D8 Series

User Manual

D8 ADVANCE / D8 DISCOVER

Original Instructions

Innovation with Integrity

XRD

We have checked the contents of this manual for agreement with the hardware and software described. Since deviations cannot be precluded entirely, we cannot guarantee full agreement. However, the data in this manual are reviewed regularly and any necessary corrections are included in subsequent editions. Suggestions for improvement are welcome.

All configurations and specifications are subject to change without notice.

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For your Safety

WARNING

X-ray, electrical and mechanical hazards due to incorrect handling

These hazards may cause severe and life-threatening injuries.

- To prevent accidents and system damage consider all safety warnings and instructions within the provided manuals.
- Read and understand **all** manuals provided with the instrument thoroughly!
- Follow the instructions given in the manuals!
- Never manipulate or modify the safety systems of the instrument!
- Strictly observe all national, state, and local regulations for the operation of X-ray systems!

WARNING

Moving parts

Injuries, e.g. by entanglement (fingers, hair, clothing), abrasion, cutting, or crushing are possible.

- Take care of your hands, long hair and clothing!
- Do not check parts in motion by using your hand!
- Stay away from moving components!
- Immediately switch off the system, when you suspect a present failure of the safety system for motorized drives.
- Contact your local Bruker AXS service representative in case of doubt.

⚠ WARNING

X-ray hazard

Exposures to radiation, even for a fraction of a second, cause severe burns and / or fatal injuries or lethal diseases (cancer). Even without visible injuries or sensible pain, lethal diseases might evolve many years later.

- Switch off the system immediately when any part of the enclosure gets damaged!
- Switch off the system immediately when the safety system does not work or does not seem to work correctly!
- Switch off the system immediately when the radiation protection is impaired!
- Never manipulate or modify the safety system or parts of it – this is strictly forbidden!
- Never remove sealed screws or parts which are protected by sealed screws – this is strictly forbidden!
- Never manipulate any interlocks or other safety-relevant devices!
- Contact your local Bruker AXS service representative in case of doubt.

⚠ WARNING

High voltages, up to several tens of thousands Volts DC in:

high-voltage generator | X-ray tube | radiation detectors | high-voltage cables

High risk of lethal electrical shock when components are damaged!

- Disconnect the system **completely** from the mains supply following this instruction step-by-step when any component carrying high voltage has been damaged:
- Step 1: Immediately switch off the system! Use the **Emergency Switch OFF**!
- Step 2: Switch off the **Mains Disconnector** switch!
- Step 3: Disconnect all **Power Supply Lines** from the mains supply!
- Step 4: Switch off the **Automatic Circuit Breaker** or the **External Power Disconnector** switch, located in the vicinity of the instrument!
- Step 5: Contact your local Bruker AXS service representative.

Contents

1 About this Manual

This manual contains general information and guidelines about the Bruker AXS instruments, which have to be observed to ensure personal safety, as well as to protect the product. These notices are highlighted in this manual by the warning symbols and are marked as follows according to the level of danger.

DANGER

DANGER indicates a hazardous situation which, if not avoided, will result in death or serious injury.

This is the consequence of not following the warning.

1. This is the safety condition.

▶ This is the safety instruction.

WARNING

WARNING indicates a hazardous situation, which, if not avoided, could result in death or serious injury.

This is the consequence of not following the warning.

1. This is the safety condition.

▶ This is the safety instruction.

CAUTION
CAUTION indicates a hazardous situation, which, if not avoided, may result in minor or moderate injury.
This is the consequence of not following the warning.
1. This is the safety condition.
▶ This is the safety instruction.

NOTICE
NOTICE indicates a property damage message.
This is the consequence of not following the notice.
1. This is a safety condition.
▶ This is a safety instruction.

SAFETY INSTRUCTIONS
SAFETY INSTRUCTIONS are used for control flow and shutdowns in the event of an error or emergency.
This is the consequence of not following the safety instructions.
1. This is a safety condition.
▶ This is a safety instruction.

This symbol highlights useful tips and recommendations as well as information designed to ensure efficient and smooth operation.

2 Getting Started

2.1 Starting the Instrument

Refer to the figure below for the instructions in this section.

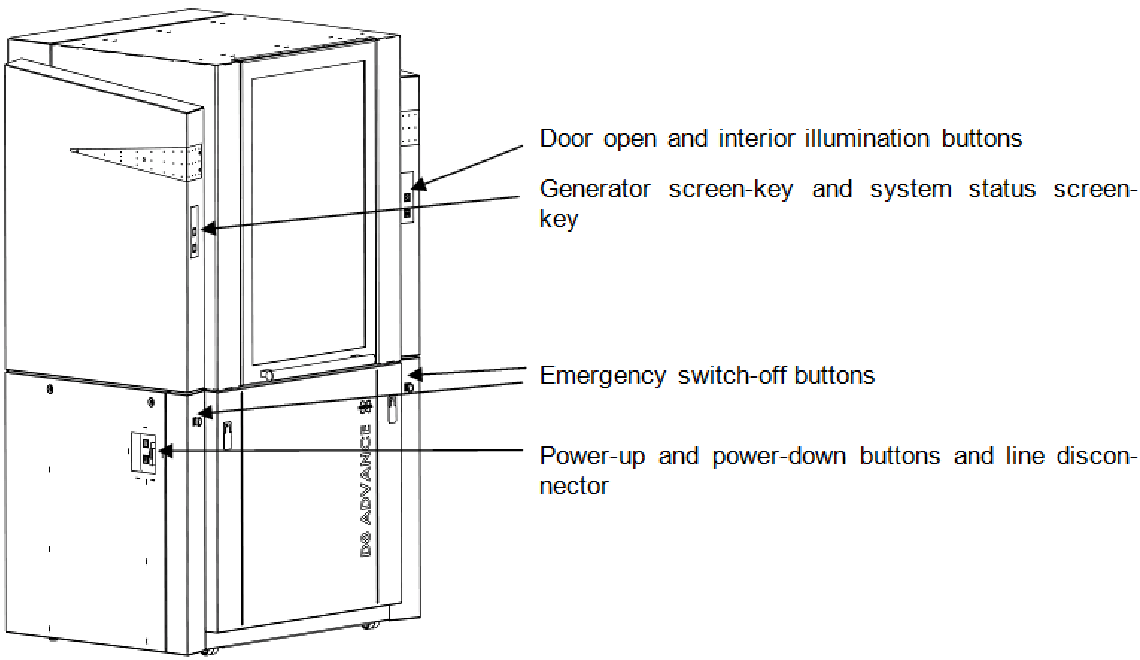


Figure 2.1: Controls of D8 ADVANCE Diffractometer

To start the Bruker AXS D8 ADVANCE diffractometer, follow these steps:

1. Turn the line disconnector, which is located in the niche in the lower left side of the instrument, clockwise from “**0**” to “**1**”.
   - ➨ The power supply to the instrument and its electrical components is switched on.
2. Press the power-up button in the niche in the lower left side of the instrument. This is the green pushbutton inscribed with “**I**”.
   - ➨ The generator screen-key to the left of the radiation enclosure on the front side of the instrument illuminates in white and the system status screen-key directly below it start blinking with a white light. After a few seconds, the blinking stops and an “**I**” symbol appears on the generator screen-key, indicating that the system is ready for X-ray production.

If, however, an error is detected, a symbol identifying the error will appear on the generator screen-key. For a list of error symbols and their meanings, refer to the table of generator screen-key states at the end of this section. Errors can be corrected using the TOOLS software module of the Bruker AXS DIFFRAC.SUITE MEASUREMENT CENTER (short: DIFFRAC.SUITE).

1. If no error has been detected or after detected errors have been repaired, press the generator screen-key to activate the X-ray generator.

   - ➨ The generator screen-key turns yellow and starts blinking, indicating that the cathode is heating up. After a few seconds, the blinking stops and an X-ray symbol appears, indicating that X-ray generation has started. In this phase, the generator voltage is ramped up to a value that has been set in the hardware configuration. When this value is reached, the X-ray symbol is replaced by its own negative image, indicating that the generator is ready for measuring operations.

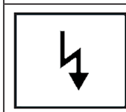
⇨ You can now prepare and start a scan.

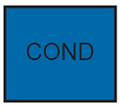
2.1.1 Upper Screen Key (X-ray Generator Status and Control)

The different states of the generator are indicated by the upper screen key on the left side of the instrument's front panel. The meaning of the symbol and the background color that appear on the screen-key are described in the table below.

Table 2.1: Upper screen key - X-ray generator status and control

Symbol	Background Color	Description / Meaning
	White	Shown during startup sequence of the instrument
	White	**Switch-on symbol** Shown when the X-ray generator is switched off and is ready to be switched on. • Press the symbol once to start up the X-ray generation.
 **Blinking**	Yellow	**Heating on** Blinking yellow when the generator's tube heating is enabled and no X-rays are being generated. **Constant yellow** when the switch off circuit has been closed, but X-rays are not being generated. • Press once to switch off the generator.
 **Blinking**	Yellow	**X-rays on, generator ramping up / down** Shown when the X-rays are switched on and the generator is ramping its power (yellow background). • Press once to switch off the generator.
	Yellow	**X-rays on, generator ready** Shows that the generator has reached its set values (yellow background).

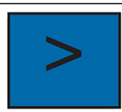
Symbol	Background Color	Description / Meaning
		• Press once to switch off the generator.
	Red	**X-ray safety circuit error** The X-ray generator will be switched off if a safety error occurred. For details look into the safety board in the diagnostic plug-in **TOOLS**, which can be found within the DIFFRAC.SUITE MEASUREMENT CENTER.
 **Blinking**	White	**X-ray safety circuit not closed** Safety Warning. The X-ray safety circuit is not closed. This is not an error, but X-rays cannot be generated. See **TOOLS** for further details.
 **Blinking**	Green	**Selftests in progress** Safety board selftest / Enclosure selftest in progress
 **Blinking**	Yellow	**Generator selftest in progress** X-ray warning displays may be enabled for a very short time.
	Red	**System error** Either the generator or one of its connected components (tube, safety circuit, and water cooling system) have an error. For details look into the X-ray generator in **TOOLS** (plug-in DIFFRAC.SUITE MEASUREMENT CENTER). When the problem is fixed, the symbol will disappear and the **Switch ON** symbol will appear (if no subsequent error is pending)
	White	**Generator warning**

Symbol	Background Color	Description / Meaning
Blinking		Check **TOOLS** for further details. Either the generator is in an undefined state or the tube was not detected successfully.
	Red	**Water Cooling Error** The water cooling system switched off the generator because of an error in the cooling system. For a detailed error message, look into the water cooling unit in **TOOLS** (plug-in DIFFRAC.SUITE MEASUREMENT CENTER). When the problem is fixed, the symbol will disappear and the **Switch ON** symbol will appear (if no subsequent error is pending)
 **Blinking**	Blue	**Tube conditioning enabled** When this symbol blinks in blue, the tube conditioning is enabled. To abort this action, press the button once. Then you are able to proceed operation in normal mode.

2.1.2 Lower Screen Key (System Status and Error Messages)

The different states of the system and the error messages are indicated by the lower screen-key on the left side of the instrument's front panel. The meaning of the symbol and the background color that appear on the screen-key are described in the table below.

Table 2.2: Lower screen keys - System Status and Error Messages

Symbol	Background Color	Description / Meaning
 **Blinking**	White	**Instrument is booting** Blinking white light shows that the instrument is booting.
	White	**Ready for operation** Constant white light signals that the instrument has booted and is ready for operation.
	Green	**Controlled by a client** Constant green light signals that the instrument is controlled by a client (e.g. Measurement Server).
 **Blinking**	Red	**Alignment mode active** This symbol is shown red flashing if the Alignment mode is active.
 **Blinking**	Blue	**Measurement in progress** This symbol is shown blue flashing if a measurement is in progress.
 **Blinking**	Green	**Door open** Instrument doors are open.
	Red	**Sample changer error** The built-in sample changer has an error and needs a user interaction before proceeding (for more detailed description of the pending error and repair see **TOOLS**).

Symbol	Background Color	Description / Meaning
	Red	**Detector error** At least one detector has an error.
	Red	**Drive collision** At least two drives have collided. For repair see **TOOLS.**
	Red	**Drive error** At least one drive has an error. For repair see **TOOLS.**

Pending system alarms or warnings (red display elements) will be usually indicated as written information by the application software on the computer screen. In addition, the diagnostic plug-in **TOOLS** can be used to get a complete diagnosis of any present alarms or warnings. Also, repairing is possible with help of this program.

2.2 Placing Sample in Sample Changer / Loading Samples by Hand

If the instrument is equipped with a sample changer, place the sample in a sample position in the tray or magazine of the changer. It will then be loaded automatically later via the **Start** (**Scan**) function of the COMMANDER module of the DIFFRAC.SUITE.

If the instrument is not equipped with a sample changer, load the sample by inserting it in the sample stage of the diffractometer.

Because the example scan described below involves the use of a Corundum sample, it is recommended that you use a Corundum sample for your trial measurement.

2.3 Starting the Software

Bruker AXS X-ray instruments are delivered with the DIFFRAC.SUITE, a set of software applications used to create and start measurements and perform other measurement-related tasks on the instruments. The DIFFRAC.SUITE forms the client component of a client-server system. Communication with the X-ray instruments takes place via the Measurement Server. When you start the DIFFRAC.SUITE, the Measurement Server will start automatically in the background.

