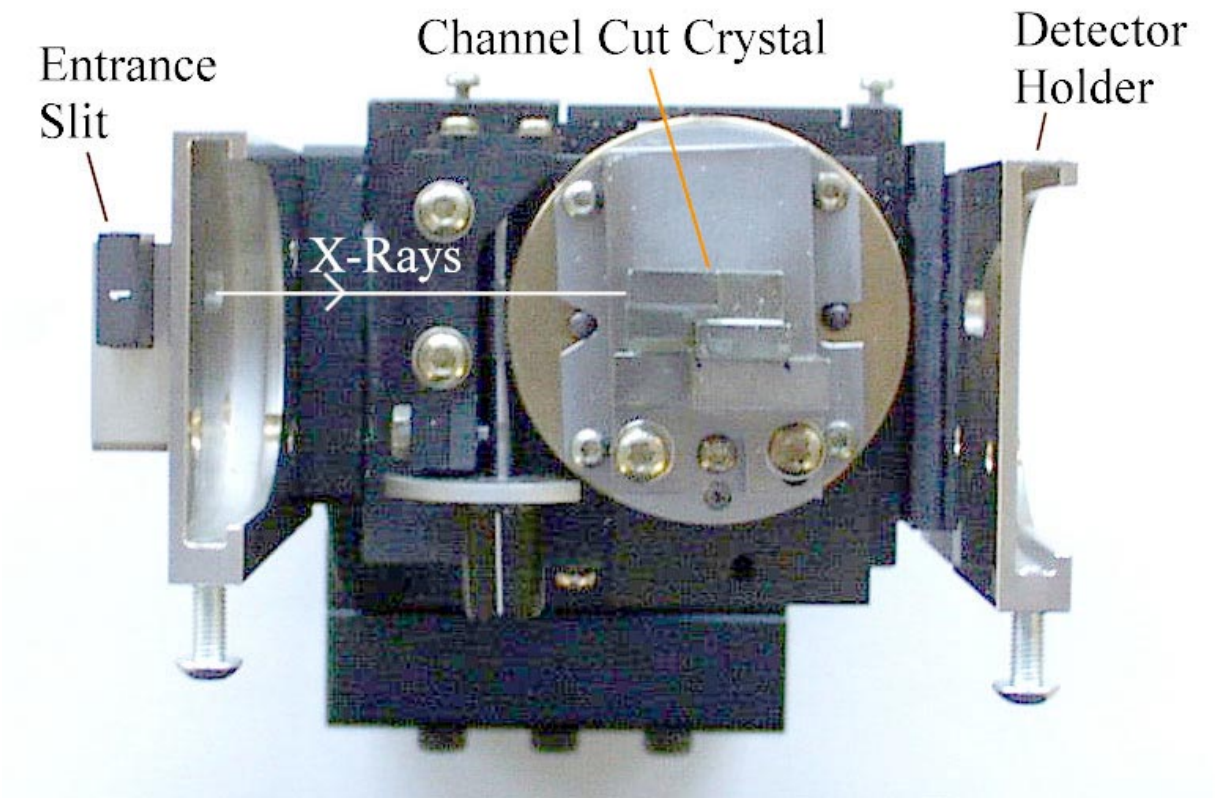


Fig. 6-46: Channel cut analyser with crystal parallel to primary beam

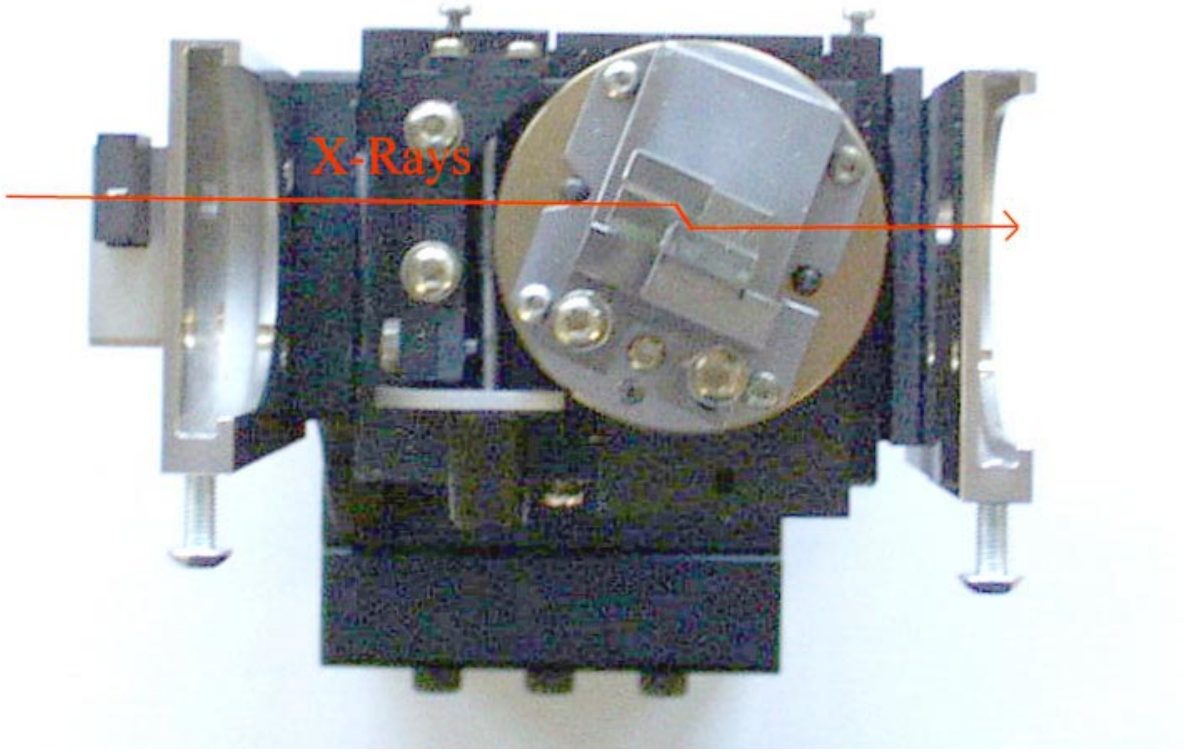


Fig. 6-47: Channel cut analyser with crystal in reflection mode after alignment

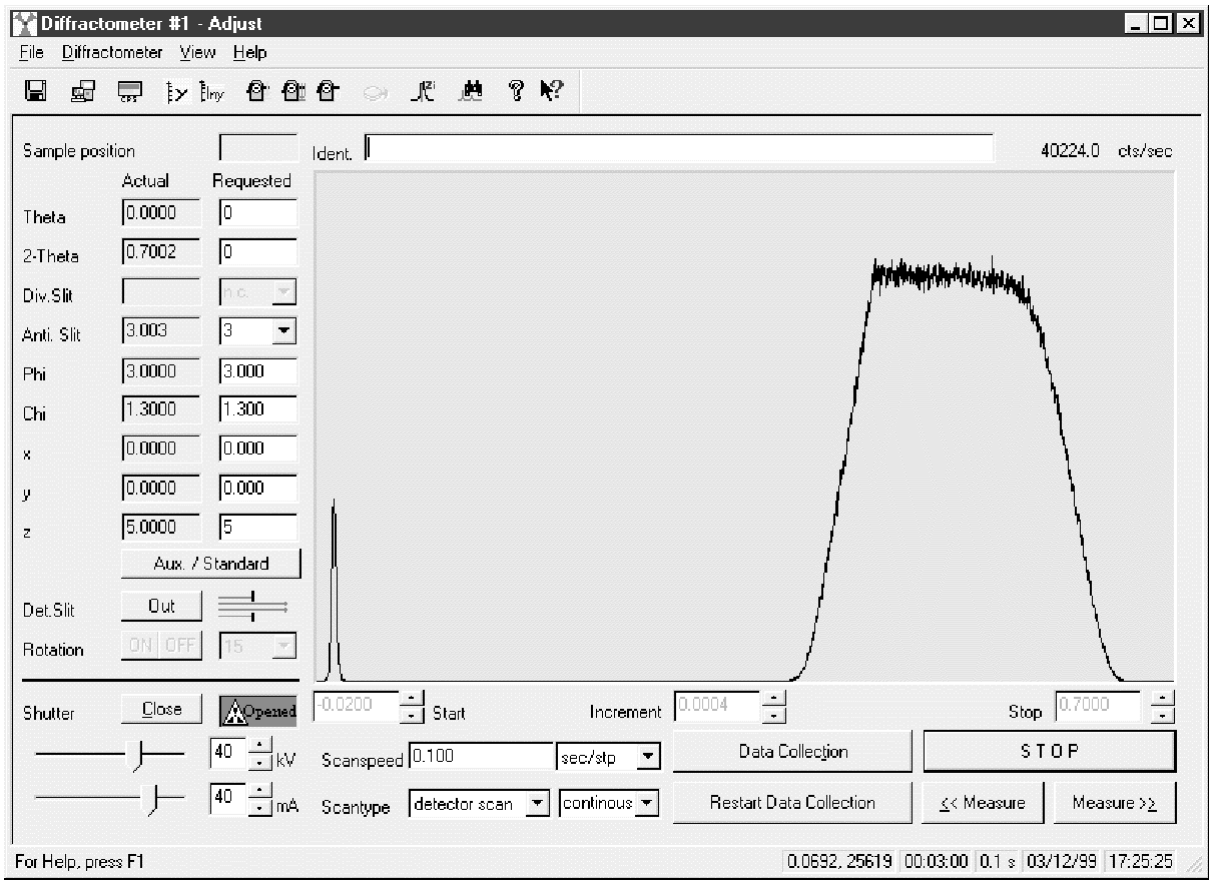


Fig. 6-48: 2theta scan after alignment without 1 mm entrance slit of analyser

Short Introduction to HR-Edit

HR-Edit will be the future Edit-DQL of the Diffrac Plus software. It is fully integrated into the Diffrac Plus suite, i.e. the same adjust, edit-job, eva, status display, ... The general features are similar to e.g. Tex-Edit for texture measurements. For sample orientation and alignment similar possibilities are given. Mostly, standard windows conventions are followed.

For a introduction to High Resolution X-ray Diffraction the “Win-HRXRD” Reference manual should be read (version 2.0).

If you want changes in HR-Edit to be valid, push “Ok” button to update values in memory. The relevant scan parameters for measurement are given in the top table.

Inside HR-Edit you have three main windows, which can be adjusted to user request (fig. 6-49).

1. Top window: Display of defined ranges. After save this will be the scans performed by Edit Job.
2. Left lower window: Tree which gives an overview of the different parts of HR-Edit. It shows which edit window is displayed in the third window
3. Right lower window: This window is used to define specific parts of a measurement. After pressing the “ok” button in this window the values will become valid and window 1 will be updated (this philosophy is identical with that in the configuration program and increases operation safety).

In the following the three most important branches of the tree will be explained. These have to be filled out if a new scan type, sample substrate, or scan range shall be defined.

Scan settings

In the branch “scan settings” the scan type is defined (fig. 6-49). The default scan type is “Rocking Curve”. For large scanning ranges “Unlocked Coupled” should be chosen.

Standard scan mode for high resolution measurements should be “step scan” because this gives highest precision. To speed up the data recording “continuous” mode can be used, especially for mappings. Of course this leads to precision below the capability of the goniometer.

Measurement geometry

For HRXRD measurement the orientation of the substrate has to be defined. This is done in the branch “Measurement geometry” (fig. 6-50).

Meaning of input parameters:

- Name: comment for user reference, e.g. silicion, Si, ...
- τ_{mis}: angle between surface and lattice planes (miscut, compare WIN-HRXRD reference manual)
- Surface (mno): Miller indices of lattice plane most parallel to surface (compare WIN-HRXRD reference manual)
- Azimuth (pqr): Miller indices of lattice plane normal to incident beam at theta=0° (compare WIN-HRXRD reference manual)
- Crystal system: definition of substrate lattice, default is silicon. If users want a different default value they should save a *.hrd file and start editing a new measurement after loading that file. (compare WIN-HRXRD reference manual)
- Diffraction Setup: This is a new feature. The D8 discover has a very high flexibility. For HRXRD measurements it is now possible to do scattering counter clockwise (plus = standard) or clockwise (- = optional) to optimise dispersion effects or to perform absolute lattice measurements.
- Analyser offset: This is new feature. Without taking out the analyser (used for reciprocal space mapping) it can be switched to ordinary rocking curve measurements by inserting the analyser offset (about 0.5°)

The values must be updated by “ok” if they shall become valid.

Scan parameter

The branch “scan parameters” defines the alignment of the sample and the scan range (fig. 6-51)

Substrate definition: Measurement

- Reflection hkl: Miller indices of reflection to be measured (compare HRXRD reference manual). The parameter s,+,- defines symmetrical incidence, steep incidence, and grazing incidence. +,- only makes a difference for asymmetrical reflections. S only makes sense for symmetrical reflections.
- τ_{Bragg}: angle between surface (mno) and reflecting lattice plane (hkl), compare HRXRD reference manual
- Theta: incident Bragg angle on Reflection hkl plane, compare HRXRD reference manual
- Omega: incident angle on surface (including τ_{Bragg} and τ_{mis}), compare HRXRD reference manual
- Fixed axis: position of the D8 drives which are not moved during the scan but are nonetheless important for sample alignment
- Write button: Very useful tool to write the file “adjust.fil” which defines the default values of the adjust program of Diffrac plus. A complete rocking curve will be predefined after starting of adjust. Just press execute measurement to find the substrate reflection.
- Get button: This tool reads the requested values as defined by Adjust with writing the Adjust.fil file (write values as default). Automatically the correct Omega, miscut, and fixed axis drives are inserted into the correspondent input fields of HR-Edit.

Scan range definition: Scan Parameters

- Scan type: scan type defined in “scan settings”
- Scan axis: drives which will be moved during scan
- Relative Start: start angle of scan, relative to substrate position defined by Reflection hkl
- Relative Stop: stop angle of scan, relative to substrate position defined by Reflection hkl
- Stepsize: angle between two measurement points. Minimum possible step size is 0.0001° (standard) or 0.00005° (after next update of configuration program).
- Time/Step: Measuring time per step
- Use Encoder: With DIFFRAC^{plus} Version 5 the Heidenhain encoders and the on axis encoders will be enabled.
- Delay time: waiting time before start of scan (e.g. some manual handling shall be done

between ranges or a heating device shall reach some temperature, ...)

- Total steps: number of steps (calculated from Start, Stop, and Stepsize)
- Total Scan Time: Time needed for the scan defined (calculated from input of user)

Write button 

Get button: 

Manual alignment with adjust

First put sample to vacuum stage and switch on pump. Adjust pressure by choosing adequate valves. Avoid too strong pressure because this might lead to sample bending and large FWHM of the rocking curves. Sometimes it is even helpful to open holes which are not covered by the sample. In some cases it is better to use graphical glue (such as “fix o gum”), some fat or a thin water film to fix the sample to glass plates. The glass plate can then easily be sucked by the pump.

Symmetrical reflections

1. Determine z with dial or as described in the reflectometry section and put this value into HR-Edit, either by pressing **Get** or manually.
2. Calculate default substrate parameter with HR-Edit and write “Adjust.fil” file for Adjust.
3. Start/Restart Adjust to read new “Adjust.fil”. Try default scan range without absorber. If necessary increase scan range, change step size, put additional absorber if intensity is above 300 kcps.
4. If reflection is found, perform a set of rocking curves with 0.0004° step size and different Chi. Change Chi by about 0.1°. Determine Chi with highest peak intensity (this is also the rocking curve with smallest FWHM). Check that FWHM is reasonable

for the sample (e.g. Si 111: 0.0033°, Si 004: 0.0045°). If FWHM is too large either sample quality or sample fixing is poor. Double Click on peak maximum. This gives a new requested value of theta.

5. Write “Adjust.fil” by using command “Write values as default”
6. Use “Get” command of HR-Edit to read the optimum values for chi, theta, ...HR-Edit automatically calculates the miscut of the sample.
7. Define scan range of interest (start, stop, step size, step time)
8. Save *.hrd file. Parallel a version 4 dql file is saved (next generation of Diffrac plus with encoder and script capability. This version 4 dql file is already used to maintain the adjusted measurement parameters. Therefore it should not be deleted unless the corresponding hrd file is also deleted.
9. Start new version of Edit Job (the new version knows a High resolution format “*.HRD”, mode = “HR”. This is important because otherwise the evaluation program “HRXRD.EXE” does not have the information about substrate orientation and sample alignment.

Asymmetrical reflections

1. If possible put sample in a defined horizontal orientation. Very often wafers have flats which define their in plane orientation. Choose an orientation which puts the desired reflection into the scattering plane of the goniometer.
2. Align a symmetrical reflection (e.g. 004)
3. In HR-Edit change reflection to the asymmetrical reflection to be measured e.g. from 004 s to 113 -.
4. Use Write command of HR-Edit to create a new “Adjust.fil”.
5. Start/Restart Adjust and execute Rocking curve measurement (without absorber).
6. If no reflection is found change phi in 10° steps.
7. If a reflection is found optimise peak intensity in repeated rocking curves with different phi setting (approximately 0.2° steps).
8. If phi must be changed a lot (>1°) iterate the alignment of the symmetrical and asymmetrical reflection. Always optimise the symmetrical reflection by changing chi and the asymmetrical reflection by changing phi.

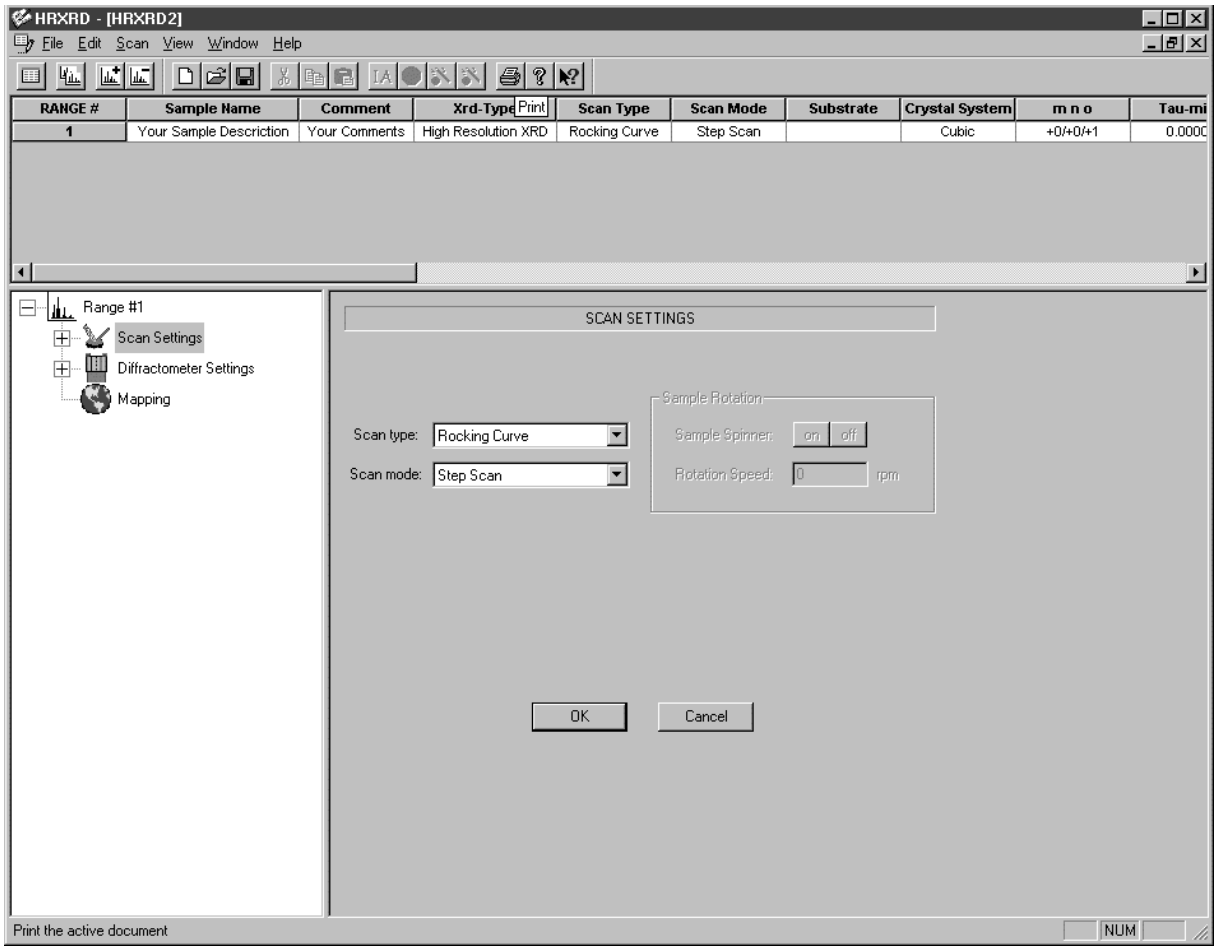


Fig. 6-49: HR-Edit (1)

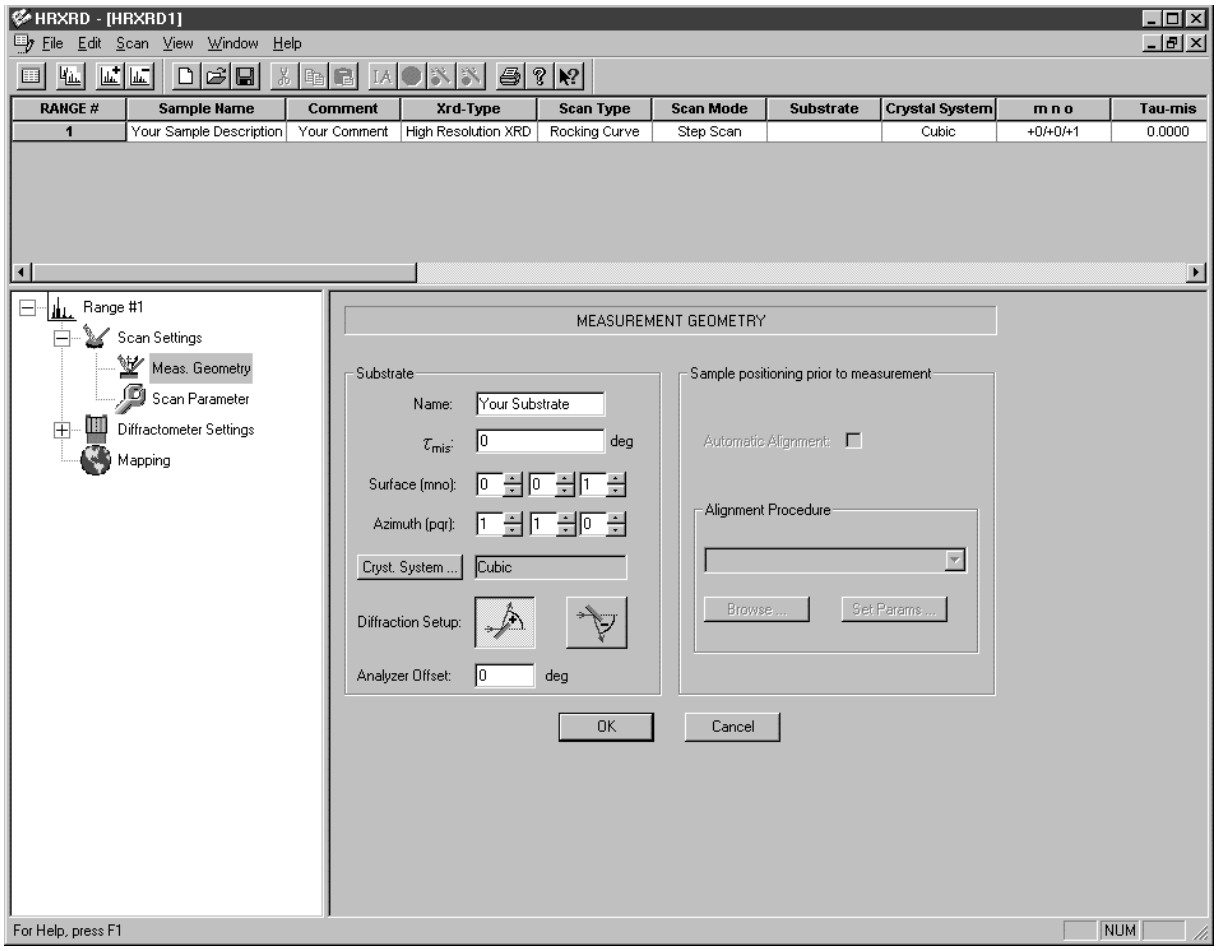


Fig. 6-50: HR-Edit (2)

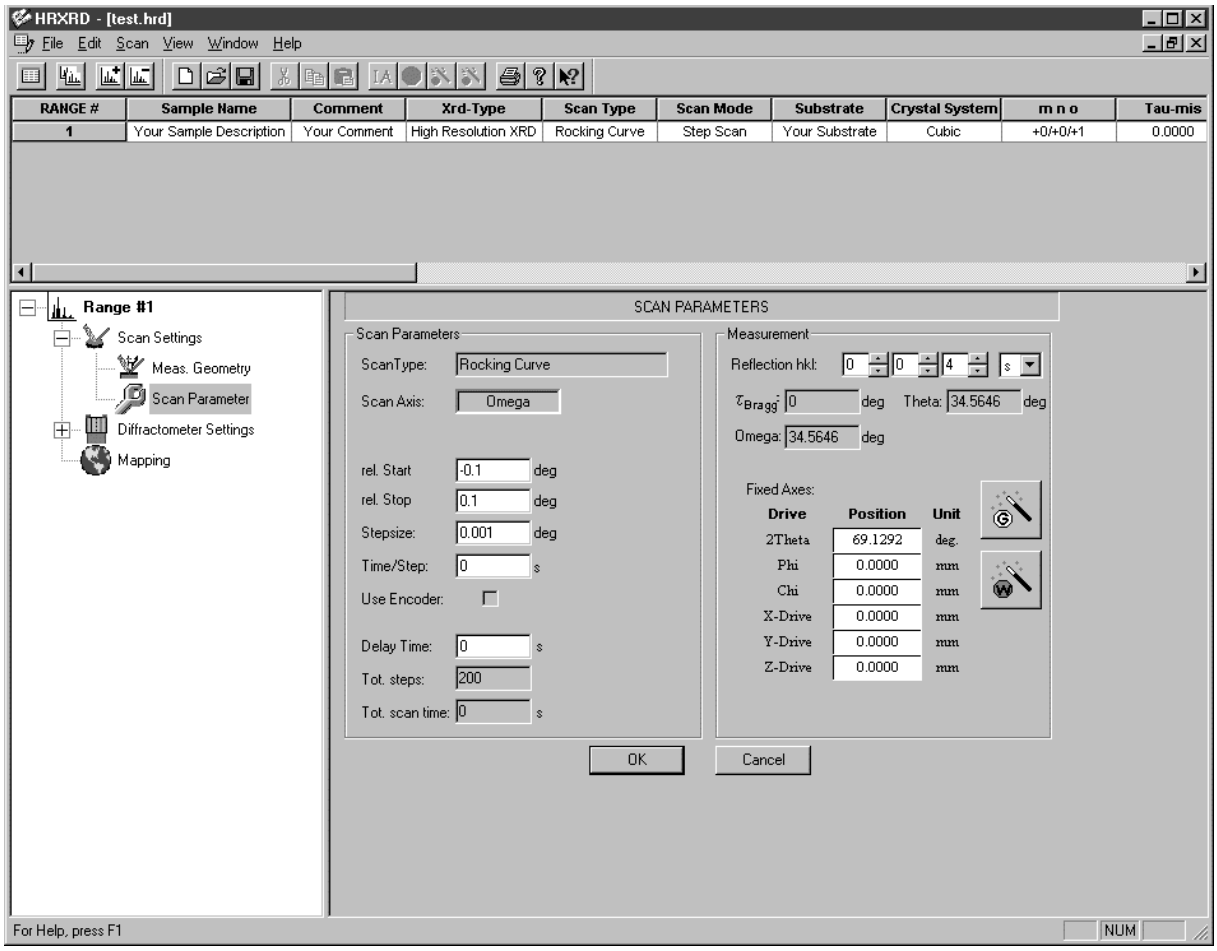


Fig. 6-51: HR-Edit (3)

7 Beam Expander and Compressor for D8

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7 Beam Expander and Compressor for D8

Introduction

The beam compressors and expanders (Fig. 7-10) are specially shaped channelcut crystals (v-grooves). They are designed for mounting behind a Cu Goebel mirror **with prefigured substrate** (C79298-A3161-D203) for further optimisation of the primary beam (Fig. 7-11).

They are capable to select pure Cu-Kα_1 radiation from the beam of the Goebel mirror. At the same time they provide a parallel beam with excellent divergence (approx. 0.006°). Each unit can be used either in compression or expansion mode. The compression / expansion factor is fixed by design.

Mounting and alignment takes about 15 - 30 min.

Of course, the compressor / expander can be removed to use the Goebel mirror without it.

Typical applications might be

- Moderate High Resolution X-ray Diffraction
- High resolution reflectometry
- Diffuse scattering with high resolution
- Powder diffraction in capillary mode with Cu-Kα_1 radiation
- Powder diffraction in transmission with Cu-Kα_1 radiation

Mounting

The housing of the compressor / expander unit (Fig. 7-1) is quite similar to the housing of the Goebel mirrors. The mounting is modular, i.e. several housings, slit translations, pinhole holders can be combined in various ways.

Before mounting the alignment of the Göbel mirror should be checked regarding tilt, Bragg angle, and position (cf. Alignment of the Goebel mirror). Fig. 7-12 shows a typical scan of the primary beam of a 60 mm Goebel mirror with prefigured substrate (C79298-A3161-D203) for checking the integrated intensity. The settings are 600 mm measurement circle diameter, 40 kV, 40 mA, detector slit system with 2° vertical Soller, 0.2 mm Cu absorber (in this case about $1.6*10^9$ cps, the centre of mass being at about 0°). Fig. 7-13 shows the same scan with an additional 0.1 mm detector slit. From the width (FWHM) the actual beam width

can be calculated: width=tan(FWHM)*measurement circle radius. In this case
width=tan(0.24°)*300=approx.1.3 mm.

For mounting proceed as following:

1. Remove the exit slit of the Göbel mirror. Therefore, translate the slit until two of the four mounting screws are visible (cf. Fig. 7-2). Unscrew them and translate the slit to the other side. After unscrewing the other two screws (Fig. 7-3) you can remove the slit holder from the Göbel mirror housing.
2. Connect the housing of the compressor/expander unit to the exit of the Göbel mirror by using the translation stage in the same manner as described for the removal of the exit slit of the Göbel mirror (Figs. 7-4, 7-5). The orientation of the housing with respect to the Göbel mirror is important (cf. Fig. 7-4): The beam enters the housing close to the middle and is offset by the v-shaped crystal by about 8 mm towards higher angles (this leads to approx. 1.5° in 2theta for a 600 mm diameter measuring circle).
3. The crystal can be mounted inside the housing in two ways (Figs. 7-4, 7-6). For changing between the two possibilities unscrew the two allen screws fixing the brass base of the crystal inside the housing (Fig. 7-7).
   – The beam is expanded if the broad opening of the “V” shows towards the sample (Fig. 7-6).
   – The beam is compressed if the narrow opening of the “V” shows towards the sample (Fig. 7-4).