To log into the DIFFRAC.SUITE, you must be registered in the database and assigned to a user group. This task is accomplished by a person who has been assigned the role of **Lab Manager** or **IT Administrator**. He/she will give you a password that you can use to log in for the first time.

Before working with the DIFFRAC.SUITE, you must select the instrument you want to work with, establish a connection between it and the Measurement Server, and then enable the Measurement Server to get control of the instrument. You do this via the dialog boxes **Select Instruments** and **Status Window** of the Measurement Server. To learn how to setup the system with the Measurement Server, see the Chapter “Measurement Server” in the *DIFFRAC.SUITE User Manual*.

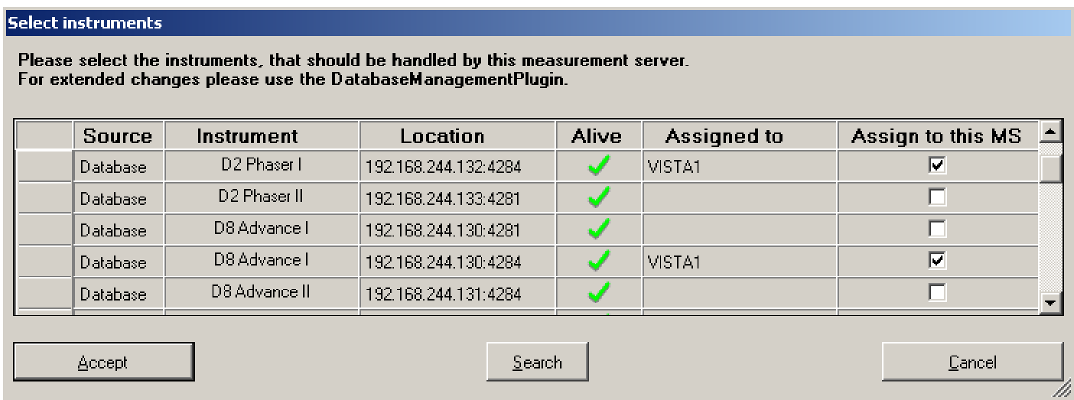


Figure 2.2: Dialog box Select instruments of Measurement Server

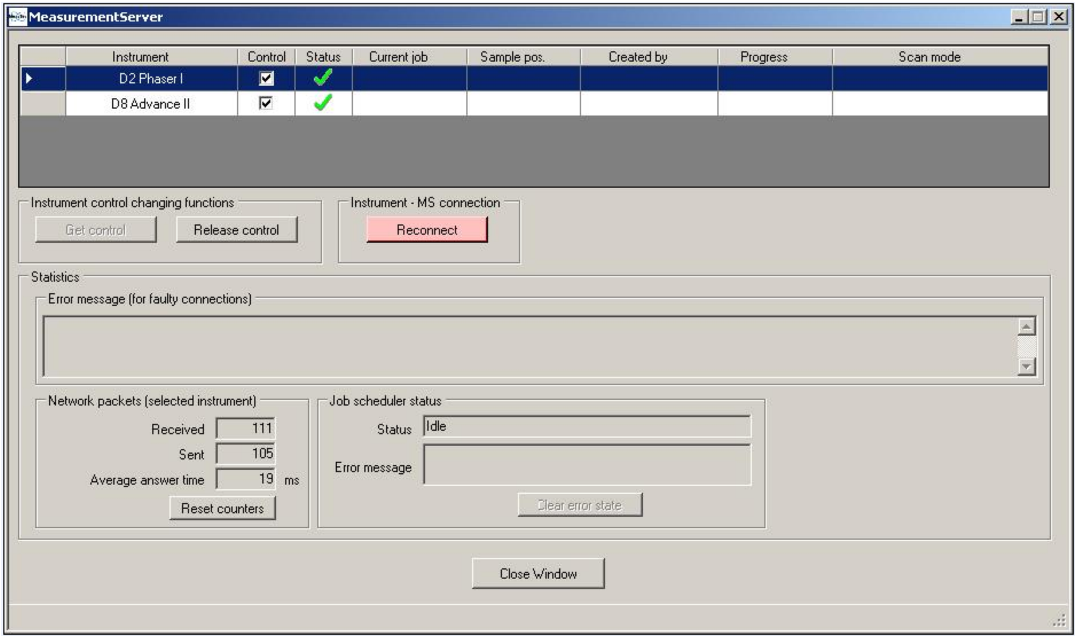


Figure 2.3: Dialog box Status window of Measurement Server

2.4 Initializing the Drives

After each new start of the instrument, its drives must be initialized before you can create and start measurements. All of these tasks can be accomplished using the COMMANDER module of the DIFFRAC.SUITE.

To move to the COMMANDER page, click on the corresponding page tab or on the corresponding icon in the outlook bar of the DIFFRAC.SUITE user interface.

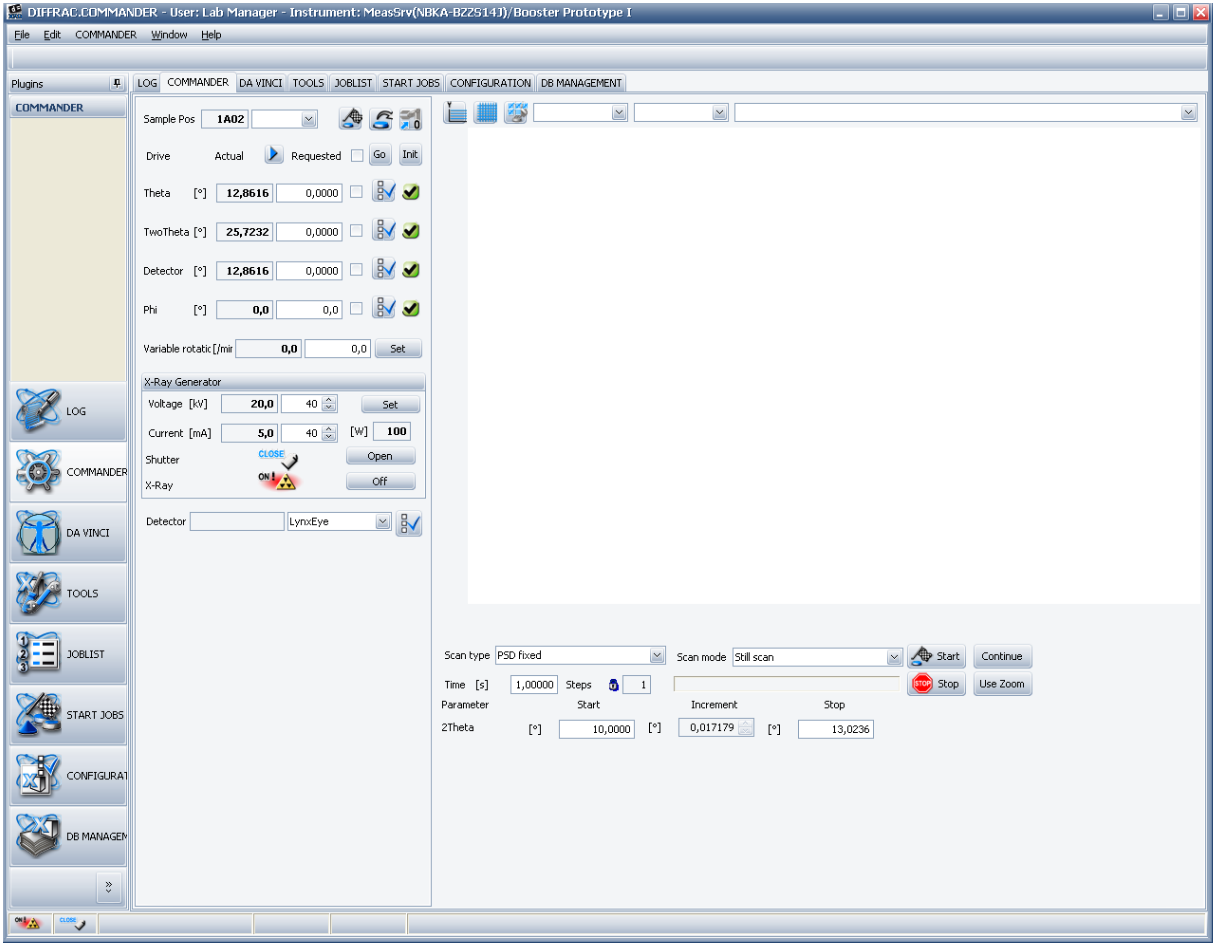


Figure 2.4: COMMANDER interface – appearance after connection established with instrument

Establish a connection between COMMANDER and the instrument via the item **Connect** of the **File** menu according to the instructions given in the section “Getting a Connection to an Instrument with the Measurement client” of the *DIFFRAC.SUITE User Manual*.

When this connection has been established, the illuminated system status screen-key on the instrument will change from white to green. This means that commands can now be sent to the instrument from COMMANDER.

The drives of the connected instrument are listed in the **Drive Control** area of the COMMANDER page.

To initialize the drives to be used in the measurement,

1. check the corresponding check boxes individually or
   click on the check box next to the **Go** button to check all the check boxes as a group.
2. Then click on the **Init** button.

For more detailed information on the initialization of drives using COMMANDER, see the section “Drive Control” in the chapter COMMANDER of the *DIFFRAC.SUITE User Manual*.

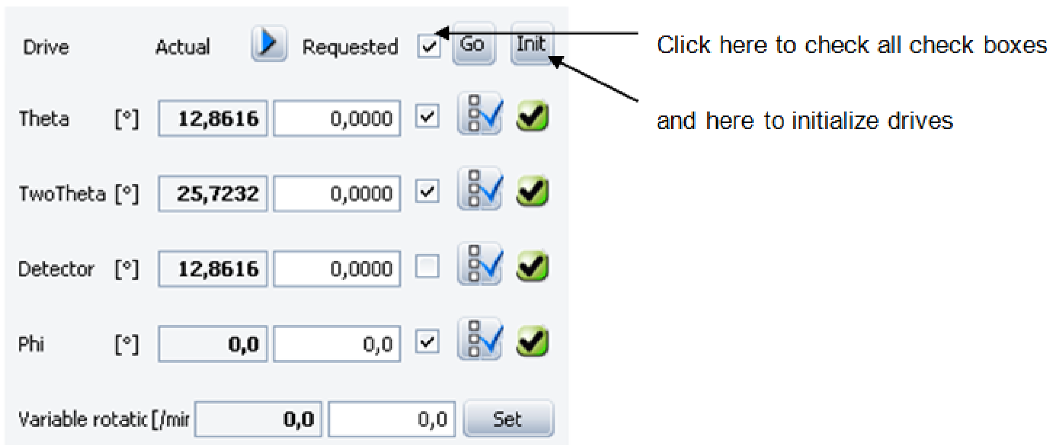


Figure 2.5: Drive Control area of COMMANDER page

Drives can also be initialized using the TOOLS module of the DIFFRAC.SUITE. However, since most of the software tasks described in this document can be carried out in COMMANDER, drive initialization with TOOLS will not be discussed here.

2.5 Performing a Scan

The COMMANDER plug-in is used to create and start measurements (called “scans” in this application module) on an individual basis. In the following, creating and starting a scan on a Corundum sample is described as example. The instrument used is a theta/theta system and the anode material is Copper.

1. Set the voltage and current values for the X-ray generator to 40 kV and 40 mA respectively in the **Generator Control** area of the COMMANDER page. These are the values most suited to a Copper anode. Then click on the **Set** button to set the values.

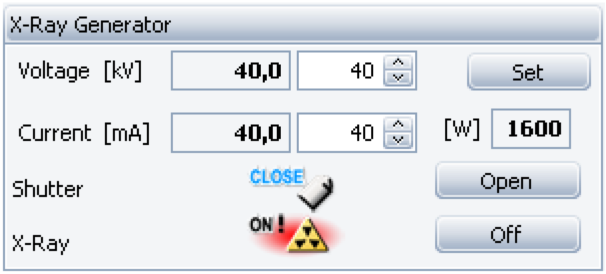


Figure 2.6: Generator Control area of COMMANDER page

2. Create the scan by making the following settings in the **Scan Control** area.

Scan type	Coupled Theta/TwoTheta scan
Scan mode	Continuous scan
Time [s]	0.1
2Theta Start [°]	34
Increment	0.01 or smaller
2Theta Stop [°]	approx. 36

In this example, a peak at 35.149° on the 2theta scale for a Corundum sample measured using a Copper anode and with a (104) Kα reflection is expected. The angular position of the peak corresponds to the angle formed by the diffracted X-ray beam and the path of the incident beam.