Alignment

After mounting three parameters of the expander/compressor unit must be optimised (Fig. 7-8):

1. Bragg angle: range +/- 1.5°, precision < 0.005°
2. Tilt angle: range +/- 3° (1 revolution = 1.5°), precision < 0.2°
3. Translation: range +/- 8 mm (1 revolution approx. 1 mm), precision < 0.1 mm

As a starting point put the translation in a way that the compressor housing is flush with the housing of the Göbel mirror. The tilt should be adjusted in a way that the crystal is parallel to the housing. The Bragg angle should be put in the middle of the alignment range (approx. 4 revolutions). The exit plate should be mounted to reduce stray light from the direct beam from the Göbel mirror.

Alignment of Bragg angle

Before tuning anything perform a detector scan from –2° ... 5°, 0.04° steps and no absorbers. The $K\alpha_1$ beam from the v-groove crystal appears between 2° and 3° (v5 and v10, compressor and expander). Fig. 7-14 shows an example for the V10 compressor. The peak above 3° stems directly from the Göbel mirror. In the next step move 2Θ to this $K\alpha_1$ peak and activate the rate meter. Optimise the intensity with the help of the Bragg angle of the v-groove (Fig. 7-8). Fig. 7-15 shows a detector scan after this optimisation with 0.1 Cu absorber and 6 mm detector slit.

Move the beam through the centre of the goniometer with the translation of the DOF. In a first step this can be done roughly with a fluorescent screen. It is necessary to darken the room to see this $K\alpha_1$ beam. Fig. 7-16 shows the result for a v10 compressor, 6 mm detector slit and 0.1 mm Cu absorber. Typical values for the translation of the tube mount are listed in table 7-2.

Fig. 7-17 shows that the compression works. The scan measured with 0.1 mm detector slit and 0.1 mm Cu absorber has still half of the maximum intensity visible in Fig. 7-16 (measured with 6 mm slit) and is much sharper than the beam from the Göbel mirror (Fig. 7-13).

Alignment of tilt

Position the detector at the peak maximum, use adequate absorbers (0.1 ... 0.2 mm Cu), and a 6 mm detector slit. Start the rate meter. Optimise the intensity by tuning the tilt of the v-groove crystal (Figs. 7-8, 7-9). If the maximum is found, optimise again the intensity by tuning the Bragg angle of the v-groove.

Alignment of translation

At this stage of alignment it is possible that only part of the Göbel mirror beam is reflected by the v-groove. To check this move the detector to the peak maximum, use adequate absorbers (0.1 ... 0.2 mm Cu) and a 6 mm detector slit. Start the rate meter. Optimise the intensity by tuning the translation of the v-groove (Fig. 7-8). +/- 1 mm is a typical range.

Check the specifications

The guaranteed specifications and conditions are listed in table 7-1.

For checking the intensity insert an adequate aborber (0.1 ... 0.2 mm Cu) and a 6 mm detector slit. 0.1 mm Cu means an absorption factor of about 100 for Cu $K\alpha$ radiation. The maximum of the detector scan gives the intensity of the primary beam.

The width of the beam can be measured with a detector scan and 0.1 mm detector slit inserted: width = tan(FWHM)*300 mm. For a compressor setup the width of the slit cannot

be neglected and has to be subtracted from the calculated value.

The divergence of the beam must not be checked to validate the function of the v-groove. This is already within the specification if width and intensity of the beam are correct. The divergence may be checked by determining the FWHM of Si 111 reference crystal (can be ordered separately, order number K230-C10). For alignment of this crystal an eulerian cradle or a reflectometry stage is necessary (cf. Chapter 5, vol. II of D8 manual: alignment of a high resolution system). Table 7-1 gives a list of the Si 111 FWHM for the different setups. Fig. 7-18 show such a rocking curve for the v10 compressor crystal.

Fig. 7-19 demonstrates that the beam does not have any $K\alpha_2$radiation. The intensity ratio of $K\alpha_1$ / $K\alpha_2$ is larger than 10^7. The measurement was done without any absorber.

Figs. 7- 20, 7-21 and 7-22 show intensity, width and divergence for a v10 expander setup. Note that the beam is broader than the broadest detector slits, i.e. this setup should only be used with grazing incident Soller slits.

Figs. 7-23, 7-24 and 7-25 show intensity, width and divergence for a v5 compressor setup.

Figs. 7- 26, 7-27 and 7-28 show intensity, width and divergence for a v5 expander setup.

Centring through the goniometer axis

This must be done with the help of the tube mount (DOF) in the manner described for the Göbel mirror alone.

Table 7-1. Generator settings 40 kV, 40 mA, measurement circle diameter 600 mm, no vertical Soller slits, adequate absorbers (Cu).

Optic	Intensity (cps)	Divergence (FWHM Si111)	Beam width (FWHM)
Prefigured Göbel mirror, 60 mm length	> $1.3*10^9$	< 0.03°	>1.1 mm
V5 Expander	> $6*10^7$	< 0.007°	> 4 mm
V5 Compressor	> $6*10^6$	< 0.007°	< 0.3 mm
V10 Expander	> $3*10^7$	< 0.005°	> 7 mm
V10 Compressor	> $7*10^6$	< 0.01°	< 0.2 mm

Table 7-2. Typical settings of the 2DOF tube mount for beam through centre and parallel to track.

Optic	Rotation	Translation
Prefigured Göbel mirror, 60 mm length	0°	4 mm
V5 Expander	0°	12 mm
V5 Compressor	0°	12.5 mm
V10 Expander	0°	13 mm
V10 Compressor	0°	14 mm

Figures

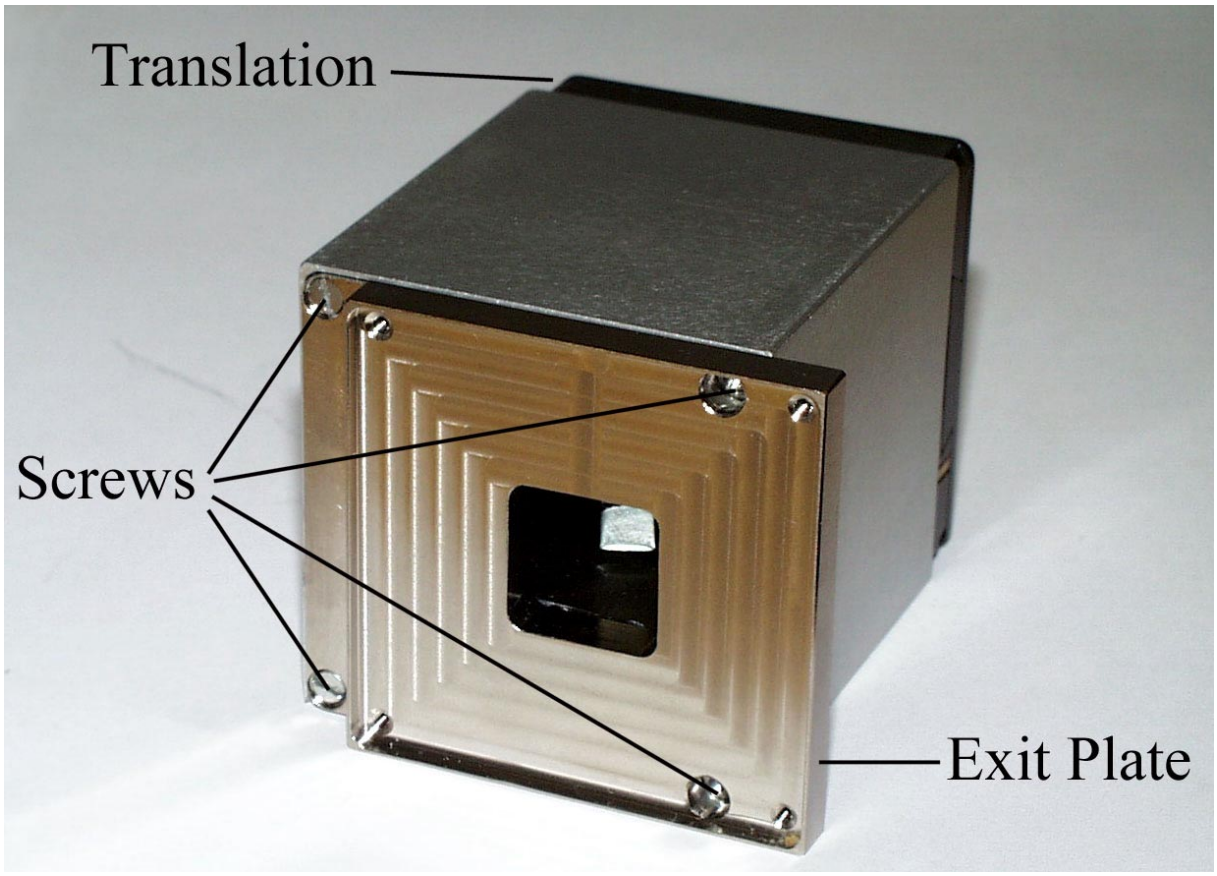


Fig. 7-1: Housing of the compressor/expander unit.

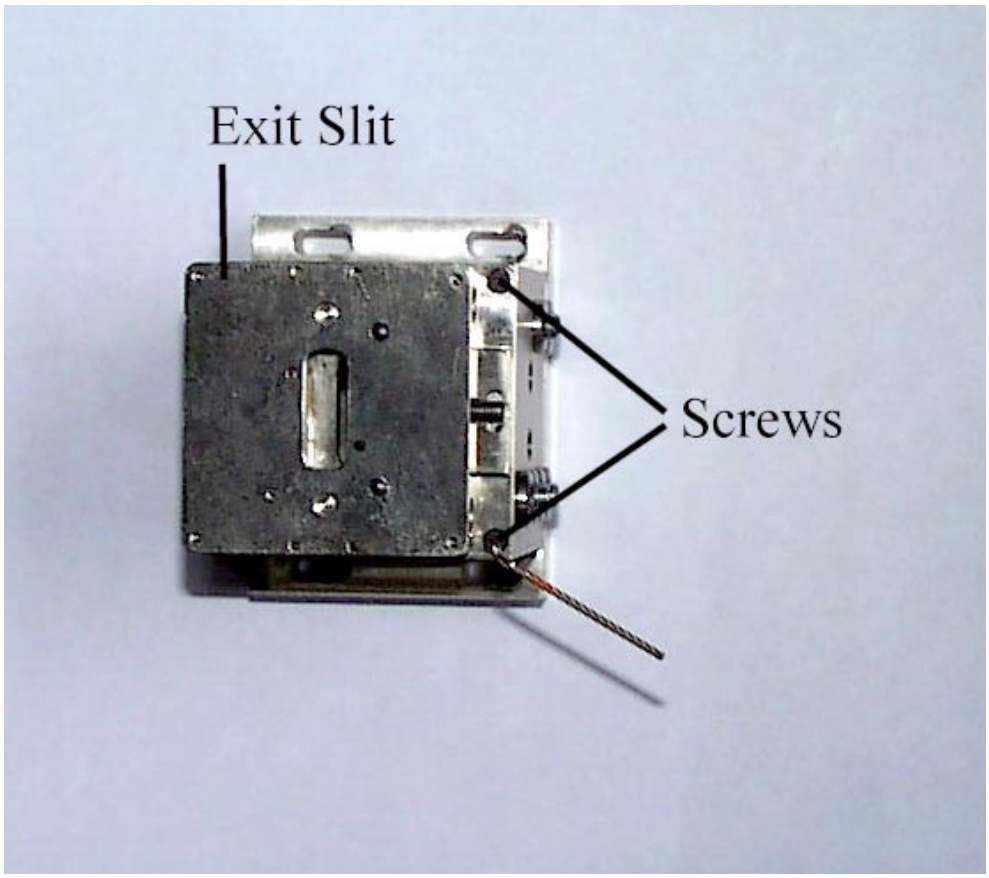


Fig. 7-2: Removing the exit slit of the Göbel mirror (screw 1 and 2).

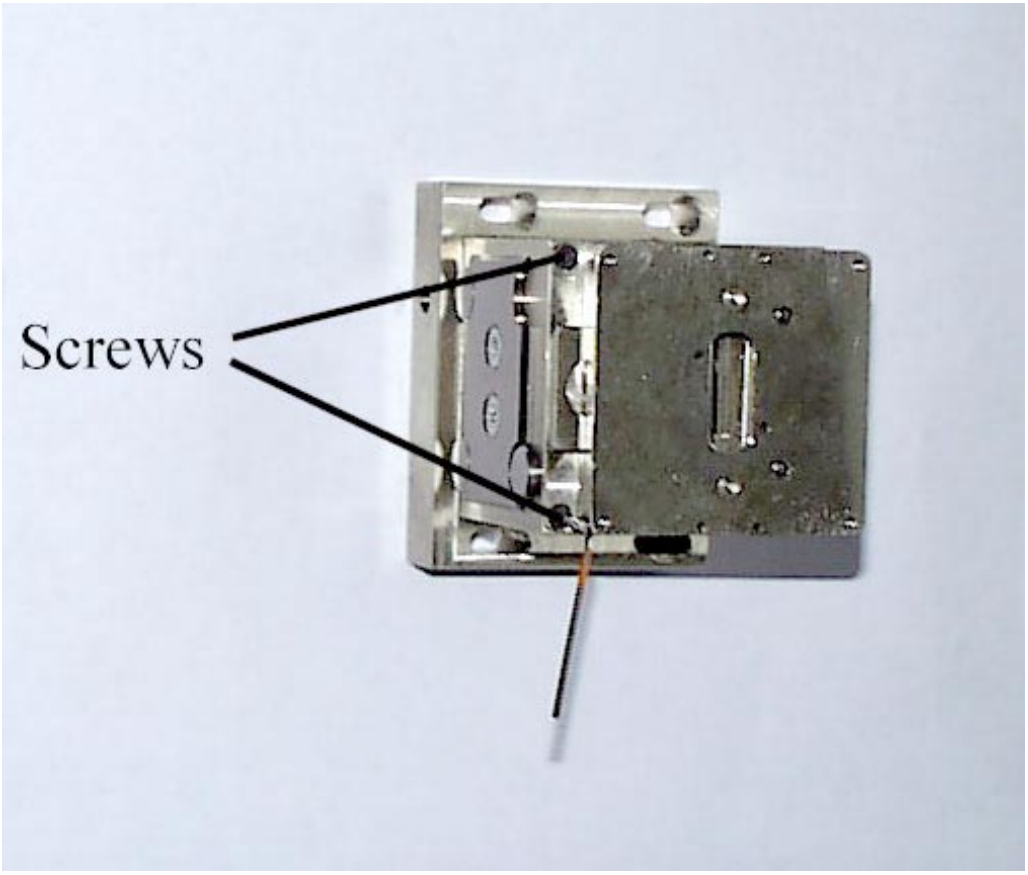


Fig. 7-3: Remove the exit slit of the Göbel mirror (screw 3 and 4).

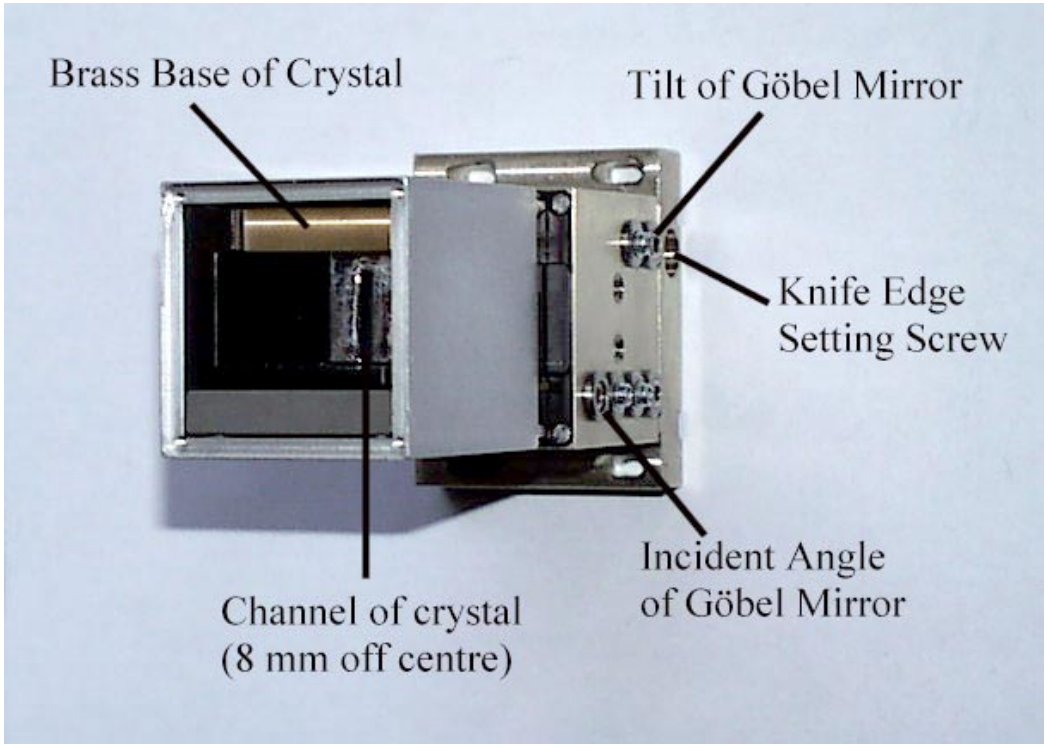


Fig. 7-4: Mounting the compressor/expander unit with setup as compressor (1).



Fig. 7-5: Mounting the compressor/expander unit (2).



Fig. 7-6: Setup as expander.

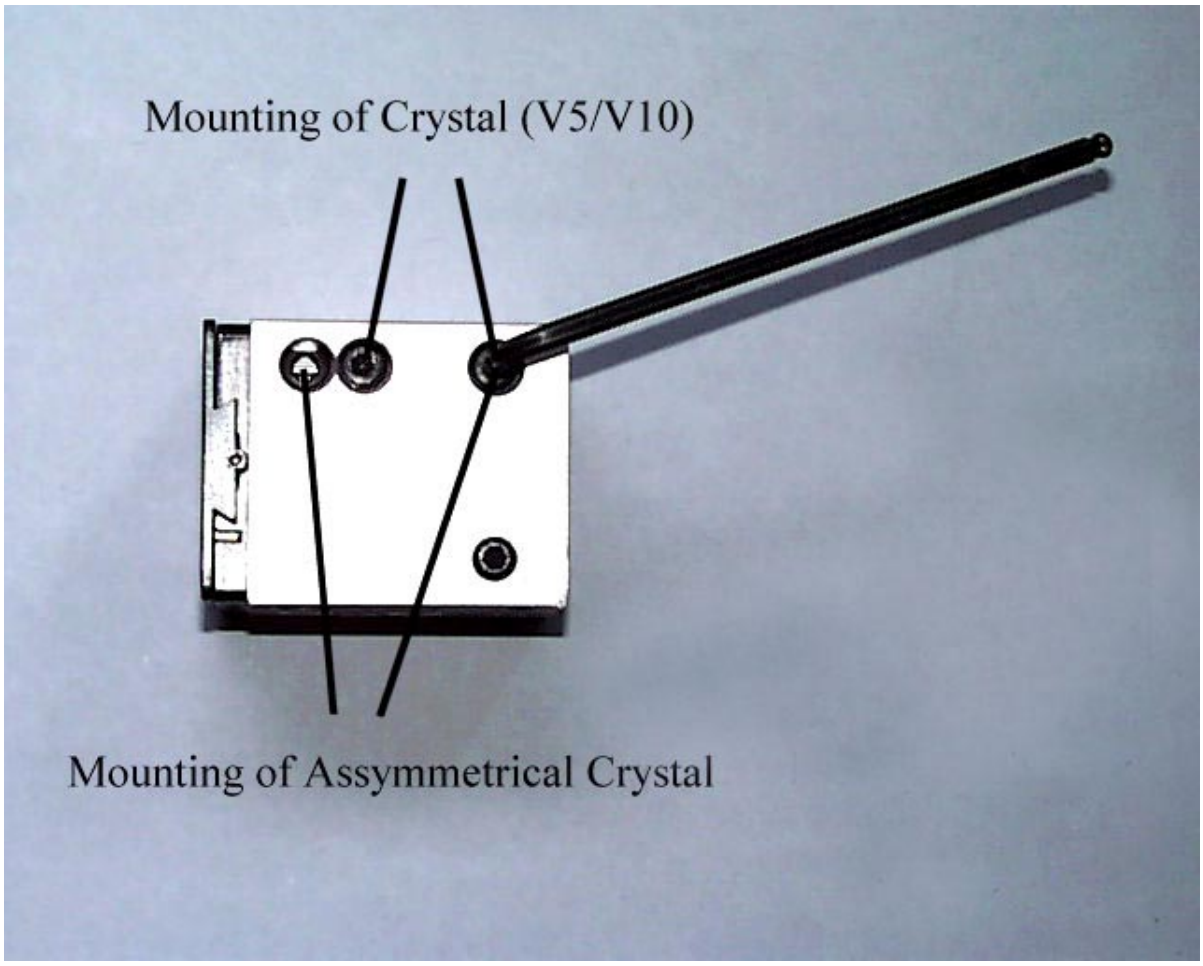


Fig. 7-7: Changing between expanding and compressing mode.

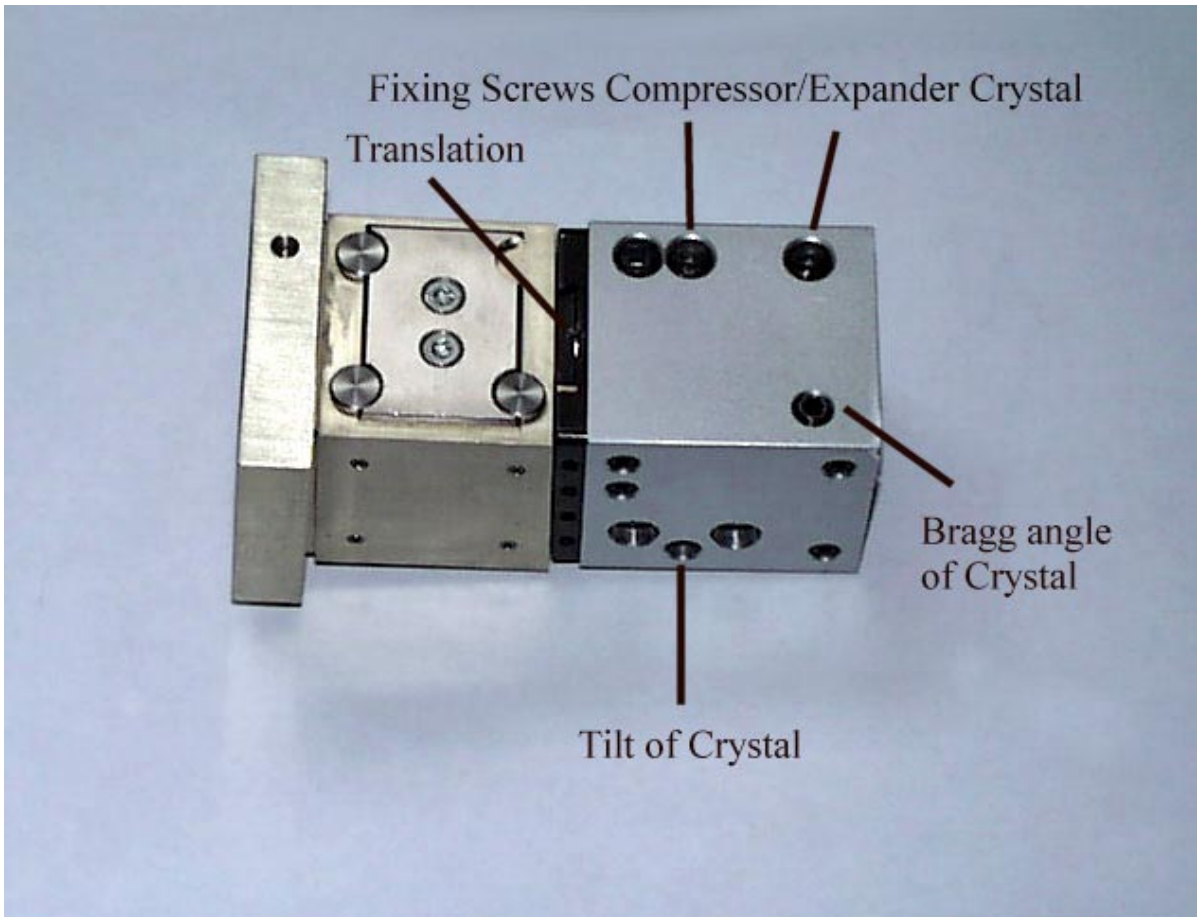


Fig. 7-8: Alignment parameters of the expander/compressor.

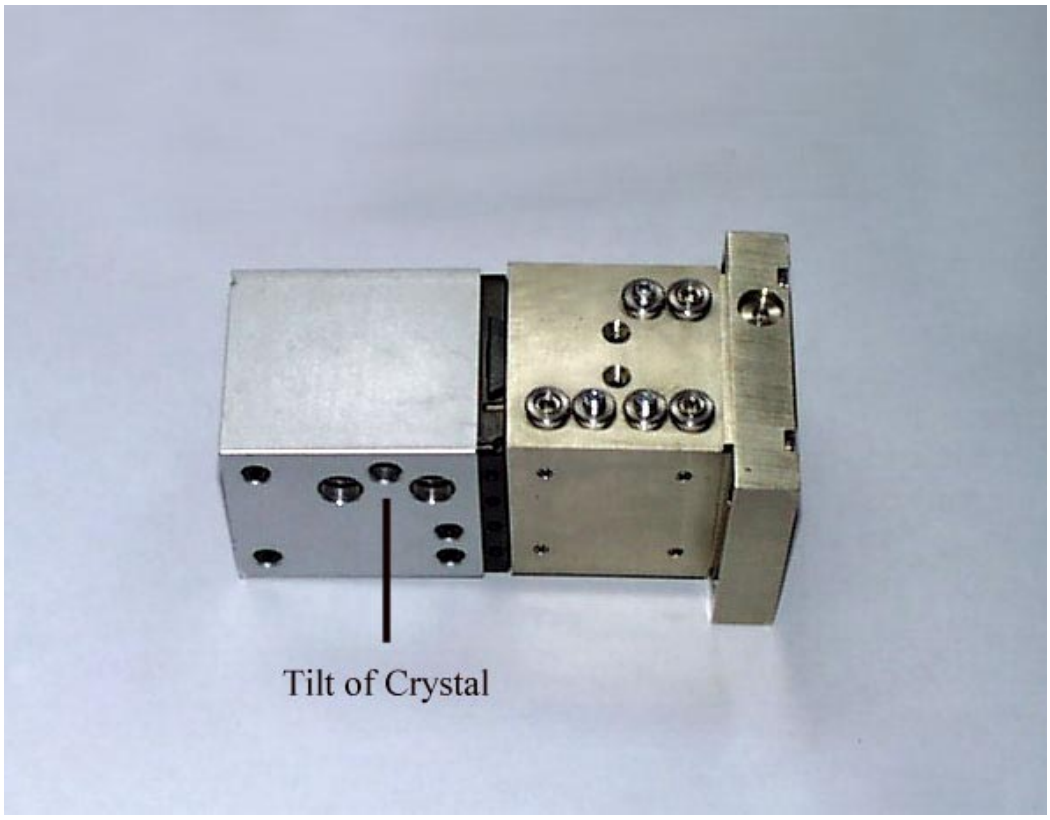


Fig. 7-9: Tilt of the excpander/compressor.

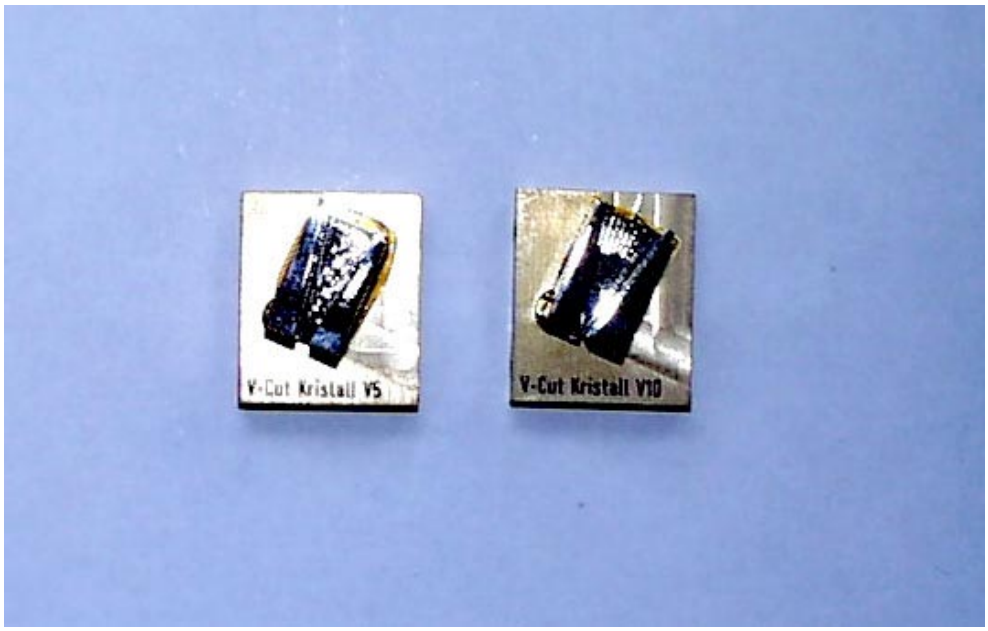


Fig. 7-10: Compressor/expander crystals.

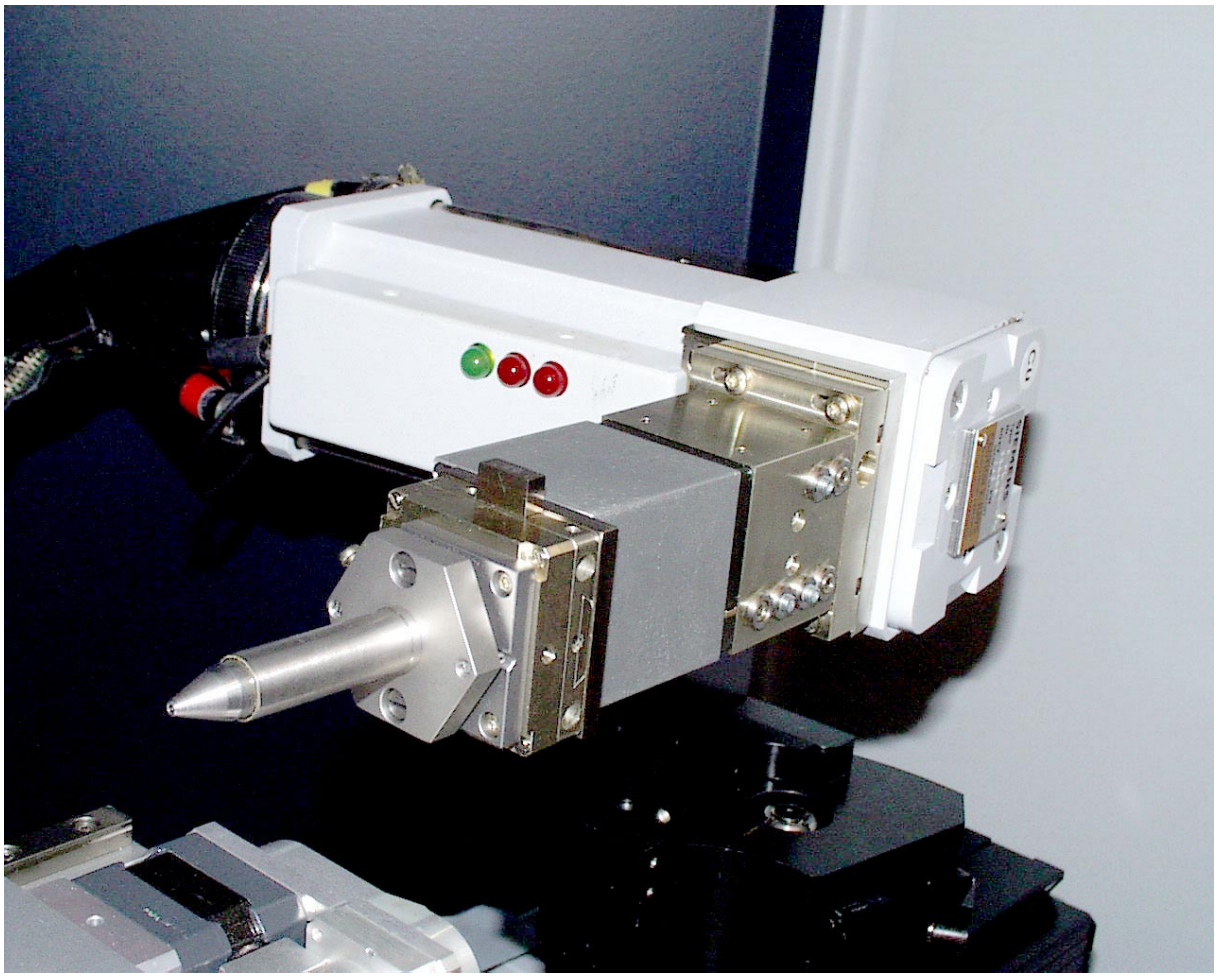


Fig. 7-11: Beam expander/compressor for D8 (with pinhole to reduce foot print).