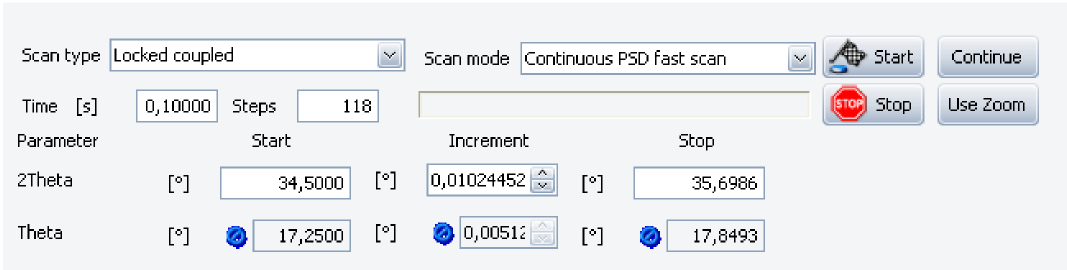


Figure 2.7: Scan Control area of COMMANDER page

The table below shows the expected positions of the maximum intensity peaks for a Corundum sample in dependence upon the anode material and the reflection.

		Corundum (A13B73) (Bragg/Brentano)		
		(012) Kα reflection	**(104) Kβ reflection**	**(104) Kα reflection**
Cu	Position (deg)	25.576	31.8	35.149
	Energy (keV)	8.05	8.9	8.05
Cr	Position (deg)	38.41	48.24	53.33
	Energy (keV)	5.41	5.95	5.41
Co	Position (deg)	29.79	37.05	41.05
	Energy (keV)	6.93	7.65	6.93
Mo	Position (deg)	11.68	14.25	15.95
	Energy (keV)	17.5	19.6	17.5

If the instrument is equipped with a sample changer, you must initialize it before it can be used. Sample changers must be initialized after each new start of the instrument or after the occurrence of an error.

To initialize the sample changer,

1. click on the button  in the **Sample Changer Control** area.

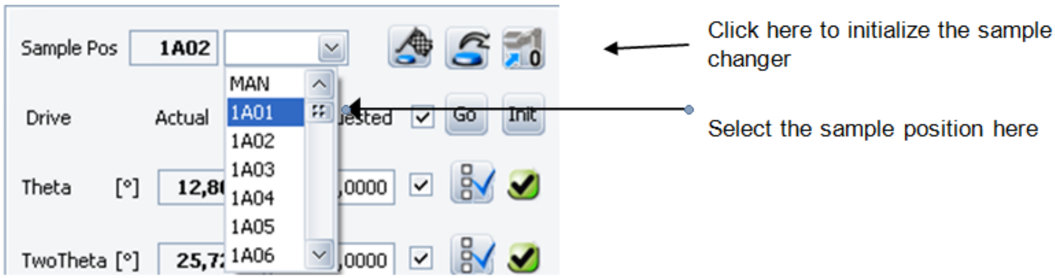


Figure 2.8: Sample Changer Control area

If the instrument is not equipped with a sample changer, the **Sample Changer Control** area will not appear on the COMMANDER page and you can omit this step. In this case, the sample must be loaded manually as described in section *Placing Sample in Sample Changer / Loading Samples by Hand [▹ 19]*.

If the instrument is equipped with a sample changer, select the sample position in the changer from the scroll list in the data entry field in the **Sample Changer Control** area. Select the position in which you have placed the samples. For further information on selecting the sample position, see the Section “Sample Changer Control” in the COMMANDER chapter of the user manual for the *Bruker AXS DIFFRAC.SUITE*.

1. Click on the **Start** button in the **Scan Control** area to start the measurement.

⇨ The > symbol appears on the system status screen-key at the front of the instrument. At the same time, the screen-key changes from green to blue and starts blinking, indicating that a scan is in progress. The results of the scan are displayed in real time and in chart form in the Chart area of the COMMANDER page.

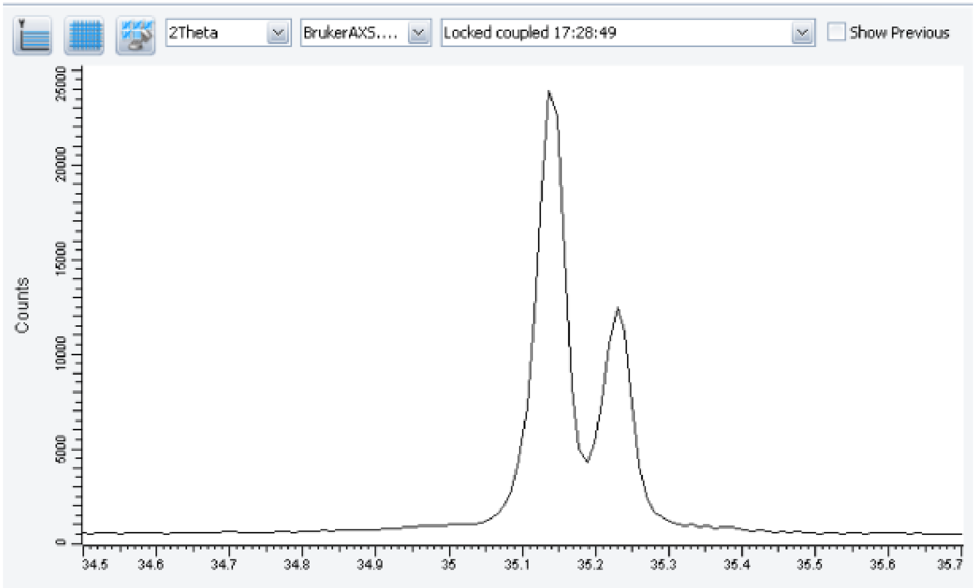


Figure 2.9: Results of a coupled Theta/TwoTheta scan using a Corundum sample

For exact evaluation of the peak position and other parameters you can save the results data to a file and then use the DIFFRAC.EVA software for evaluation.

Saving the Results of Scans

You can save the results of a scan via the item **Save Rawfile** in the **File** menu. When you select this option, the plotted graph is saved to a **brml** file in a predefined directory or a directory of your choice.

This file can later be opened and the chart can be displayed for evaluation purposes using the DIFFRAC.EVA software package.

Evaluation of Scans

The results of scans can be evaluated using the DIFFRAC.EVA, DIFFRAC.TOPAS and DIFFRAC.LEPTOS software programs. To learn about these programs, refer to the relevant user manual.

2.6 Shutting Down the Instrument

To shut down the instrument, follow these steps.

1. Press the generator screen-key to ramp down the generator.
   - ➨ The negative X-ray image on the screen-key disappears and is replaced by a normal X-ray image as the generator voltage is reduced until X-rays generation ceases. At this point the screen-key reverts to white and the "**I**" symbol reappears. You can now switch off the instrument.
2. Press the power-down button, which is the white button inscribed with **Standby** in the niche on the lower left side of the instrument.
   - ➨ The instrument is powered down, that is, switched into standby mode. The X-ray generator and all drives stop immediately.

NOTICE

Shortened life time of X-ray tube and high voltage generator

Do not force shut down the instrument while X-rays are being generated by the high-voltage generator, as indicated by the X-ray symbol on the generator screen key. This can shorten the service lives of the X-ray tube and the high voltage generator.

Each time a sample is removed, the high voltage is switched off automatically. The high voltage may also be switched off using the control software.

Shutting Down the Instrument Completely

If you want to shut down the instrument completely for servicing or safety purposes,

1. turn the line disconnector anti-clockwise from "**I**" to "**0**".
   - ➨ The power supply to the instrument and all its electrical components are shut off. If necessary, you can now remove the connector from the mains socket.

NOTICE

Components damage

Before performing a complete shut-down, shut down all connected devices (heating chambers, LYNXEYE or VÅNTEC-1 detector and controller) according to the instructions provided in the related User Manuals (see chapter *References [▸ 189]*).

3 General Physical Principles

3.1 Production of X-rays

X-rays are photons that are emitted when electrons strike a metal target and interact with the electrons orbiting the nuclei of the metal atoms. The electromagnetic waves in the λ-range 0.1 – 2 Å are emitted in all directions.

In X-ray tubes, electrons are emitted by a heated cathode, accelerated, and strike an anode material. The cathode consists of a tungsten filament. For the generation of X-rays, high voltages of 20 – 50 kV are required. Much of the energy supplied to the tube is transferred to heat, while less than 1 % of it is converted into X-rays. The anode consists of a metal (for example, Mo, Cu, Co or Cr) that produces a characteristic wavelength. A water cooling system prevents it from overheating.

X-ray tubes usually have two small Beryllium windows by which the X-rays escape to the outside. The tube is partly clad with a heavy absorbing material such as lead to prevent the X-rays emitted in other directions from penetrating to the outside.

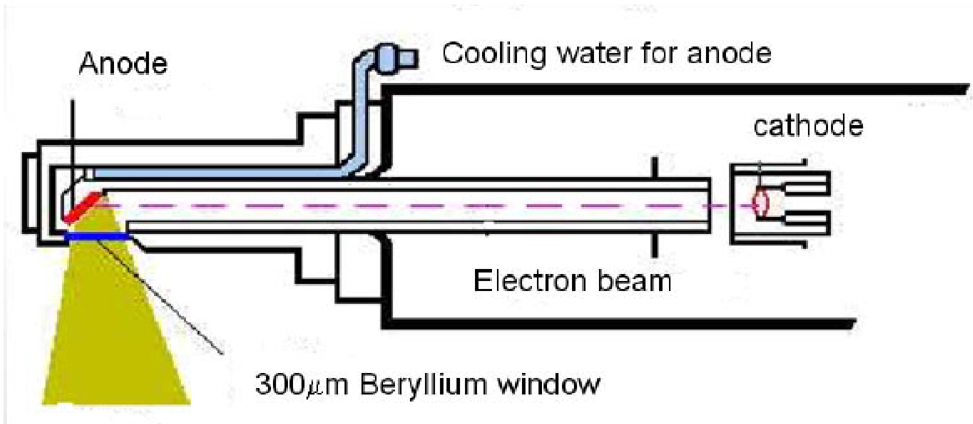


Figure 3.1: Schematic diagram of X-ray tube

3.1.1 Continuous Spectrum

The accelerated electrons collide with atoms of the anode. They are stopped on impact, in which case all their energy is converted into X-rays, or they are decelerated in steps, in which case a part of their energy is released in form of low-energy X-rays. This process is repeated when the electron deviated after an initial coulombian interaction interacts with the electric field of other atoms. This occurs until all energy is used up.

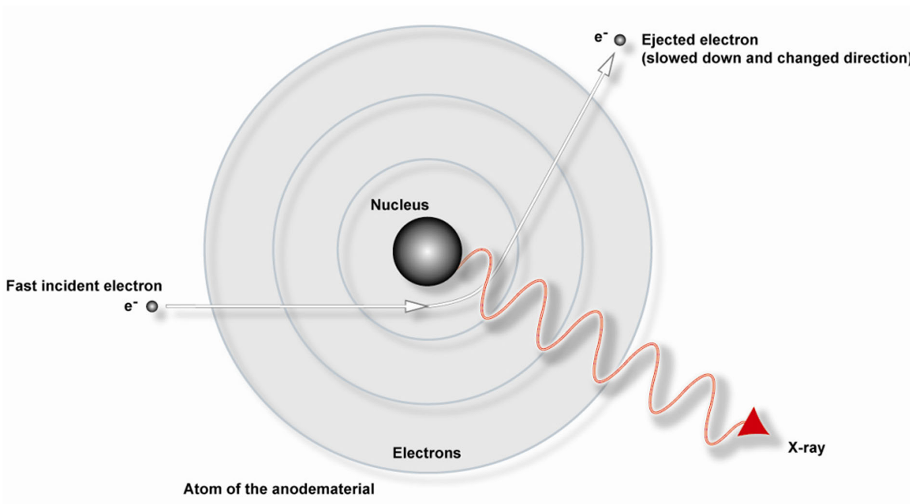


Figure 3.2: Generation of continuous (white) radiation

X-rays generated as described above are called “continuous” radiation because the radiation covers a wide energy range in the spectrum, contrary to characteristic emission lines.

3.1.2 Characteristic Spectrum

High-energy electrons can also interact with the atoms of the anode, producing “characteristic radiation” closely dependent of the anode material.

In this case, the incident electron collides with an electron of some atomic shell and knocks it out. The gap produced is filled by an electron from a higher shell. When transferring to a lower level, the electron releases “surplus” energy as X-ray fluorescence. This radiation is characteristic of the electronic structure of each element. By selecting an appropriate anode material, the excitation of the elements to be analyzed can be improved either by increasing the count rate or avoiding spectral line overlaps.

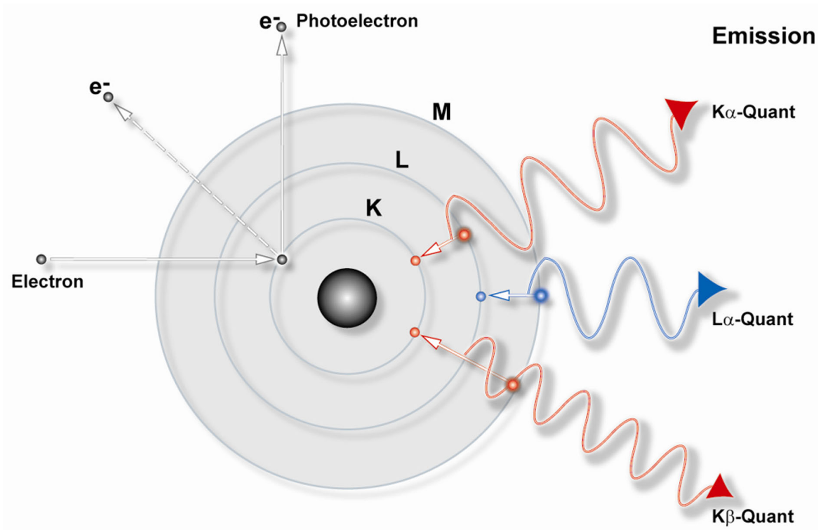


Figure 3.3: Generation of characteristic radiation, simplified representation

The figure above shows this process. In this diagram, the conventional notation for the electron shells and the photons produced by the transition of their respective electrons has been adopted:

- Kα radiation: from the L shell to the K shell
- Kβ radiation: from the M shell to the K shell
- Lα radiation by transition from the M shell to the L shell

Note that the photons have different characteristic wavelengths (energies).