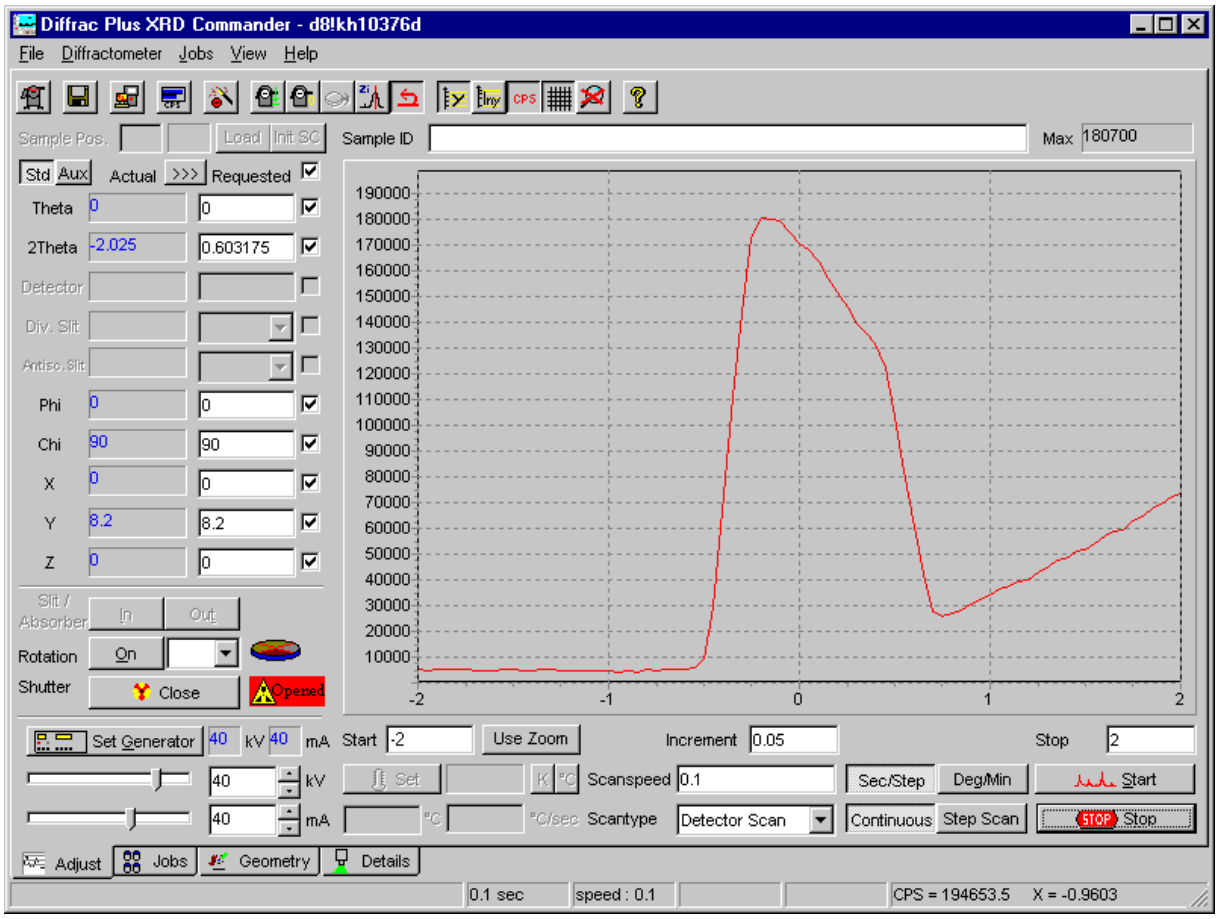


Fig. 7-12: Primary beam of the Göbel mirror with prefigured substrate (6mm detector slit, 2° Soller, 0.2mm Cu).

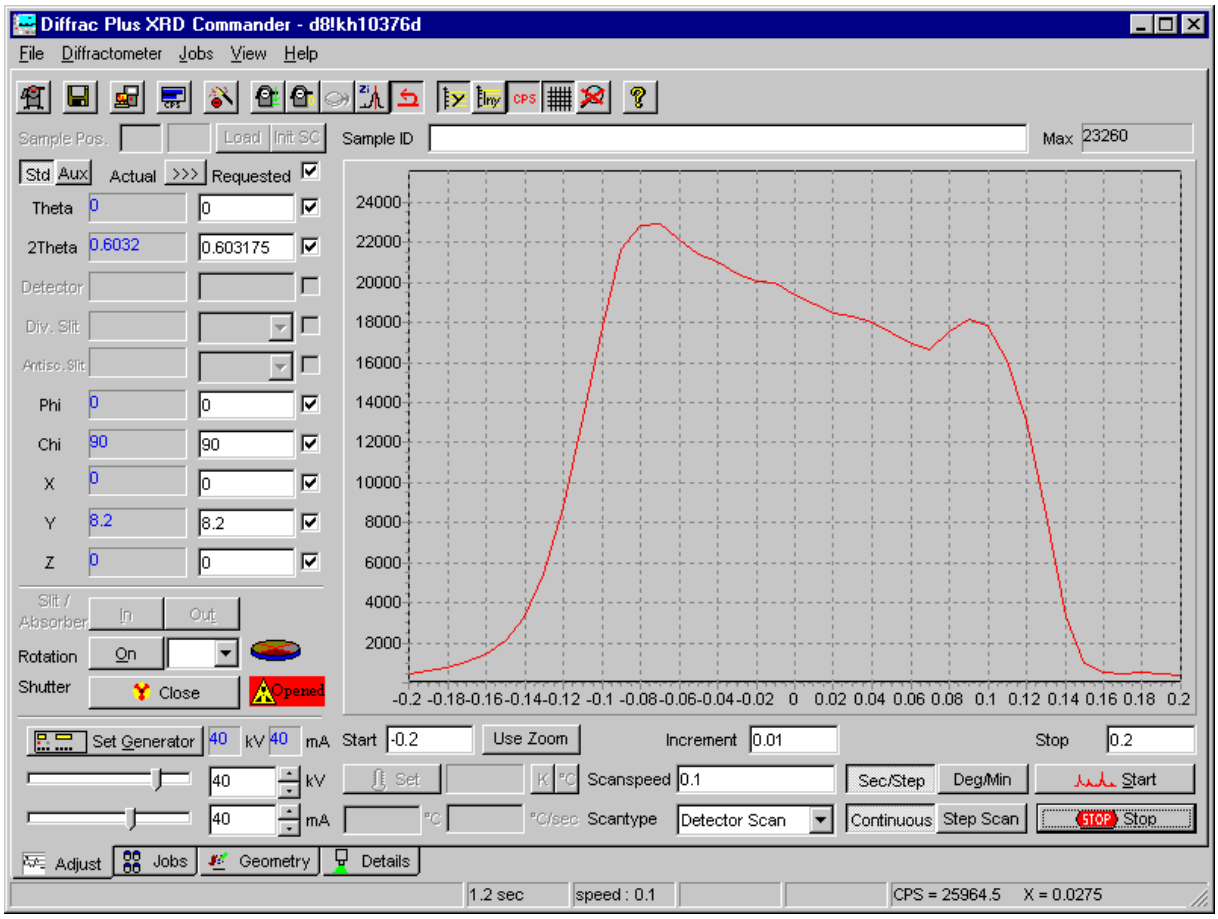


Fig. 7-13: Primary beam of the Göbel mirror with prefigured substrate (0.1mm detector slit, 2° Soller, 0.2mm Cu).

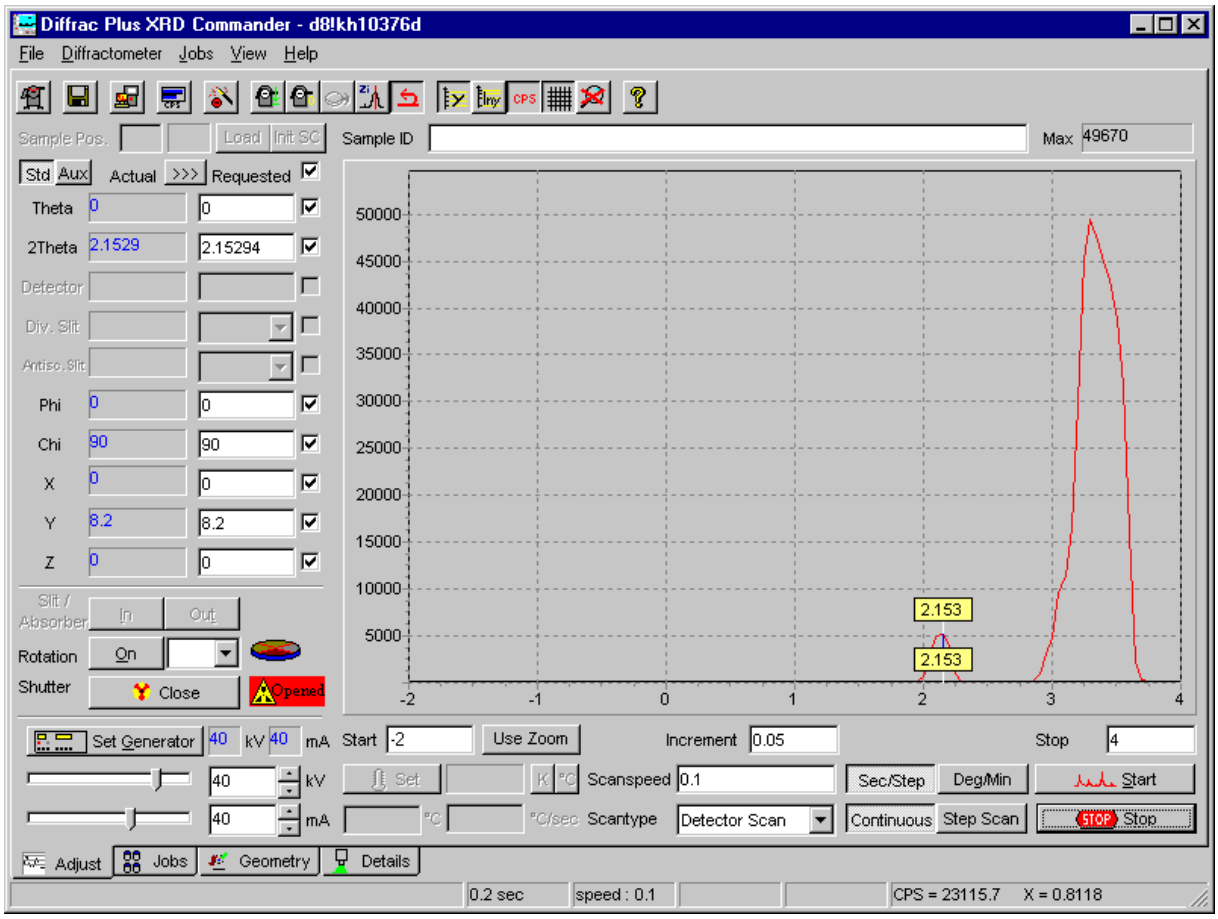


Fig. 7-14: First scan after mounting the V10 compressor.

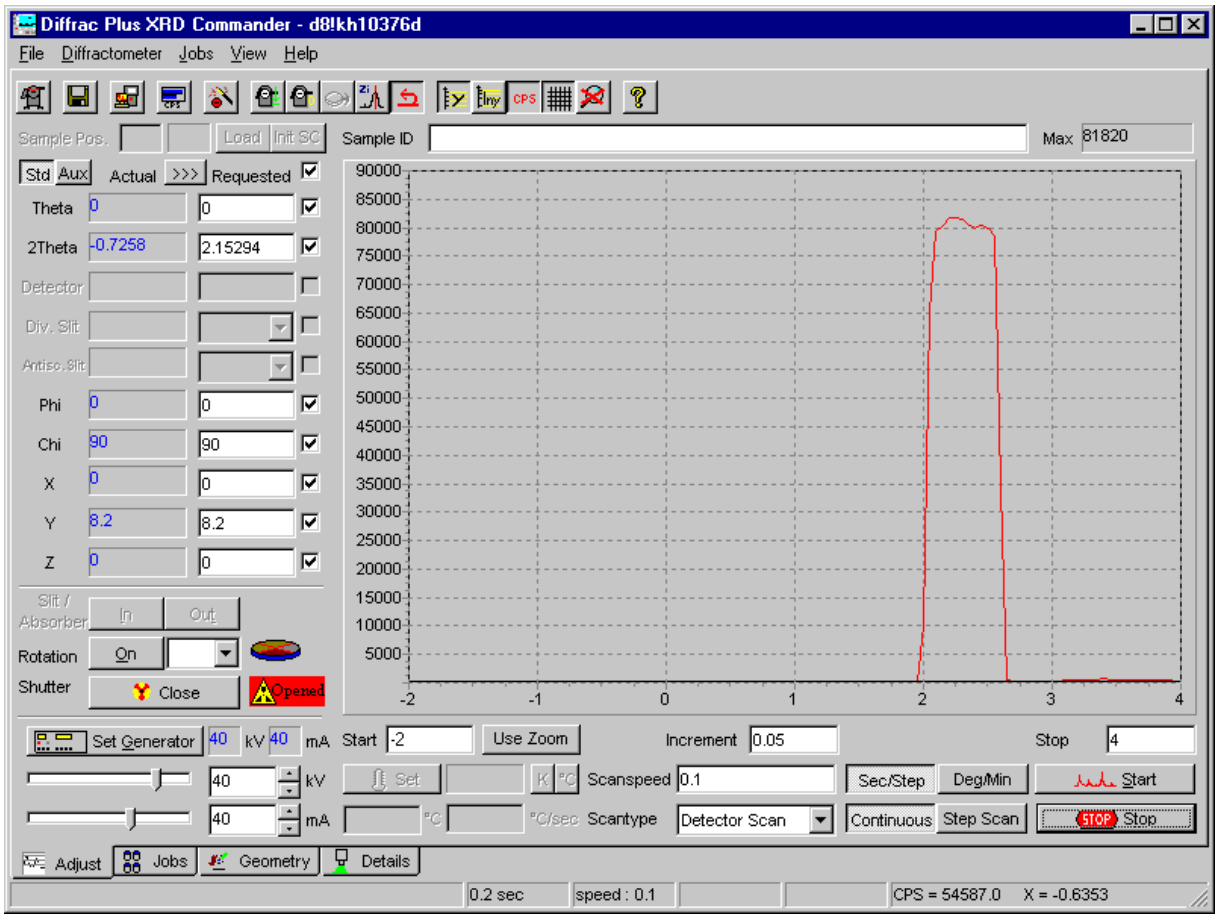


Fig. 7-15: V10 compressor after optimizing the intensity (detector scan with 6mm detector slit, 0.1mm Cu absorber).