On closer examination of an atom and its electron shells, it can be seen that the shells are divided into sub-shells as shown in the next figure. For this reason, Kα radiation can be divided into $K\alpha_1$ and $K\alpha_2$ radiation, Kβ radiation into $K\beta_1$ and $K\beta_2$ radiation etc. The difference between the wavelengths of these transfers is very small.

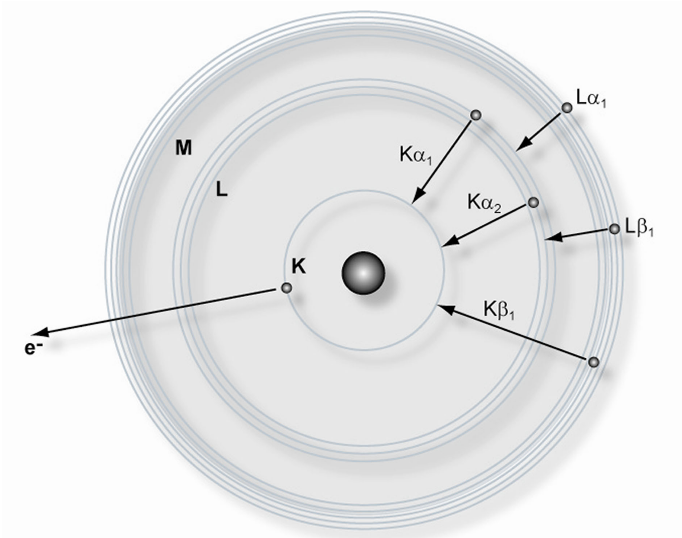


Figure 3.4: Sub-shells and transfered electron

The following figure shows the wavelength distribution of both continuous and characteristic radiation in a spectrum. Intensity (number of counts per second) is represented against wavelength; the latter converted to energy is the reciprocal of the wavelength multiplied by Plank's constant and the speed of light. The characteristic lines of the anode material $K\alpha_1$, $K\alpha_2$ and $K\beta_1$ are clearly visible above the continuous spectrum.

The point on the wavelength scale at which the continuous spectrum begins is known as the short wavelength limit. This radiation is produced by the electrons that give up all their energy on the initial impact, corresponding to the maximally reachable energy in the spectrum.

With an increase in supply voltage, this point moves down the wavelength scale. In other words, an increase in voltage produces a shift of the continuous spectrum in the direction of lower wavelengths and higher energy. The characteristic lines of the anode material merely undergo an increase in intensity (count rate); their position in the spectrum remains unchanged.

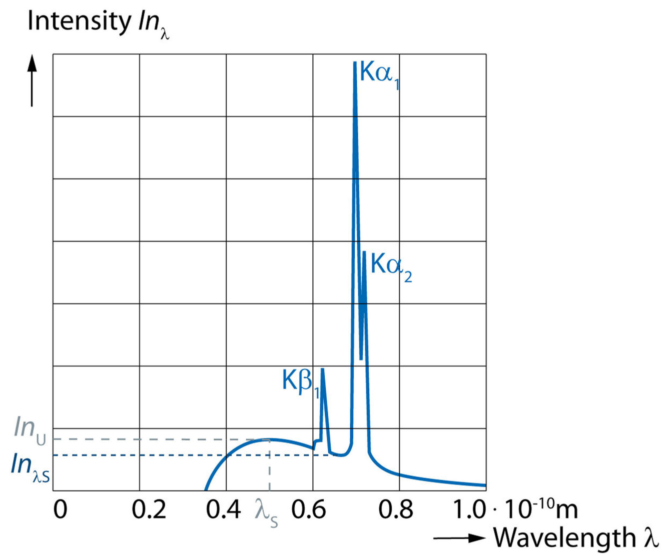


Figure 3.5: Spectrum of an X-ray tube

3.2 X-ray Diffraction

X-ray diffraction is a process in which X-rays inciding on a crystalline material are reflected by the atoms, which are arranged in ordered arrays, if certain geometrical conditions are met. The resulting X-ray diffraction pattern is characteristic of the material and can be used to identify it and elucidate the geometry of the unit cells in its crystal structure.

Diffraction results from the phenomenon known as “scattering”. Periodically changing electric fields of the incoming waves of X-rays excite the electrons of the atoms to periodic vibrations; these electrons then become the source of secondary X-rays with the same wavelength as the incoming X-rays (elastic scattering).

In a crystalline material, secondary radiation emanates from families of atom-containing planes in the three-dimensional crystal lattice structure. This radiation is usually weak and absorbed by the material. However, under certain conditions, the waves of secondary X-rays have sufficient intensity to escape absorption and be measured by a detector. This occurs when the waves are in phase and thus rein-force one another (constructive interference). Waves constructively interfere when the incident X-ray beam strikes the surface of the crystal at a definite angle, called the Bragg angle, which is related to the distance between the planes and the wavelength of the X-rays. When the Bragg equation (see below) is fulfilled, diffraction occurs.

In powder diffractometry, a powdered sample of a crystalline material is used rather than a single crystal. If a single crystal were used, the chances of a particular family of planes being in the correct position to satisfy the Bragg equation would be very small. Powdering a crystalline material does not destroy the crystal structure but simply produces millions of very small crystals pointing in all possible directions. For a particular family of planes, this increases the chance of being in the correct position to satisfy the Bragg equation.

3.2.1 The Bragg Equation

The secondary X-ray waves emanating from a family of atom-containing planes in the crystal lattice structure are in phase and constructively interfere with one another when the Bragg condition is met. This is expressed in the equation

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

where N is an integer multiple called the order of reflection, λ is the wavelength of the X-ray beam, d is the distance between the planes, and θ is the angle of deflection.

The Bragg equation is derived in the following way.

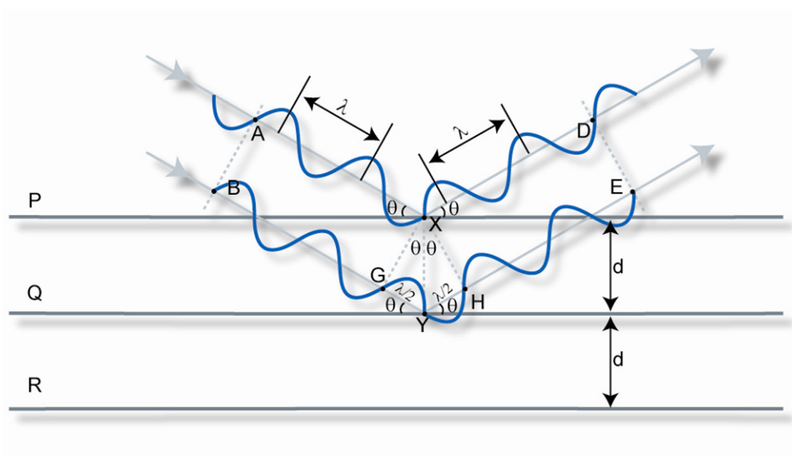


Figure 3.6: Fulfillment of the Bragg equation

The figure above shows a monochromatic beam of X-rays incident on the surface of a crystal at an angle θ. P, Q and R represent a family of planes separated by the distance d. Plane P reflects AX in XD. Similarly, plane Q reflects BY in YE at the same angle θ. Although the beam penetrates many more planes we need only consider the top two.

Since Q is lower than P, the beam path BYE is longer than AXD by the amount GY + YH. This is called the path difference.

Since angle AXG = θ + angle PXG = 90°

Then angle PXG = 90° - θ

Since angle PXY = PXG + GXY = 90°

Then angle GXY = θ

Similarly, you can show that angle YXH = θ

From the triangle GXY, $\sin\theta = \frac{GY}{d}$ Therefore GY = d sin θ

From the triangle YXH, $\sin\theta = \frac{YH}{d}$ Therefore YH = d sin θ

Therefore the path difference (GY + YH) = 2d sin θ

Now the two reflected rays will constructively interfere with one another, that is, they will be in synch, when the path difference is equal to the wavelength (λ) or a multiple of it.

Thus Nλ = 2d sin θ.

The Bragg equation is used in diffractometry to determine the nature of the irradiated specimen. Once the Bragg angle θ is determined, this can be used, together with the known wavelength, to calculate the distance d and thus the geometry of the unit cells of the lattice.

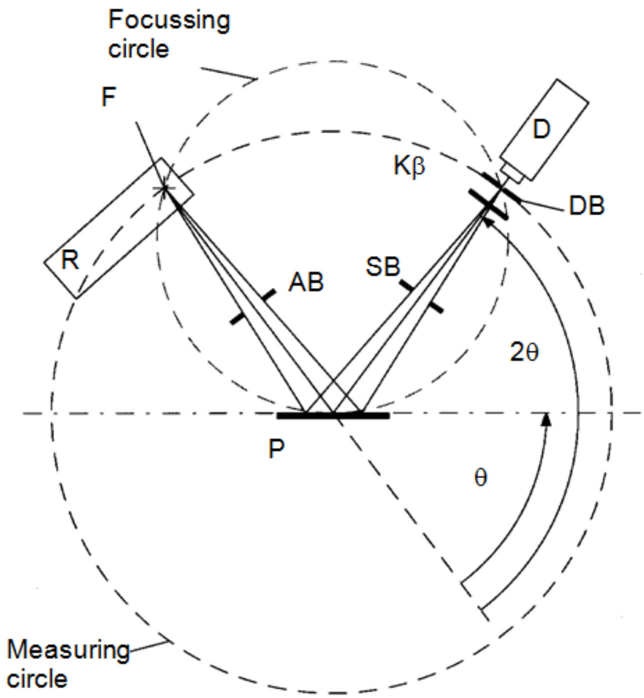
3.3 Measurement Geometries of X-ray Diffraction Systems

3.3.1 Bragg-Brentano Geometry

Most commercial diffraction systems employ the Bragg-Brentano parafocusing geometry. This results in both high resolution and high intensity of the diffracted beam.

The Bragg-Brentano geometry consists of two circles, the measuring circle and the focusing circle, as shown below. The X-ray source F and the detector slit DB lie on the circumference of the measuring circle, whose center is formed by the sample. When the Bragg equation is fulfilled, a divergent X-ray beam emitted by the X-ray source F is diffracted by the sample P and converges to a point at the detector slit DB. The average angle of incidence is θ and diffraction occurs at an angle of 2θ to the incident beam. The self-focusing of the reflected X-ray beam is most ideal if the sample curves around the circumference of the focusing circle, which is formed by drawing a circle through the three points: centre of sample, X-ray source and detector slit. However, because the use of curved samples is not practical, a close approximation to these ideal conditions is realized: the sample lies at a tangent to the focusing circle. This is sufficient to produce accurate results.

The fact that the self-focusing produced by the Bragg-Brentano geometry is not ideal is the reason why the term “parafocusing” is used to describe this method.



θ	*Incident angle*
2θ	*Diffraction angle*
AB	*Aperture slit*
D	*Detector*
DB	*Detector slit*
F	*Focus*
$K\beta$	*Kb filter*
P	*Sample*
R	*X-ray tube*
SB	*Scattered-radiation slit*

Figure 3.7: Bragg-Bretano diffraction geometry

3.3.2 θ-θ and θ-2θ Systems

X-ray diffraction systems realizing the Bragg-Brentano parafocusing geometry employ two different configurations.

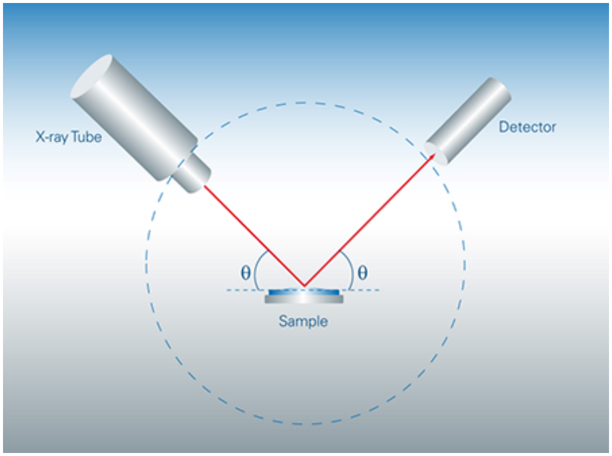


Figure 3.8: θ:θ configuration

In θ:θ goniometers (see figure above), the X-ray source and the detector are mounted on goniometer arms that rotate around a common axis located at the center of the goniometer. At the center of the goniometer is also located the sample, which is stationary. The center of the goniometer coincides with the center of the measuring circle of the Bragg-Brentano geometry

In the θ:θ arrangement the X-ray source, the detector and the sample, and thus the primary and secondary beams, form a vertical plane (the scattering plane) while the surface of the sample lies on a horizontal plane.

The movements of the X-ray source and detector arms are synchronized so that the arms form equal angles with the surface of the sample. Thus, the conditions are created to capture the reflected beam when diffraction occurs at the Bragg angle. In practice, the arms move with the same rotational speed around the goniometer axis, thus ensuring that the angles they form with the surface of the sample are increased and decreased equally. For the measurement of these movements, the horizontal surface of the sample has traditionally been used as the reference point. For this reason, the X-ray source and the detector are said to vary in the ratio of θ:θ.

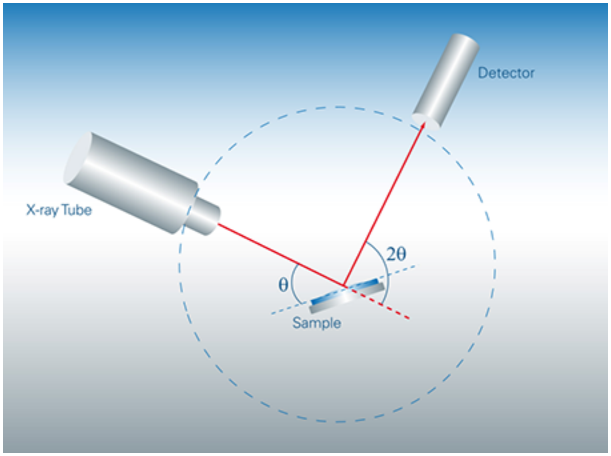


Figure 3.9: θ-2θ configuration

In θ-2θ diffactometers (see figure above), the X-ray source is stationary. Variation in the angle of incidence of the X-ray beam is ensured by movement of the sample, which turns around the same axis as the detector. The surface of the sample lies on a plane that is perpendicular to that formed by the primary and secondary X-ray beams. To ensure that the detector is correctly positioned when diffraction occurs, its movement is geared to that of the sample. In practice, it moves at twice the rotational speed of the sample. Traditionally the incident beam and its path have been used as reference points for measurement of the movement of the sample and the detector arm respectively. For this reason, the sample and the detector are said to vary in the ratio of θ-2θ.

3.3.3 Parallel Beam Geometry

Parallel beam geometry is used as an alternative to Bragg-Brentano geometry. The divergent incident X-ray beam from a line focus of the X-ray tube is transformed into a parallel beam with the help of a Göbel mirror. Göbel mirrors produce an intense and parallel beam that is free of Kβ radiation. The secondary beam path also requires a parallel beam optic. Very commonly Soller slits are used.

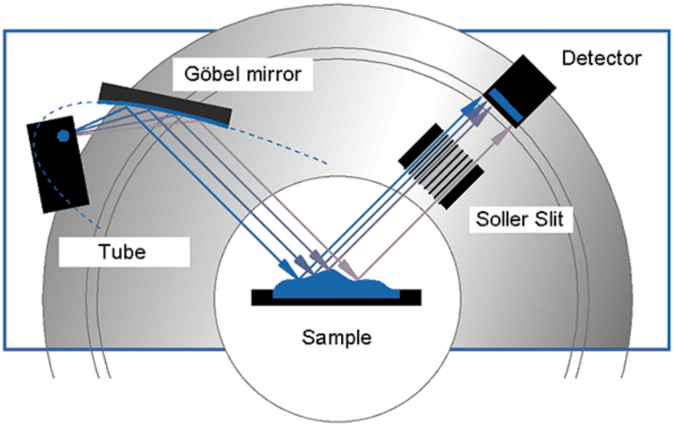


Figure 3.10: Parallel beam path of diffractometer

3.3.3.1 Applications

The D8 ADVANCE / D8 DISCOVER X-ray diffractometer can be used for nearly all X-ray diffraction applications, such as structure determination, phase analysis, stress and texture measurements.

Various accessories can be used together with the diffractometer. These include:

- Extended mask systems
- Various primary and diffracted beam optics
- Sample stages
- Eulerian Cradles: for texture and residual stress determination
- Accessories for grazing incidence
- High and low temperature chambers.
- Detectors (scintillation counter, position sensitive detector, Si(Li) semiconductor detector)
- Sample rings

4 Locations and Functions of Basic Components

4.1 Common components of the Instruments

4.1.1 Power Switch

The instrument power-up and power-down buttons and the line disconnector are located in a niche on the lower left side of the instrument. In addition, two emergency switch-off buttons are located in the lower part of the instrument, one on each side of the enclosure.

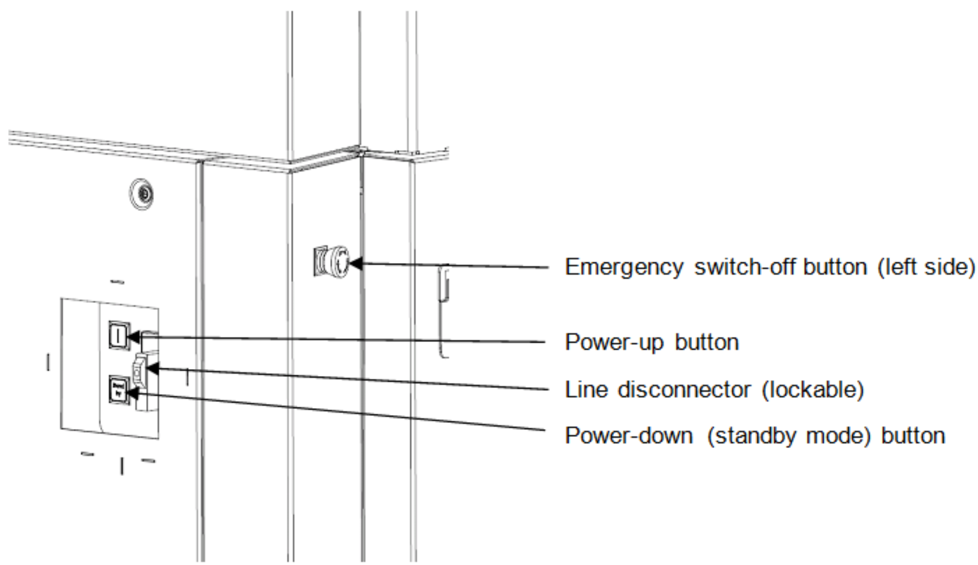
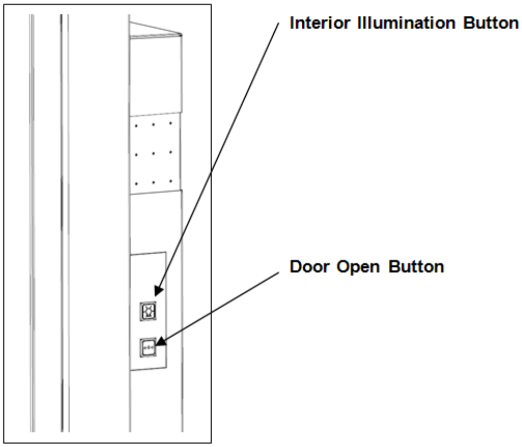


Figure 4.1: Power switch

4.1.2 Enclosure Control Buttons

Two buttons located on the right side of the instrument control the interior illumination and door lock mechanism of the instrument enclosure.



Interior Illumination Button

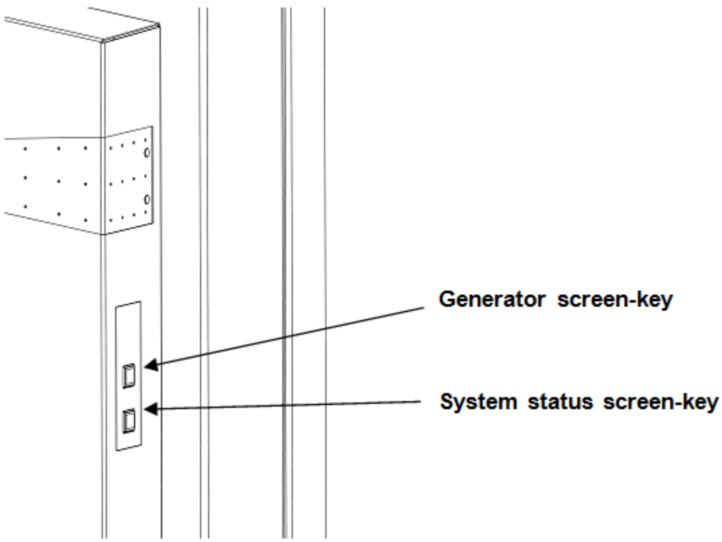
This button switches the enclosure illumination on and off. During boot-up of the instrument, the enclosure is not illuminated and this button is deactivated. After booting, the intensity of the illumination is controlled by the door opening/closing mechanism. With the default setting, opening the door increases the brightness and closing the door reduces it. Intensity levels can be set using the CONFIG software module. If the instrument has been powered down, you can continue to switch the illumination on and off. However, in this case the intensity is not controlled by the door opening/closing mechanism

Door Open Button

The front door is locked by means of a locking bolt. To unlock the door, you must press this button. Then you can open the door outwards. If you press the door open button when the X-ray tube window is open, the X-ray shutter will close automatically. The X-ray shutter can be re-opened after the front door has been closed. The door can also be unlocked using the TOOLS software module.

4.1.3 Generator Screen-key and System Status Screen-key

On the left side of the instrument are located two screen-keys.



Generator screen-key
This screen-key is used to control the status and operation of the generator.

System status screen-key
This screen-key displays system status information. It does not have any other function. Pressing it does not have any effect on the instrument.

For more details on the generator screen-key states please refer to table 1.1 in chapter *Starting the Instrument [▸ 13]* and for the system status screen-key states to table 1.2.

The errors and warnings indicated by the symbols (which appear against a red background) of the system status screen key are usually indicated by icons in the TOOLS software module. In addition, information on the errors and warnings is available in TOOLS. The programme can also be used to diagnose all existing errors and warnings and carry out repairs.

4.2 The Experimental Area

4.2.1 Goniometer

The goniometer of the D8 ADVANCE / D8 DISCOVER diffractometer consists of two concentric rings that rotate around the same axis independently of each other.

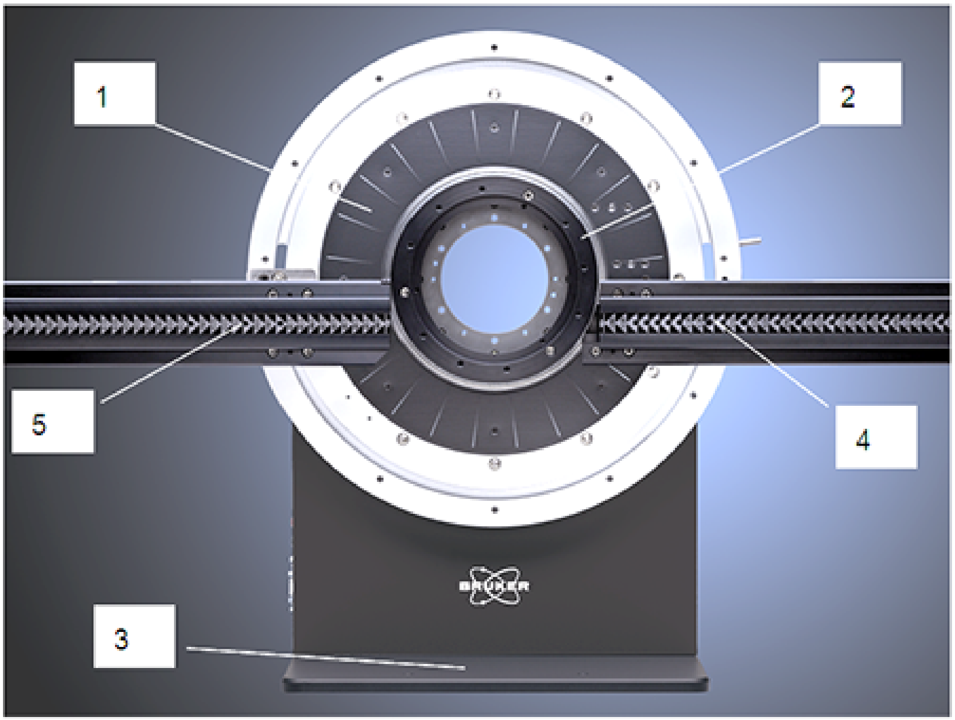


Figure 4.2: θ-2θ goniometer of the D8 ADVANCE / D8 DISCOVER.

1	Outer ring	2	Inner ring
3	Stand	4	Track for detector
5	Track for tube housing		

- The inner ring (2) and outer ring (1) are driven by a stepper motor in each case.
- A stand (3) is provided for vertical installation.
- Opto-electronic switches that serve as reference positions for the θ and 2θ angular scales are fitted inside the inner and outer rings.

θ/θ goniometer

The track (5) for the tube housing and the divergence slit system is mounted on the outer ring (1). The track (4) for the detector and the detector slit system is mounted on the inner ring (2). The sample stage is fixed to the center of the goniometer.