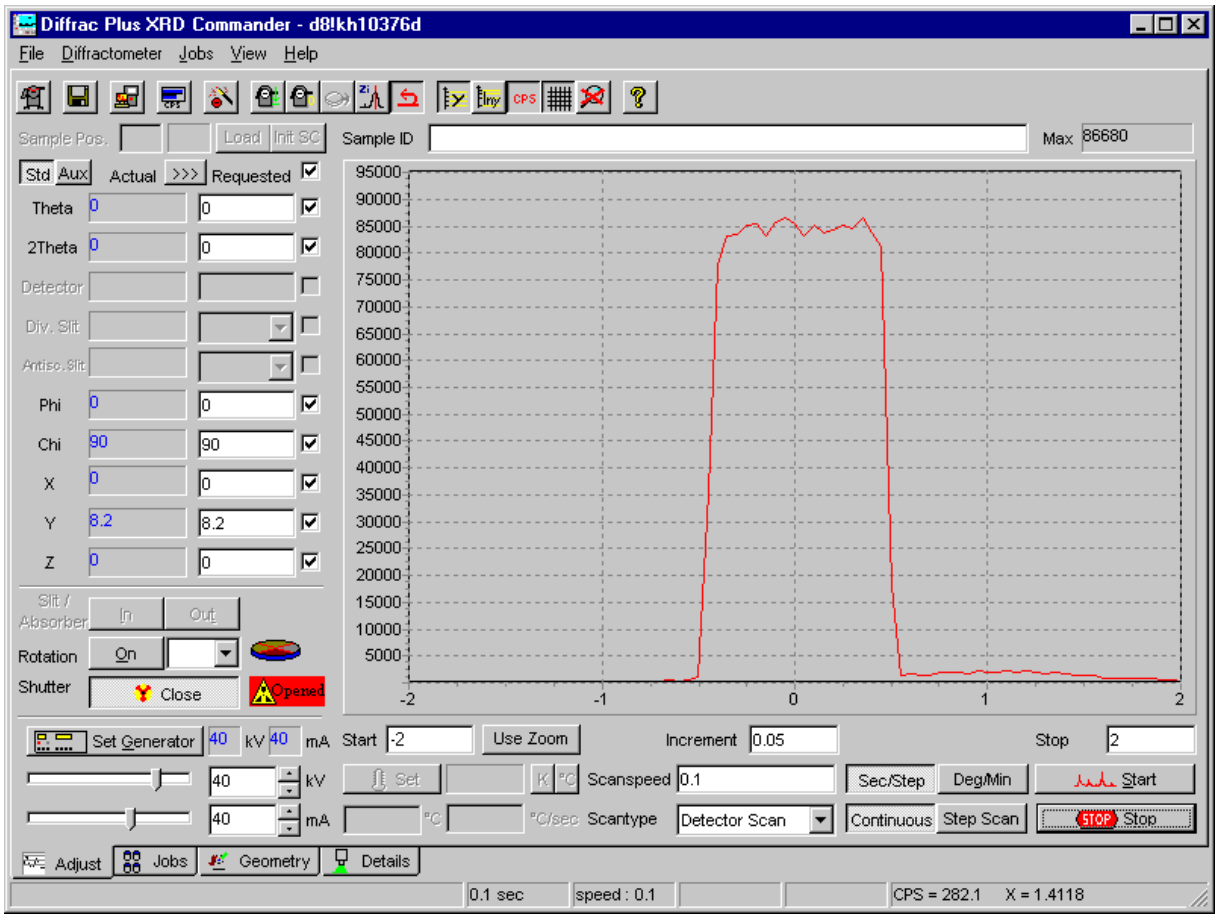


Fig. 7-16: Primary beam of the V10 compressor (6mm detector slit, 0.1mm Cu absorber).

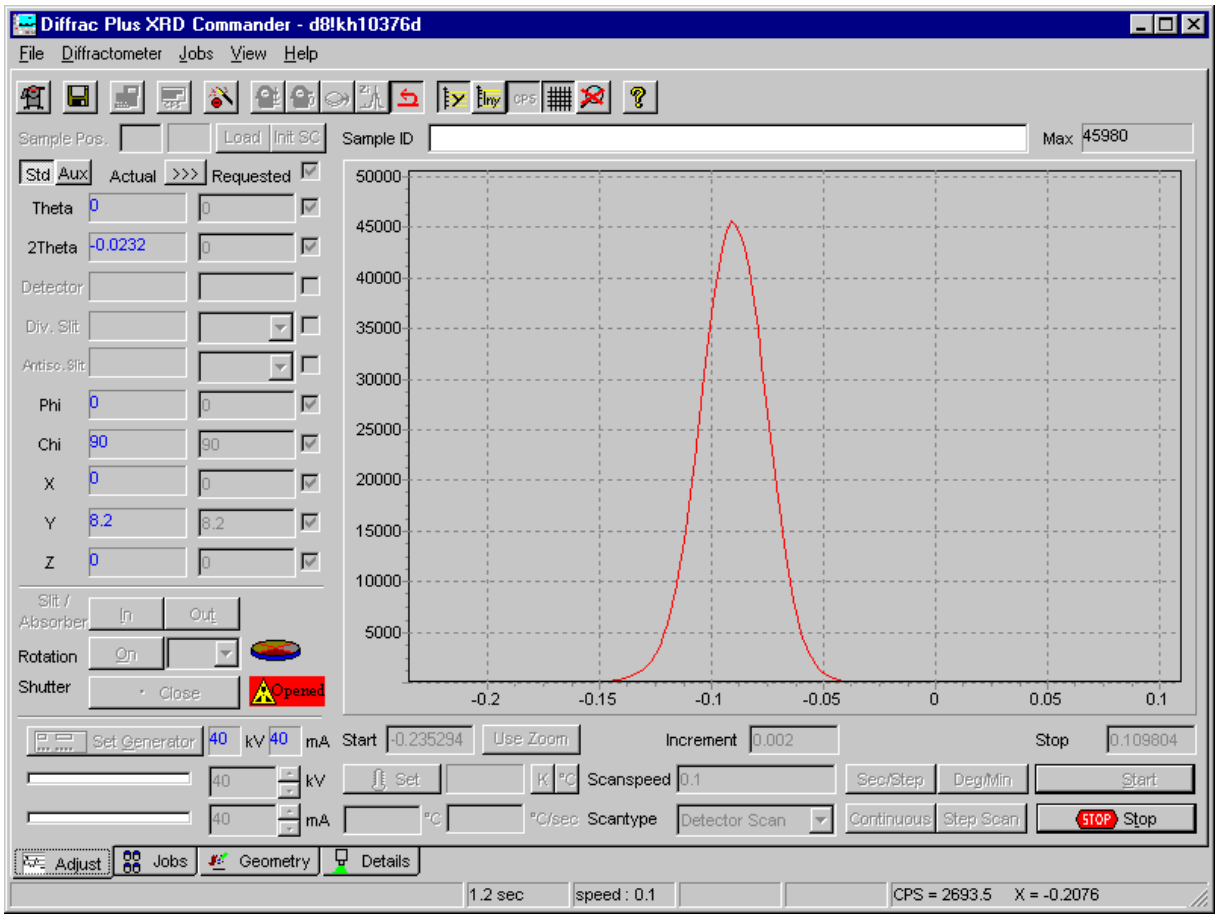


Fig. 7-17: Primary beam of V10 compressor (0.1mm detector slit, 0.1mm Cu absorber).

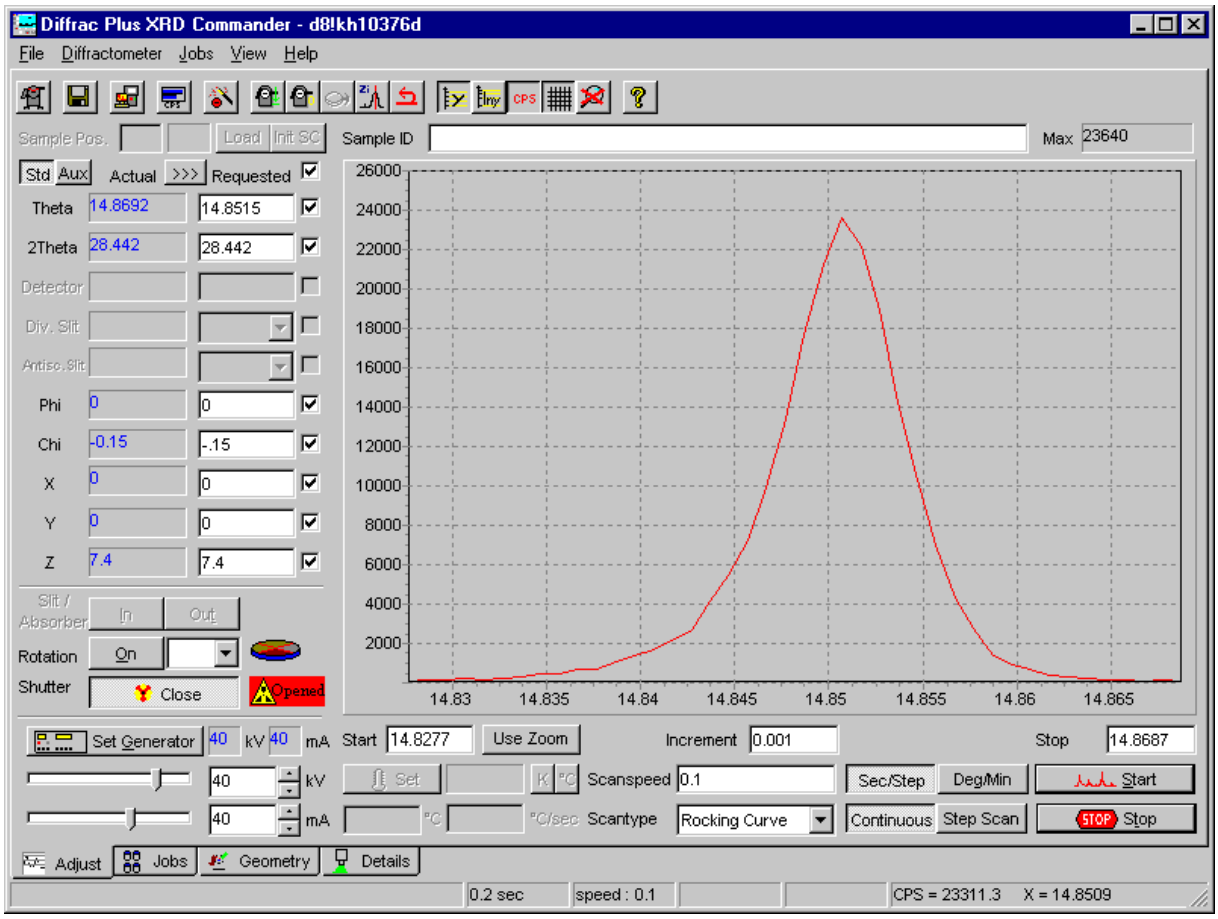


Fig. 7-18: Rocking curve of Si 111 with V10 compressor.

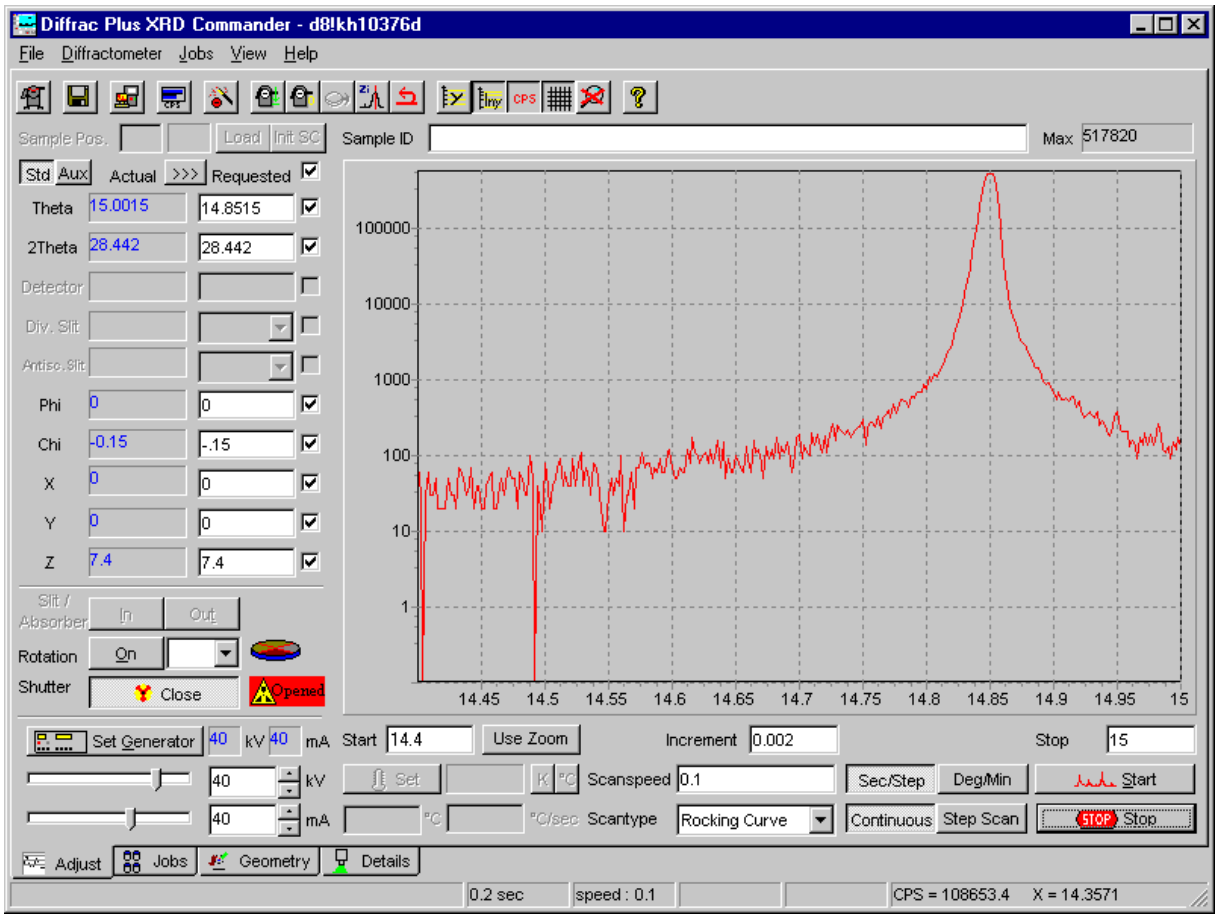


Fig. 7-19: Pure $K\alpha_1$ radiation.