θ/2θ goniometer

The track (5) for the tube housing and the divergence slit system is fixed to the goniometer housing. The track (4) for the detector and the detector slit system is mounted on the outer ring (1). The sample stage is mounted on the inner ring (2).

Measuring circle diameters

The D8 ADVANCE diffractometers have 3 convenient predefined measuring circle diameters:

- 500 mm
- 560 mm
- 600 mm

Additionally D8 DISCOVER diffractometers have predefined measuring circle diameters to be used with e.g. channel cut setups. Different optics can now be used in an easy and reproducible way, without having to measure the distances. This is achieved by using hard pin stops on the tracks. There are holes on the incident beam track as well as on the diffracted beam side. The remaining holes on the secondary side are used for different special applications.

4.2.1.1 Counterweights

Behind the goniometer are located two counterweights whose purpose is to balance the weight of the goniometer arms with their optical systems and thus the load on the motors that drive the θ and 2θ axes in the case of the θ/θ system, and the 2θ axis in the case of the θ/2θ system. In case of horizontal goniometer setups, no counterweights are needed.

The positioning of the counterweights is done before shipment. Once adjusted, these counterweights should not be touched.

4.2.2 X-ray Tube

The basic principles of X-ray generation have already been described in chapter *General Physical Principles [▸ 29]*. Used as standard for X-ray generation in D8 ADVANCE / D8 DISCOVER diffractometers are Siemens X-ray tubes. One of these tubes is shown below.

1	*Radiation window*
2	*Bore hole for positioning the tube in the tube stand*

Figure 4.3: Example of a Siemens X-ray tube

Within the framework of the type approval for the D8 ADVANCE / D8 DISCOVER diffractometer, all anode materials with an atomic number Z ≤ 42 can be used. This atomic number corresponds to that of molybdenum. Anode materials with a higher atomic number can also be used in the D8 ADVANCE / D8 DISCOVER diffractometer. However, these require approval over and above that accorded by the type approval.

The X-ray tubes have two beryllium windows from which the X-rays can escape in a single direction. One of the beryllium windows lies parallel to the long side of the luminous spot on the anode, which can be regarded in a good approximation as rectangular. This window is used for line focus operation. The other beryllium window lies parallel to the short side of the luminous spot and is used for point focus operation. The exact size of the focus in the particular operating mode is dependent on the type of tube used and is specified in the technical documents of the X-ray tube.

For line focus and point focus operation there are different cooling plates. These differ in that the cooling circuits are set at an angle of 90° to each other.

For users who want to switch between point focus and line focus without changing the tube, there is a third type of tube cooling plate. Such a rotating cooling plate (TWIST-TUBE) is shown in figure 6.2 in section *Switching Between Point and Line Focus [▸ 147]*. Refer to this chapter to learn how to switch between point focus and line focus operation.

4.2.3 Labyrinths

On the floor of the enclosure, to the left and right of the goniometer, are located two labyrinths whose purpose is to provide a safe and convenient passage for the various lines entering and leaving the enclosure. These include water cooler lines and electrical lines supplying current to the motors and the X-ray tube. The labyrinths are designed to prevent radiation from escaping to the outside of the instrument during diffractometer operation. If you use any additional equipment in the enclosure that is not included in the delivery scope, use the labyrinth on the right side of the instrument for the passage of the lines to the equipment.

- D8 ADVANCE: The labyrinth on the left side is fixed to the instrument while that on the right side can be slid in and out of position.
- D8 DISCOVER: Both labyrinths (left and right) can be slid in and out of position.

4.2.4 Accessories Shelf

Inside the enclosure, on the left and right walls of the diffractometer, and near the enclosure doors, are mounted two shelves for the storage of accessories. One of these shelves is shown below.

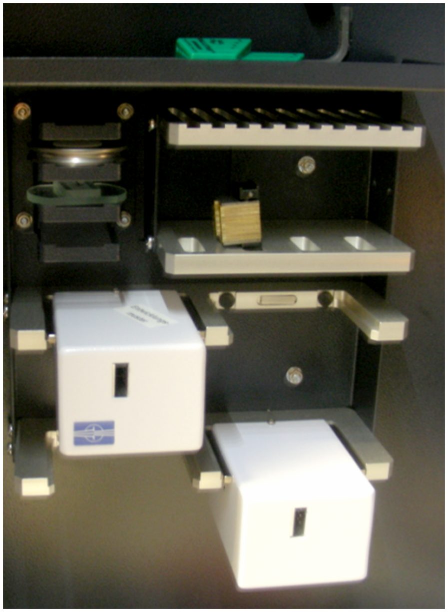


Figure 4.4: One of the two accessories shelves inside the radiation enclosure

4.3 Overview of the locations of components in the D8 ADVANCE diffractometer

Table 4.1: Location of components in diffractomer

Location	Components
Front	Distribution board, generator, safety board and water cooler
Back	F1 generator switch, water inlet and outlet lines, ventilators

Location	Components
Left	Mains distribution board with mains filter, terminals, circuit breakers and fuses
Right	Controller, universal I/O board, indexer boards and detector board
Enclosure	Goniometer, accessories shelf, labyrinths

4.3.1 The External Housing of the D8 ADVANCE

The control elements of the D8 diffractometer are located at different points on the front and left sides of the instrument.

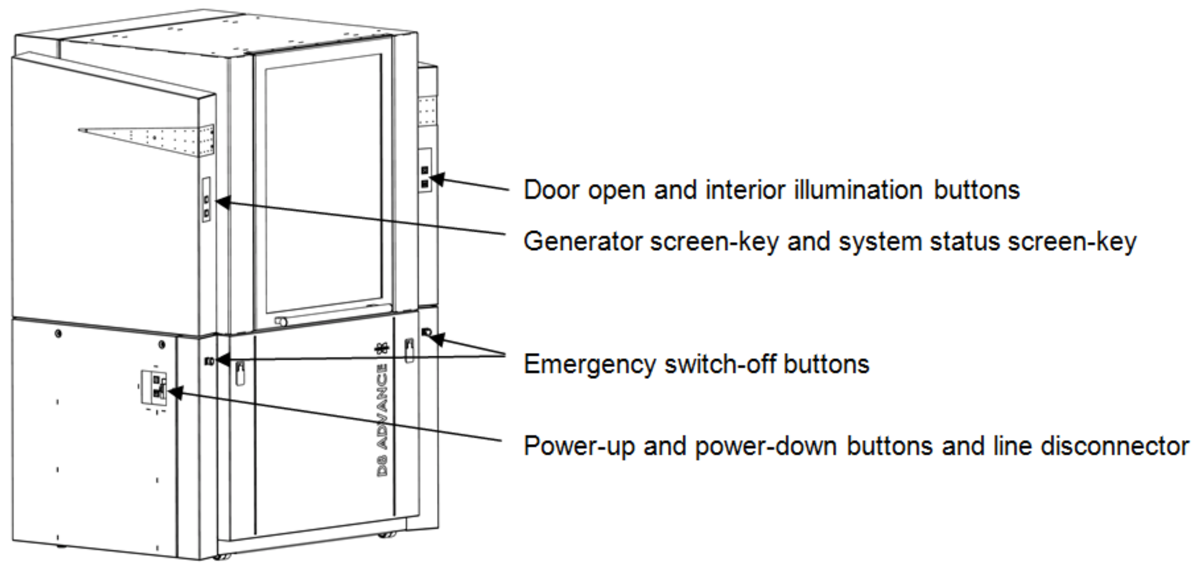


Figure 4.5: The external housing of the D8 ADVANCE

4.3.1.1 Door Mechanism D8 ADVANCE

The door catch is released by pressing the door open button.

Door Open Button

The front door is locked by means of a locking bolt. To unlock the door, you must press this button. Then you can open the door outwards. If you press the door open button when the X-ray tube window is open, the X-ray shutter will close automatically. The X-ray shutter can be re-opened after the front door has been closed. The door can also be unlocked using the TOOLS software module.

4.3.2 Underneath the Experimental Area D8 ADVANCE

4.3.2.1 Removing the Panels

1. To remove the front panel, pull up the two black latches at the top of the panel and turn them to the right or left.
   ➨ The panel can then be swung open. You can detach the panel completely by lifting it out of the suspension holes at the foot of the housing.
2. To remove one of the side panels, unscrew the cam lock screws, which are fitted with a spring, at the top of the panel.
   ➨ The panel can then be swung open. You can detach the panel completely by lifting it out of the suspension holes at the foot of the housing.
3. The rear panel can be removed only after the left side panel has been removed.
4. To remove the rear panel, unscrew the screws.
   ➨ It can then be swung open. You can detach the panel completely by lifting it out of the suspension holes at the foot of the housing.

4.3.2.2 Lower Front D8 ADVANCE

The following components are located at the lower front side of the diffractometer:

- Distribution board, generator, safety board and water cooler.

- A metal rack to the left accommodates the generator and the water cooler as slide-in units. On either side of this rack are three extra pairs of holes that can be used to accommodate additional slide-in units.
- To the right of the rack are located the distribution and safety boards.

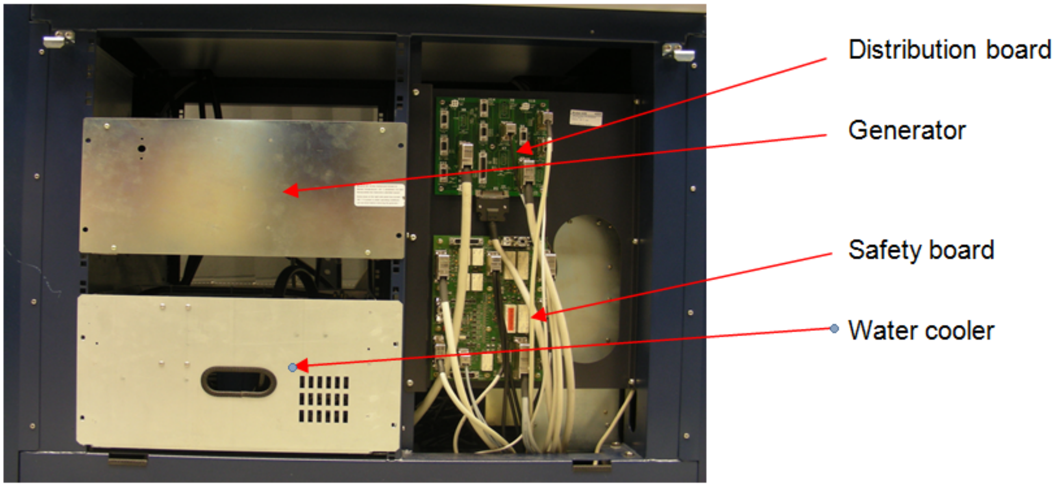


Figure 4.6: Lower front region, underneath the experimental area

4.3.2.3 Lower Back D8 ADVANCE

The following components are located at the lower rear side of the diffractometer:

- F1 generator switch, water inlet and outlet lines, and ventilators.
- The F1 generator switch is located at the rear of the generator. This switch is actuated in the case of a malfunction and interrupts the flow of high-voltage current to the generator. It can be reset manually.
- Openings are provided in the rear panel for two water lines (inlet and outlet) and a power supply line for the generator.
- Two ventilators are housed in the rear panel and are connected to the instrument by a power line.

4.3.2.4 Lower Left Side

The following components are located at the lower left side of the instrument:

- Mains distribution board with the mains filter, terminals, circuit breakers and fuses.

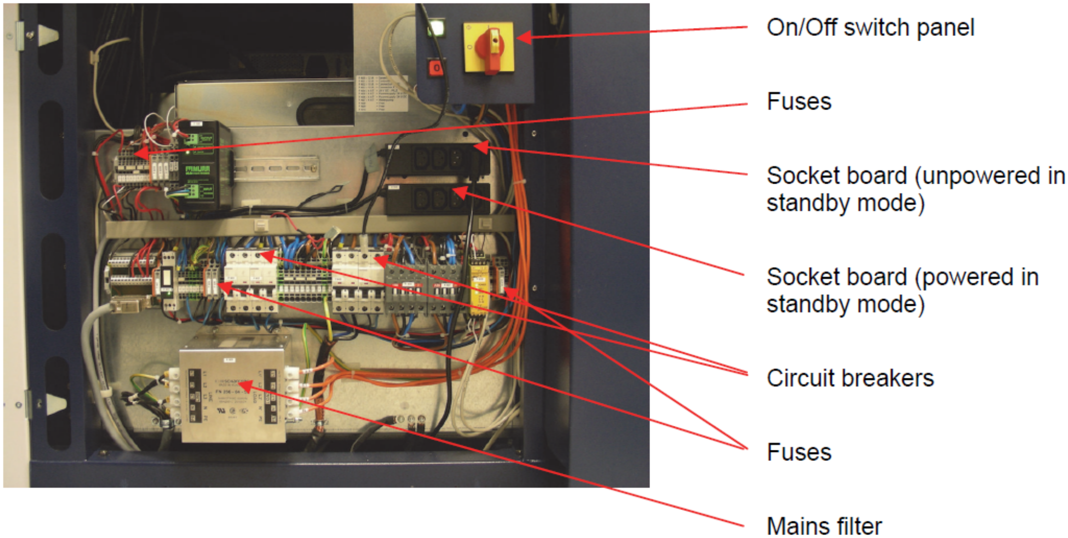


Figure 4.7: Lower left side of the instrument

- The mains filter is located at the bottom left. In the center portion of the mains distribution board is a row of terminals, circuit breakers and fuses. Some fuses are also located above this row at the top left. The circuit breakers are automatically triggered in the case of overload or a short circuit and can be manually reset to resume normal operation. The fuses are accommodated in fuse holders that are identified by a number prefixed by F, for example, F604, F611 and F606.
- Two black socket boards which regulate the flow of current to the various components are located on the right hand side above the row of terminals, circuit breakers and fuses. Depending on the electrical state of the instrument, these socket boards can be supplied or not supplied with power. The power supply is controlled by the **Power ON** and **Power OFF** buttons and the **Mains disconnector** switch, which are visible at the top right and can be accessed from the outside of the instrument.