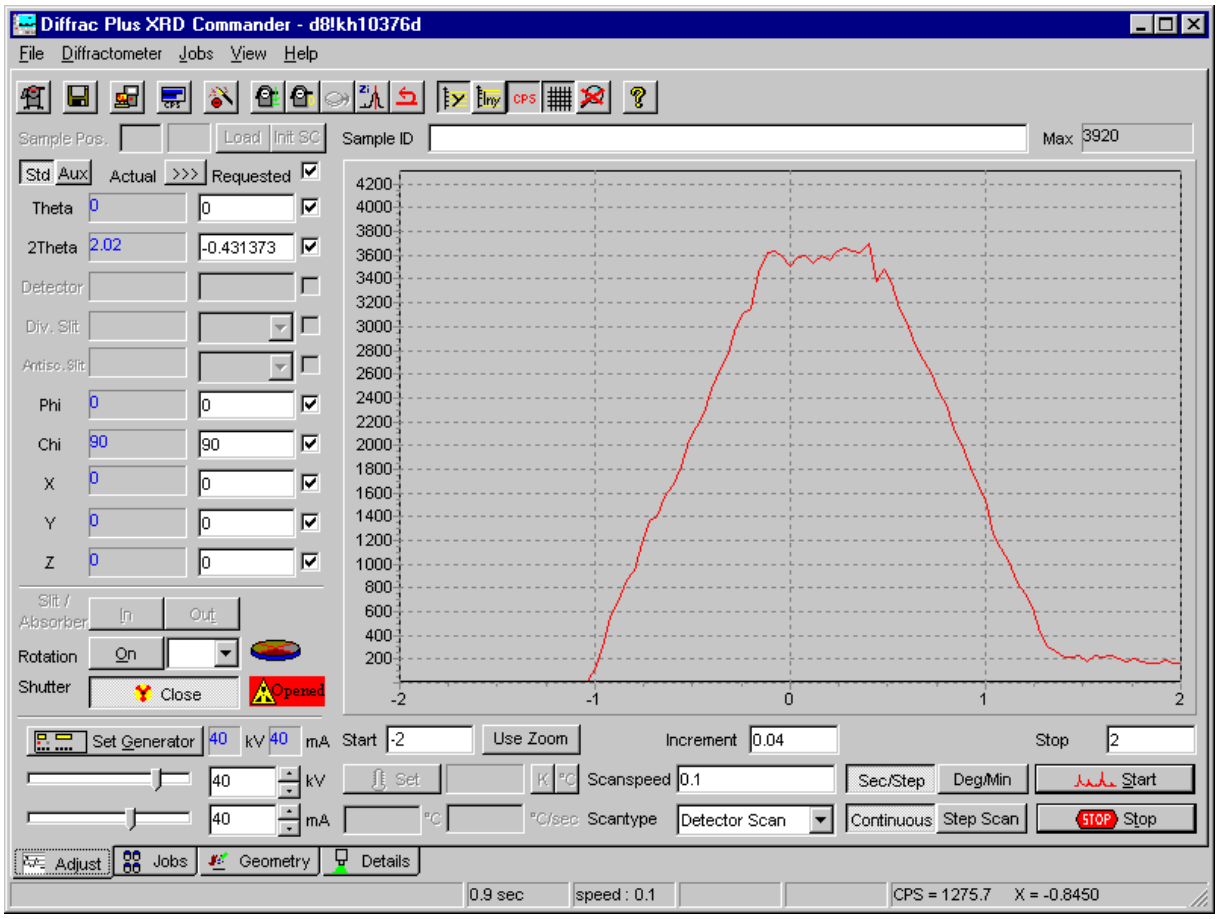


Fig. 7-20: Primary beam intensity of V10 expander (detector scan with 6mm detector slit and 0.2mm Cu absorber).

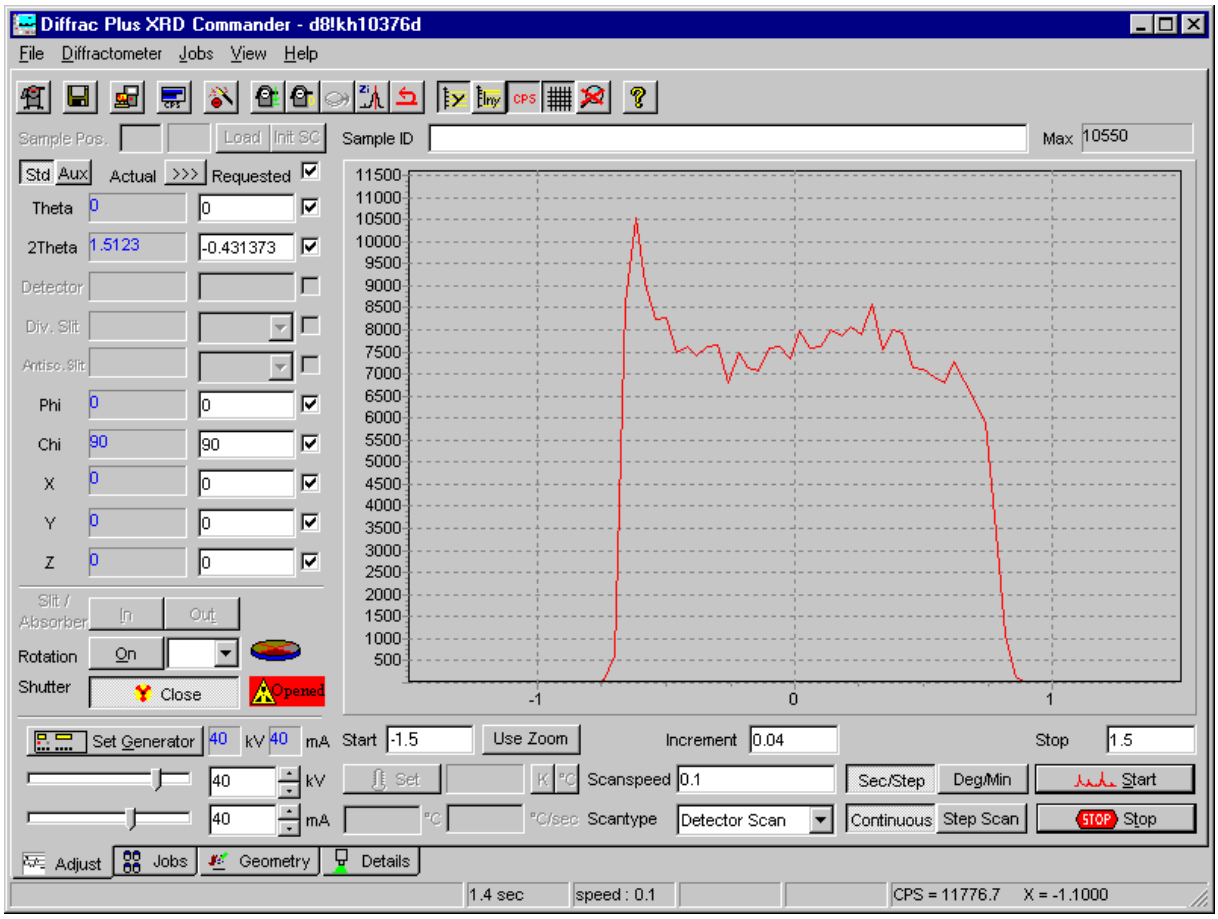


Fig. 7-21: Primary beam width of V10 expander (detector scan with 0.1mm detector slit and 0.1mm Cu absorber).

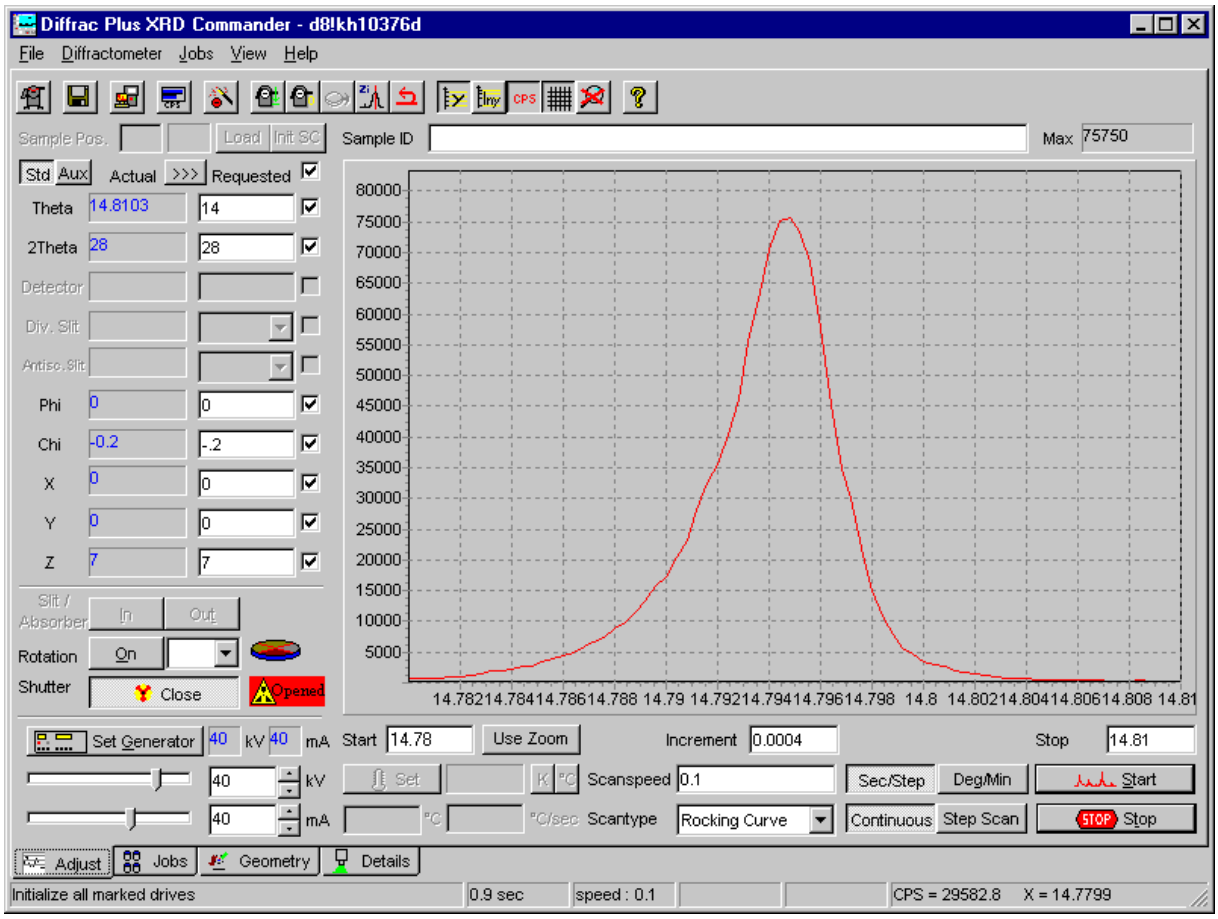


Fig. 7-22: Rocking curve of Si 111 with V10 expander.

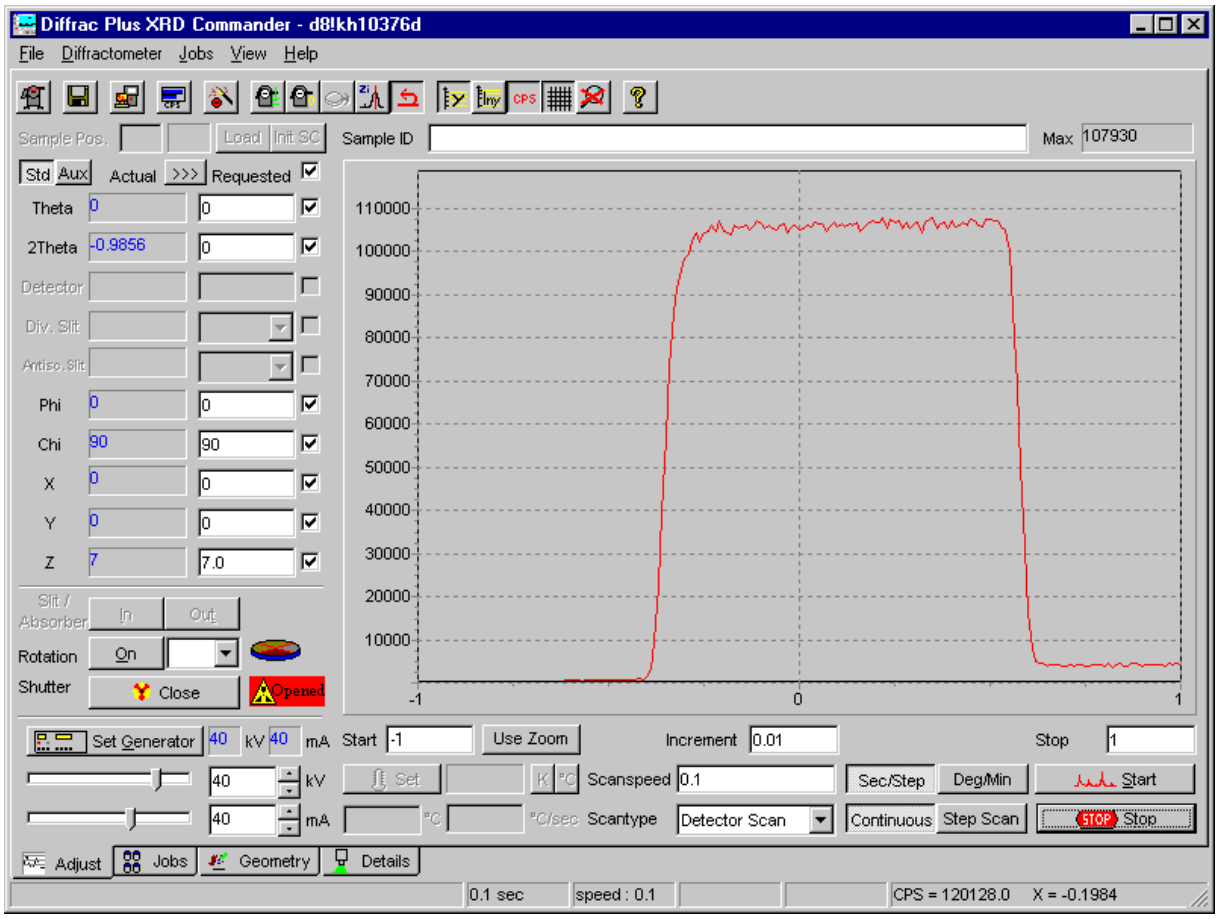


Fig. 7-23: Primary beam intensity of V5 compressor (detector scan with 6mm detector slit and 0.1mm Cu absorber).

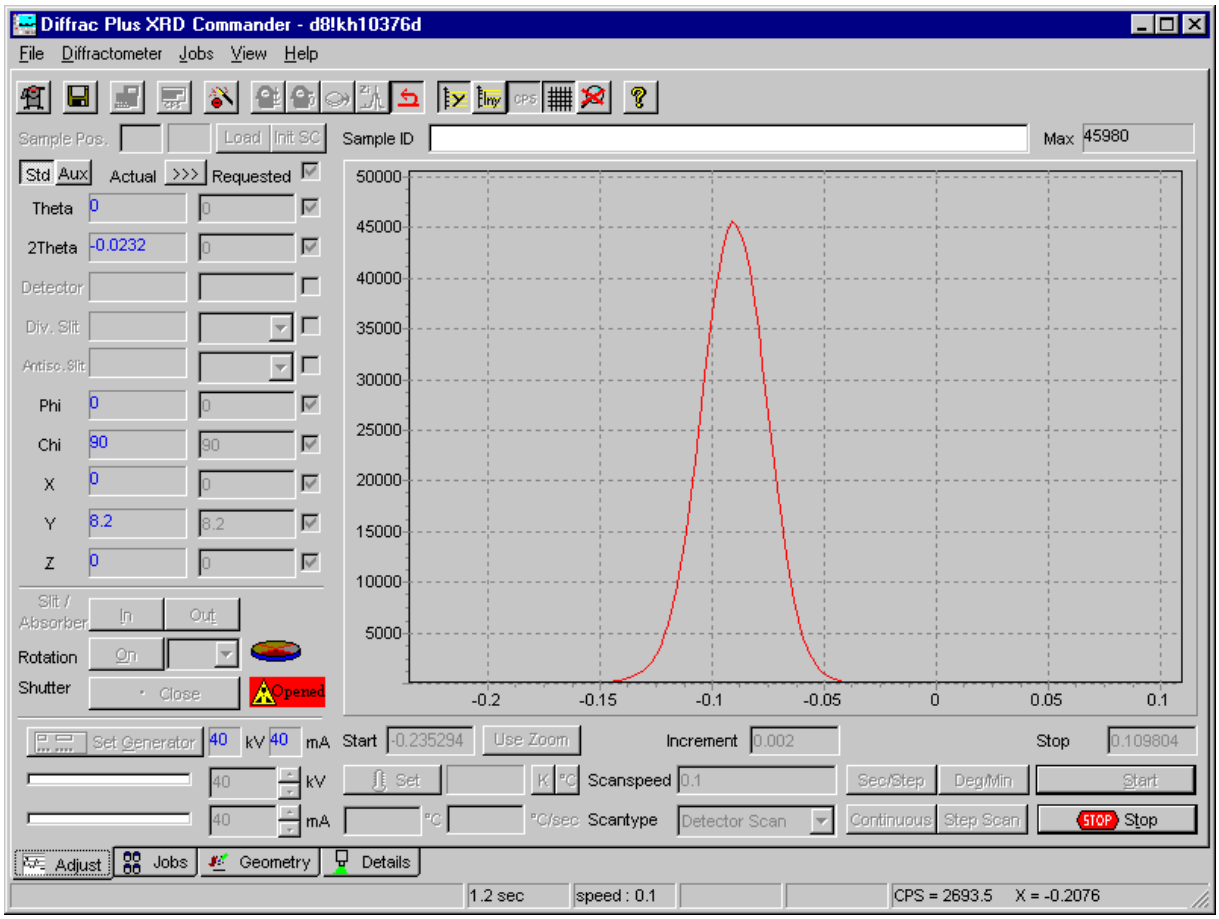


Fig. 7-24: Primary beam width of V5 compressor (detector scan with 0.1mm detector slit and 0.1mm Cu absorber).