To change a fuse:

1. Flip the fuse holder upwards,
2. open its cover, and
3. replace the fuse by the spare fuse stored to the left of it in the holder.
4. Then close the cover and flip the fuse holder back into place. The fuses can be located with the help of the diagram attached to the distribution board.

The instrument has three electrical states:

Table 4.2: Electrical states

State	Power ON Button	Power OFF Button	Mains Disconnect or switch	Upper Socket X101	Lower Socket X102
On	On	Off	On	Supplied with power	Supplied with power
Stand-by	Off	Off	On	No power	Supplied with power
Off	Off	Off	Off	No power	No power

4.3.2.5 Lower Right Side D8 ADVANCE

The following components are located at the lower right side of the instrument:

- Control rack, universal I/O board, indexer boards, detector board.
- The control rack accommodates the detector board, the universal I/O board and indexer boards. Each indexer board can be either two-axis or four-axis, meaning control of either two or four motors, respectively. Attached to the control rack at the top right is the Ethernet switch, which is connected to the control PC, the detector controller, the control rack, and the network (LAN).
- In the frame accommodating the control rack are four extra sets of slots which can be used to accommodate additional components. In the instrument shown in below, two of these sets are occupied by a controller.

1. To remove this panel, raise the two black latches at the top of the panel and turn them.

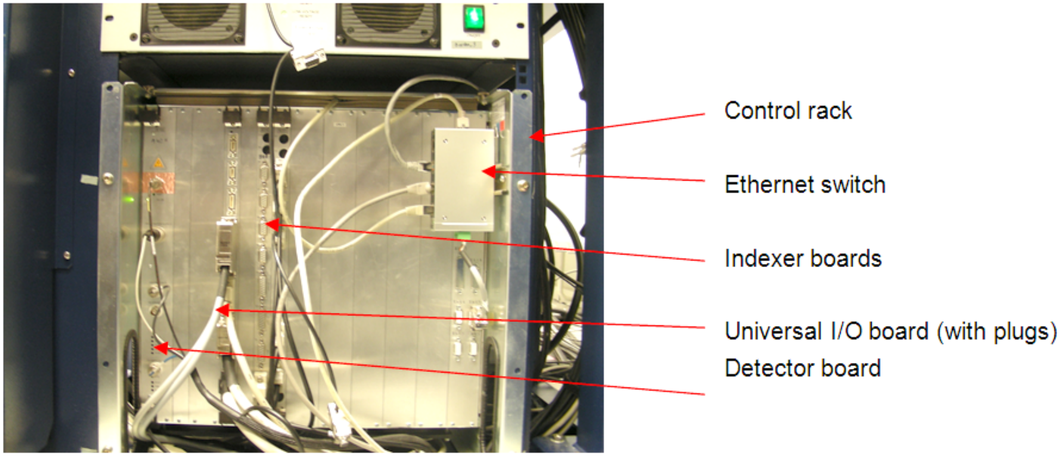


Figure 4.8: Lower right side of the instrument

4.4 Overview of the locations of components in the D8 DISCOVER diffractometer

Table 4.3: Location of components in diffractometer

Location	Components
Front	Generator, Distribution Board, Control Rack, additional Controllers
Back	F1 generator switch, Safety Board, water chiller, water inlet and outlet lines, ventilators
Left	Mains distribution board with mains filter, terminals, circuit breakers and fuses
Right	--
Enclosure	Goniometer, accessories shelf, labyrinths

4.4.1 The External Housing of the D8 DISCOVER

The control elements of the D8 diffractometer are located at different points on the front and left sides of the instrument.

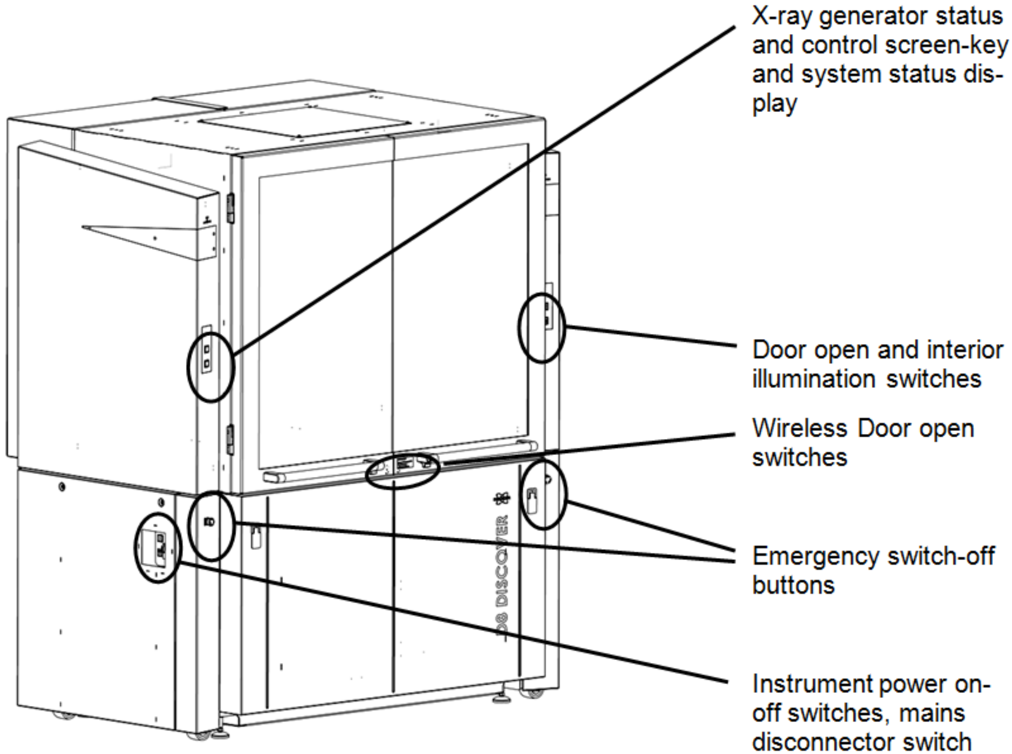


Figure 4.9: The external housing of the D8 DISCOVER

4.4.1.1 Door Mechanism D8 DISCOVER

The door catch is released by pressing the door open button. One **Door open** button is located in the left side panel. Two more wireless **Door open** buttons are mounted in the end of each door handle.

4.4.1.1.1 Door Open Button

The front door is locked by means of a locking bolt. To unlock the door, you must press this button. If you press the **Door open** button when the X-ray tube window is open, the X-ray shutter will close automatically. The X-ray shutter can be re-opened after the front door has been closed. The door can also be unlocked using the TOOLS software plug-in.

4.4.1.1.2 Wireless Door Open button

Inside the door handles, towards the center of the instrument, two wireless door open button are located. Pushing these buttons will unlock the doors.

4.4.1.1.3 Slide- / Swing Mechanism

- In standard operation mode, both doors can be slid open. If necessary, e.g. to bring large equipment into the box, operation of the doors can be switched to swing mode.
- In order to switch between both modes use the lever underneath the door handle (see figure below). With locked doors, push the lever to enable swing mode and pull the lever to enable slide mode.
- When in swing mode, unlock the door by using one of the **Door open** buttons, slide the doors open until mechanical stop is reached and then swing it open.
- When in slide mode, unlock the door by using one of the door open buttons, slide the doors open until mechanical stop is reached.

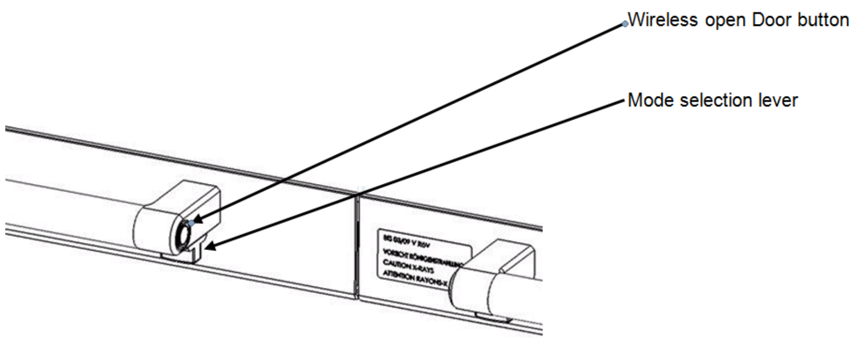


Figure 4.10: The mode selection lever is used to switch between swing and slide mode of the doors

4.4.2 Underneath the Experimental Area D8 DISCOVER

4.4.2.1 Lower Front D8 DISCOVER

The following components are located at the lower front side of the diffractometer:

- Distribution board, generator and the control rack. Optionally additional controllers are mounted here.
- A metal rack to the left accommodates the generator. On either side of this rack are nine extra pairs of holes that can be used to accommodate additional slide-in units.
- In the center of the lower box the distribution board is located.
- The right side of the box contains the control rack and optionally additional slide-in units.

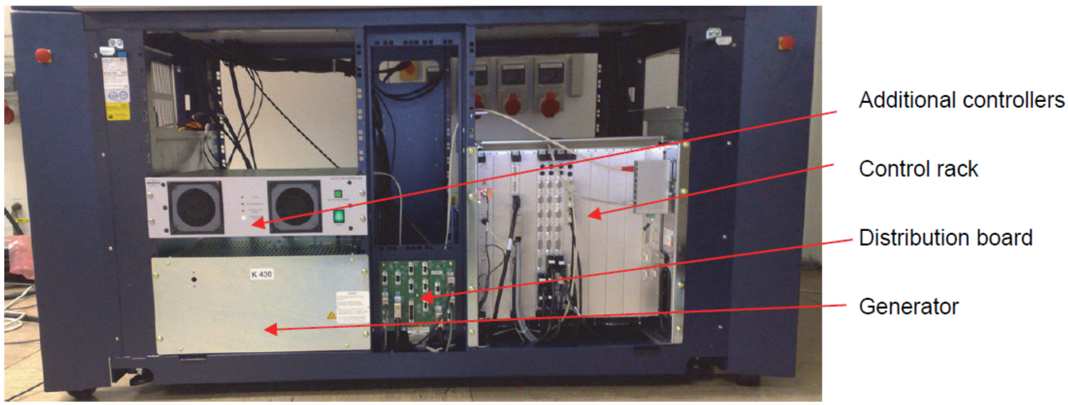


Figure 4.11: lower front region of the D8 DISCOVER, underneath the experimental area

4.4.2.1.1 Control rack, universal I/O board, indexer boards, detector board.

The control rack accommodates the detector board, the universal I/O board and indexer boards. Each indexer board can be either two-axis or four-axis, meaning control of either two or four motors, respectively. Attached to the control rack at the top right is the Ethernet switch, which is connected by four lines to the control PC, the detector controller, the control rack, and the network (LAN).

In the frame accommodating the control rack are four extra sets of slots which can be used to accommodate additional components.

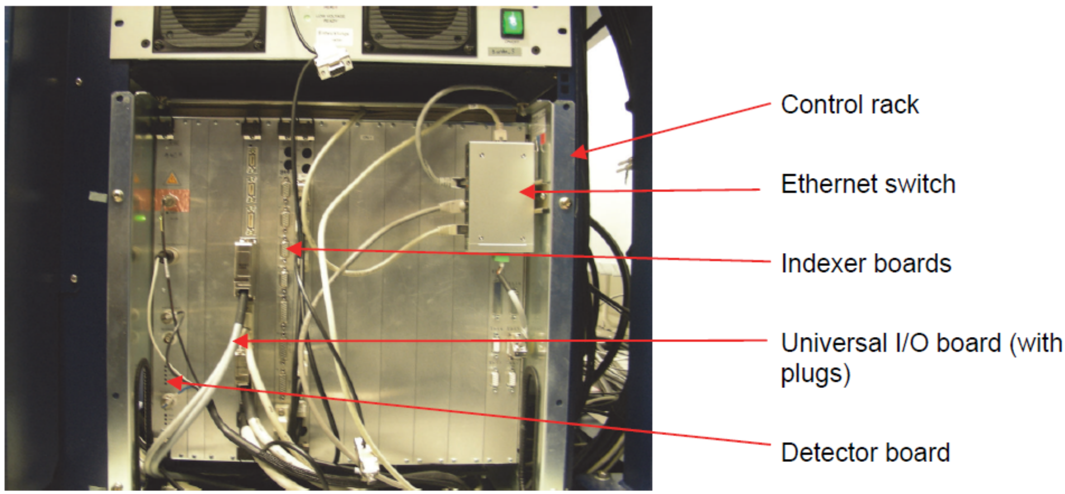


Figure 4.12: Control Rack

4.4.2.2 Lower Back D8 DISCOVER

The following components are located at the lower rear side of the instrument:

- Water chiller (optional but recommended), Safety Board, water inlet and outlet lines, and ventilators.
- The F1 generator switch is located at the rear of the generator. This switch is actuated in the case of a malfunction and interrupts the flow of high-voltage current to the generator. It can be reset manually.