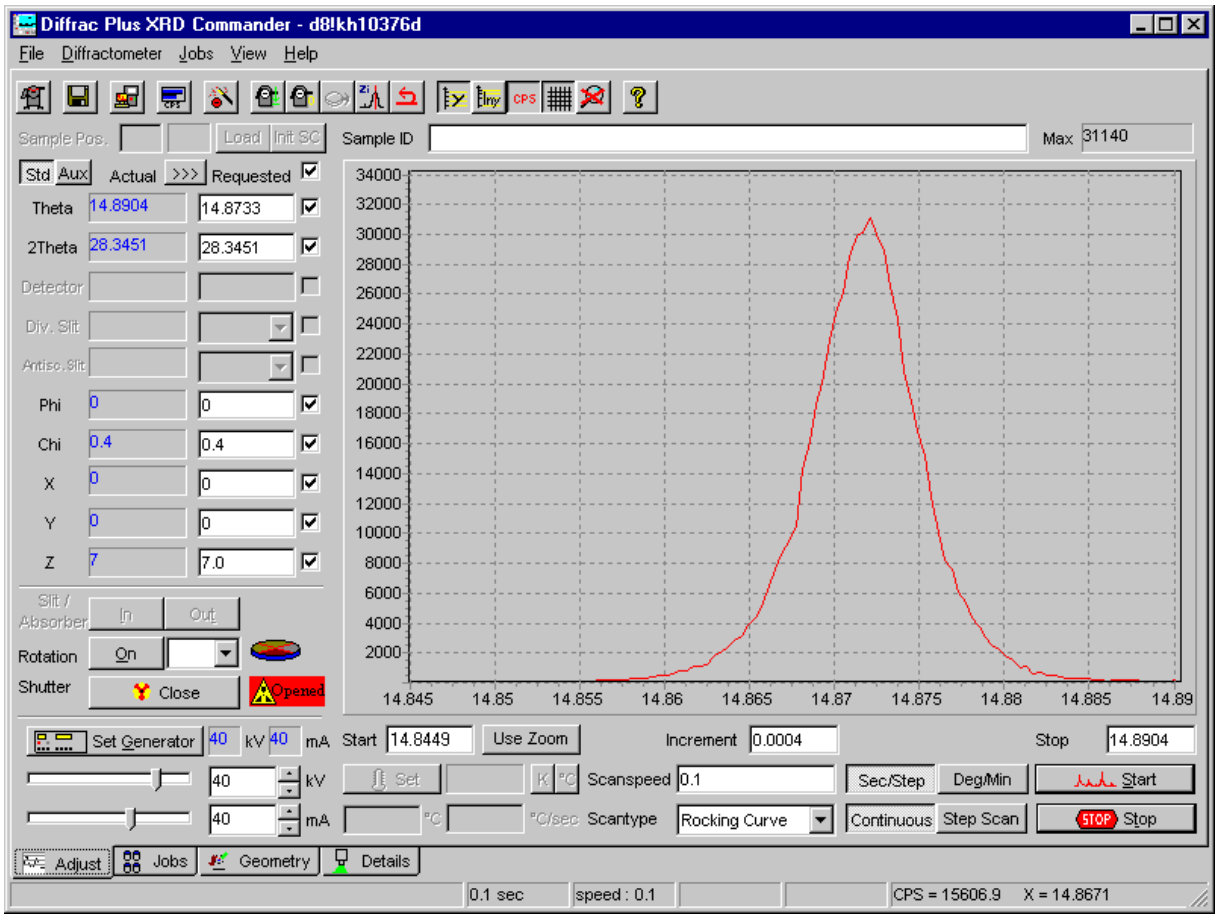


Fig. 7-25: Rocking curve of Si 111 with V5 compressor.

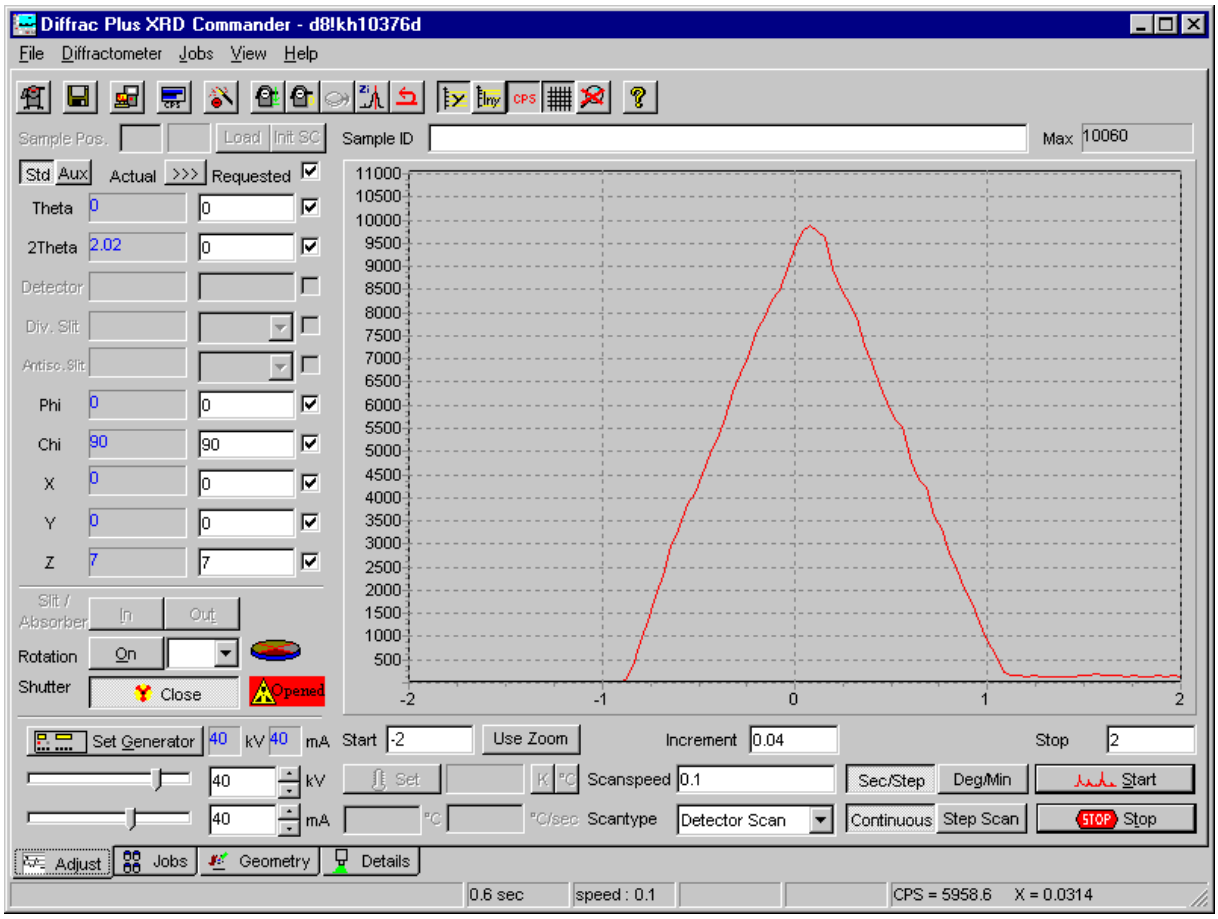


Fig. 7-26: Primary beam intensity of an V5 expander (detector scan with 6mm detector slit and 0.2mm Cu absorber).

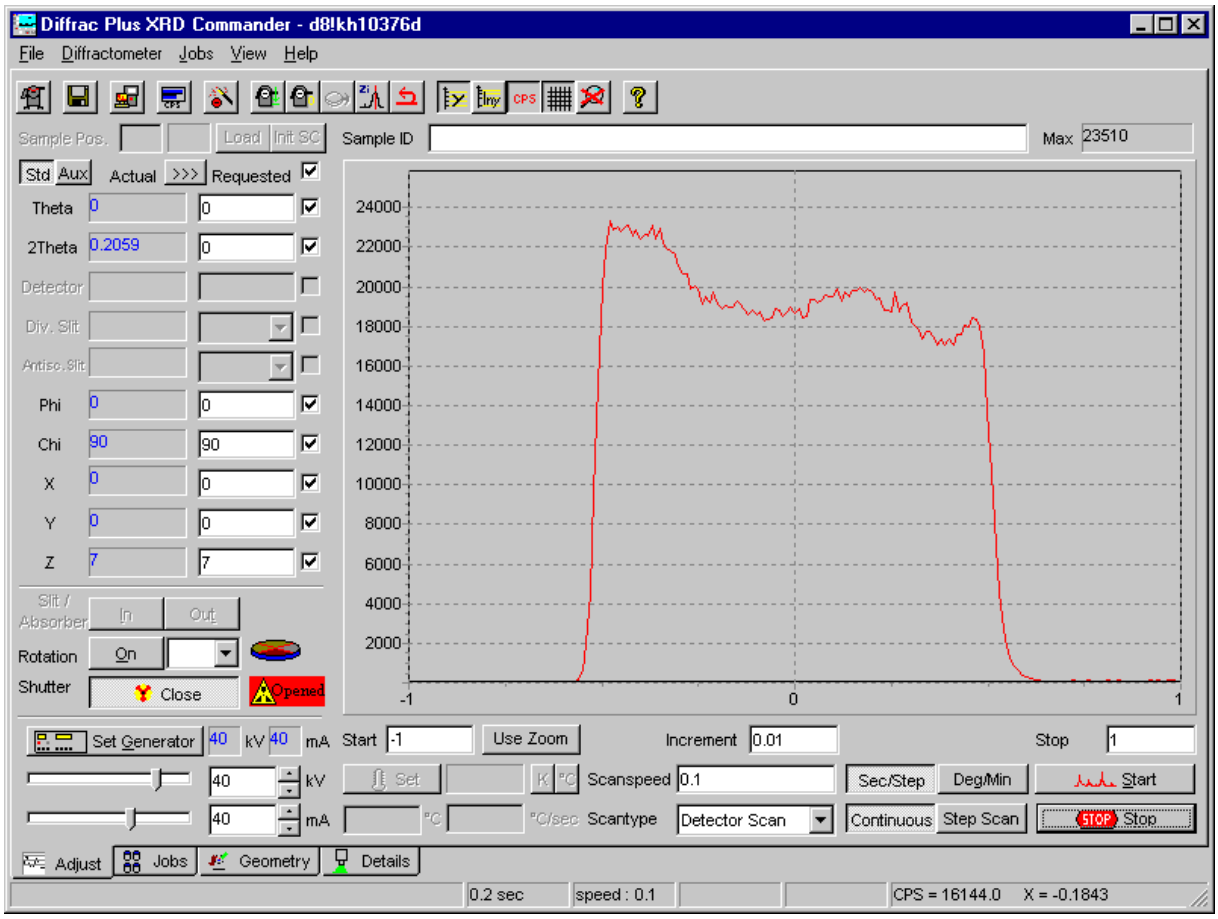


Fig. 7-27: Primary beam width of an V5 expander (detector scan with 0.1mm detector slit and 0.1mm Cu absorber).

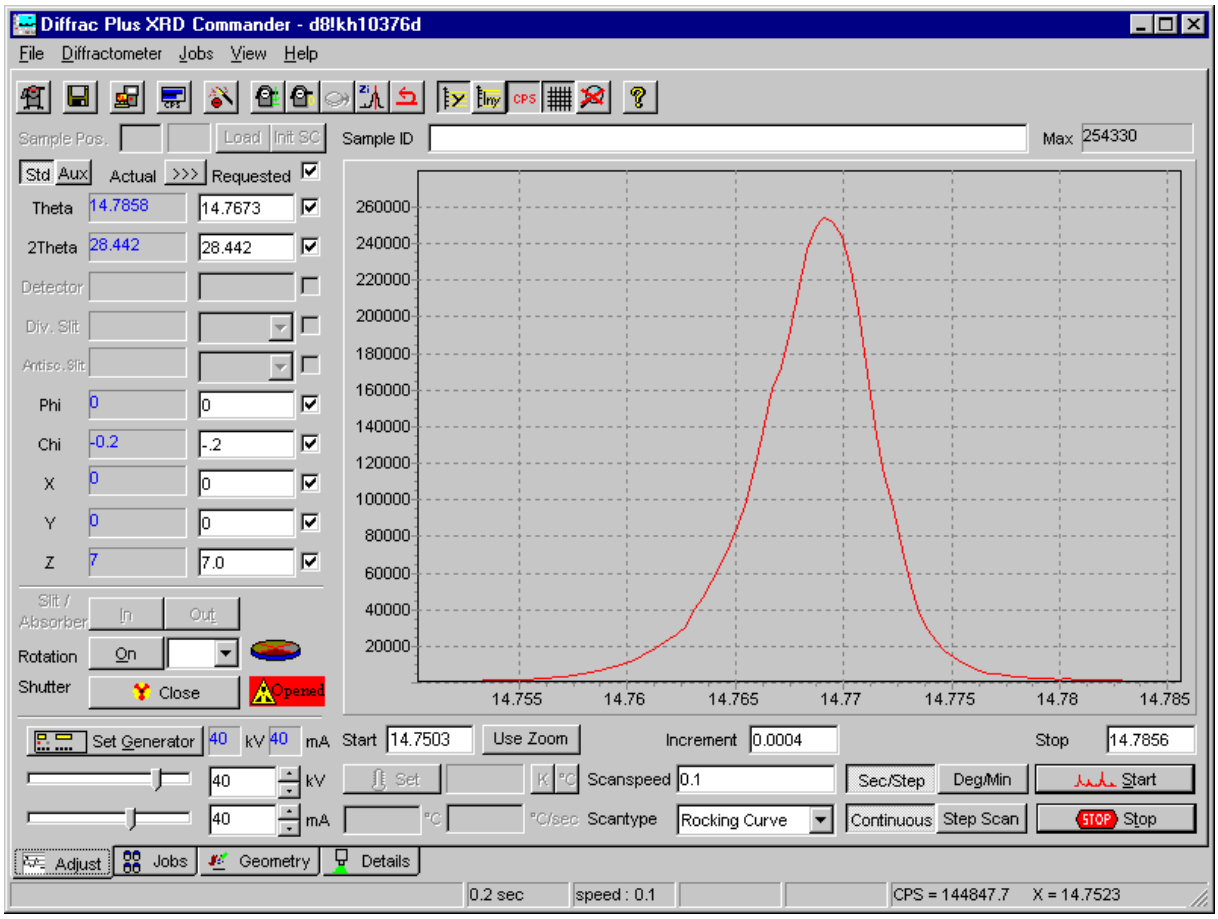


Fig. 7-28: Rocking curve of Si 111 with V5 expander.

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