- Openings are provided in the rear panel for two water lines (inlet and outlet) and a power supply line for the generator. Two additional holes on the upper left and upper right side of the rear panel are provided for e.g. vacuum hoses.
- Two ventilators are housed in the rear panel and are connected to the instrument by a power line.

4.4.2.3 Lower Left Side

The following components are located at the lower left side of the instrument:

- Mains distribution board with the mains filter, terminals, circuit breakers and fuses.

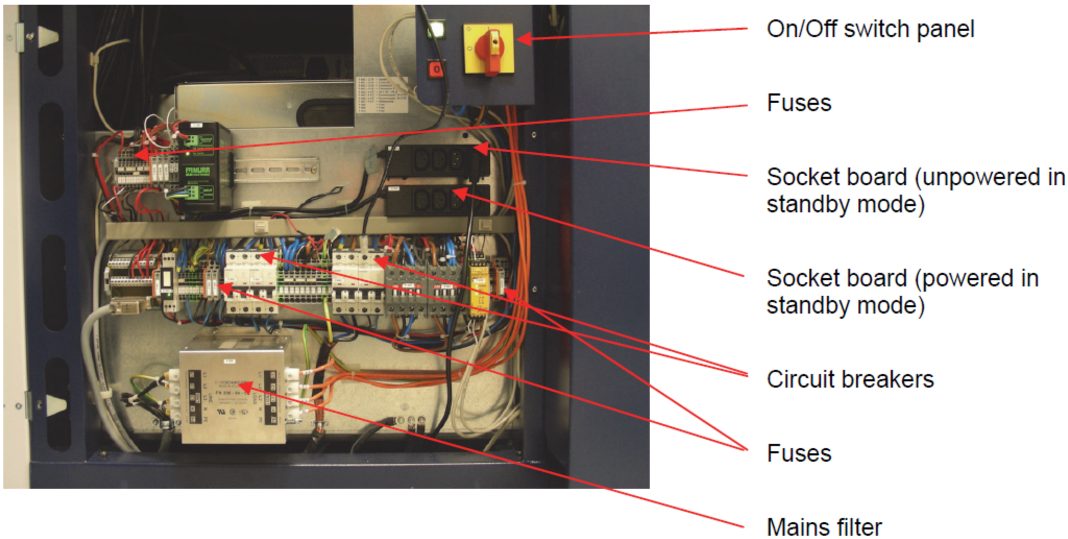


Figure 4.13: Lower left side of the instrument

- The mains filter is located at the bottom left. In the center portion of the mains distribution board is a row of terminals, circuit breakers and fuses. Some fuses are also located above this row at the top left. The circuit breakers are automatically triggered in the case of overload or a short circuit and can be manually reset to resume normal operation. The fuses are accommodated in fuse holders that are identified by a number prefixed by F, for example, F604, F611 and F606.

- Two black socket boards which regulate the flow of current to the various components are located on the right hand side above the row of terminals, circuit breakers and fuses. Depending on the electrical state of the instrument, these socket boards can be supplied or not supplied with power. The power supply is controlled by the **Power ON** and **Power OFF** buttons and the **Mains disconnector** switch, which are visible at the top right and can be accessed from the outside of the instrument.

To change a fuse:

1. Flip the fuse holder upwards,
2. open its cover, and
3. replace the fuse by the spare fuse stored to the left of it in the holder.
4. Then close the cover and flip the fuse holder back into place. The fuses can be located with the help of the diagram attached to the distribution board.

The instrument has three electrical states:

Table 4.4: Electrical states

State	Power ON Button	Power OFF Button	Mains Disconnector switch	Upper Socket X101	Lower Socket X102
On	On	Off	On	Supplied with power	Supplied with power
Stand-by	Off	Off	On	No power	Supplied with power
Off	Off	Off	Off	No power	No power

4.4.2.4 Lower Right Side D8 DISCOVER

The optional water chiller can be accessed from the right side of the instrument. This might be necessary to fill the water tank in the chiller.

5 Descriptive Listings of Common Components

5.1 Primary Side: Alignment Controls and Optics Mounts

In its most stripped-down form, the D8 ADVANCE / D8 DISCOVER experimental area consists of a goniometer and two tracks. Most measuring environments require the mounting of components to the tracks and the central region of the goniometer. The mounting of X-ray tubes, X-ray optics, sample stages and detectors is described in the following sections.

The basic stacking series for mounting components is shown below and the functions of these elements are in the following table. The component labelled with an asterisk * is the safety slit. The optics mount is also called “optical bench”.

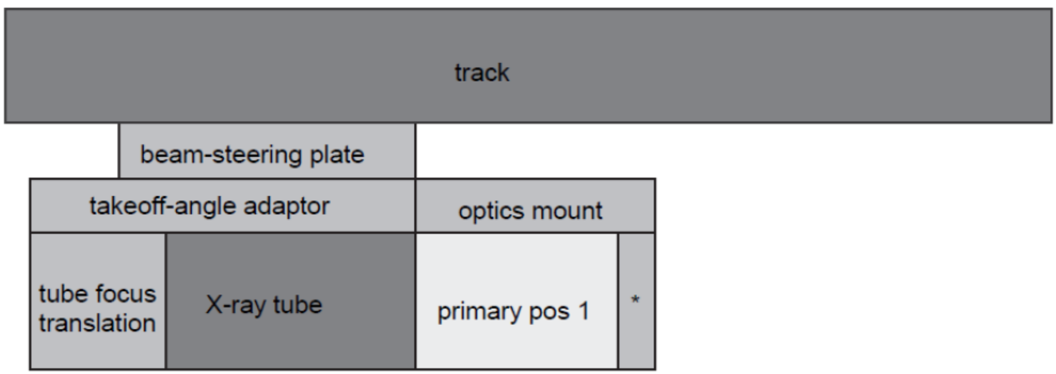


Figure 5.1: Schematic diagram for mounting components to the primary track

All optics of the D8 ADVANCE / D8 DISCOVER are mounted on the optics mount. Only exception is in case of an D8 DISCOVER the Rotary Absorber and the UBC-collimator mount which can in some configurations be mounted from the track.

Table 5.1: Primary side adjustments and their functions

Tube focus translation	Translates the tube relative to the optics mount (“bench”) and its optics. For line-focus tubes, one translation direction is needed. For point-focus tubes, two translations are needed.
Takeoff-angle adaptor	Changes the angle of inclination between the tube and optics mount. Typically, one adaptor is sufficient for most applications. Additional adaptors may be required if changing tubes over a large energy range or changing between Johansson optics and other optics.
Beam-steering plate	Rotates or rotates & translates the tube and the optics, to get the intensity-optimized beam through the goniometer center. The rotation and translation is in the scattering plane. In case a 2-bounce channel cut monochromator is used the translation might be motorized to automatically adapt for evolving beam displacement.

5.1.1 Alignment Concept

The X-ray optics have been factory-aligned for optimum performance and for a beam direction parallel to the optics mount (no tilt out of the plane of diffraction or scattering), at the correct height above the track and 150 mm (or 214 mm or 258 mm in case of D8 DISCOVER) above the goniometer plane. The positions of the optics on the optics mount are pre-defined and positioned such that the beam will go through the goniometer center, with maximum intensity, for the appropriate beam-steering plate setting(s). To account for changes in or replacement of the X-ray tube, the tube can be translated relative to the optics with the tube focus translation.

5.1.2 Beam-Steering Plates

The beam-steering plates provide either rotation or rotation plus translation.

- The **rotation** is used for centering the primary beam towards the goniometer centre.
- The **translation** is needed if optics changes arise that produce shifts in the beam position. Therefore, this is mostly not needed. Normally, all D8 ADVANCE optics are aligned on the optical bench in such a way, that the primary beam is pointing towards the goniometer centre. The software takes care of the angular offsets with help of the

recognition chips. Only, if some of the optics have beam offsets relative to each other, a translation is needed when switching an optics via SNAP-LOCK. In a D8 DISCOVER these beam displacements are ermerging when using 2-bounce channel cut monochromators.

- The **focus translation** (line and point) is needed for positioning the tube focus with respect to the optical bench. This must be done e.g. after changing to a different X-ray tube.
- The **measurement circle** can be changed by loosening the fixing screw (cf. following figure) and sliding the beam-steering plate along the primary track. The fixing screw must be fixed again afterwards for getting precise measurements. To allow for a convenient change of measurement circle, the most common positons have been predefined. The whole optics mount can be moved against a solid pin mounted on such a predefined position in the track

5.1.2.1 Rotation Beam-Steering Plate

The rotation beam-steering plate is shown in two figures below.

Table 5.2: Specifications Rotation beam-Steering Plate

Range for rotation:	± 4° (equivalent to ± 17 mm at the goniometer centre for 500 mm diameter)
Range for line focus translation:	± 600 µm
Range tube focus-goniometer centre:	
D8 DISCOVER	140 mm - 540 mm (diameter 280 mm - 1080 mm) depending on configuration
D8 ADVANCE	140 mm - 360 mm (diameter 280 mm - 720 mm) depending on configuration
Measurement height:	
D8 DISCOVER	150 mm, 214 mm, 258 mm depending on configuration
D8 ADVANCE	150 mm

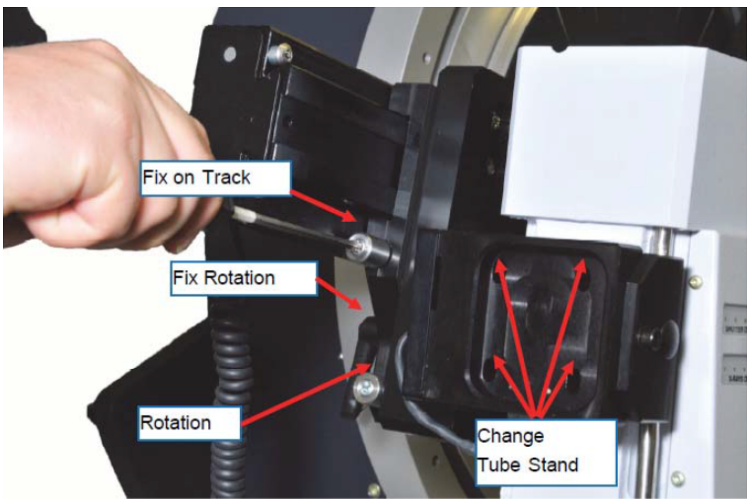


Figure 5.2: Rotation beam-steering plate: front side

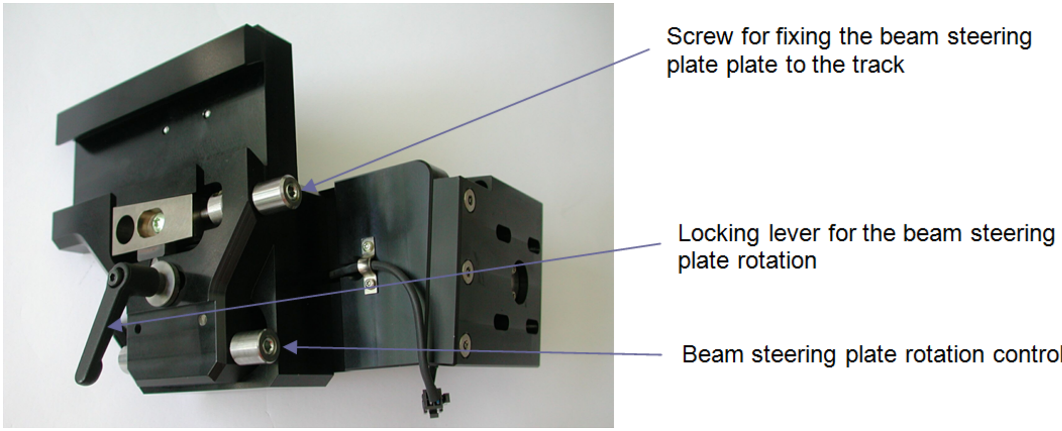


Figure 5.3: Rotation beam-steering plate: front side

5.1.2.2 Rotation and Translation Beam-Steering Plate

The manual version of the rotation and translation beam-steering plate is shown in the figure below. A motorized translation is available.

Table 5.3: Specifications

Range for rotation:	± 4° (equivalent to ± 17 mm at the goniometer centre for 500 mm diameter)
Range for translation:	from +20 mm to -5 mm
Range for line focus translation:	± 600 µm
Range for point focus translation out of plane (optional):	from -2.5 mm to +8.5 mm
Range tube focus-goniometer centre:	
D8 DISCOVER	140 mm - 540 mm (diameter 280 mm - 1080 mm) depending on configuration
D8 ADVANCE	140 mm - 360 mm (diameter 280 mm - 720 mm) depending on configuration

Measurement height:	
D8 DISCOVER	150 mm, 214 mm, 258 mm depending on configuration
D8 ADVANCE	150 mm

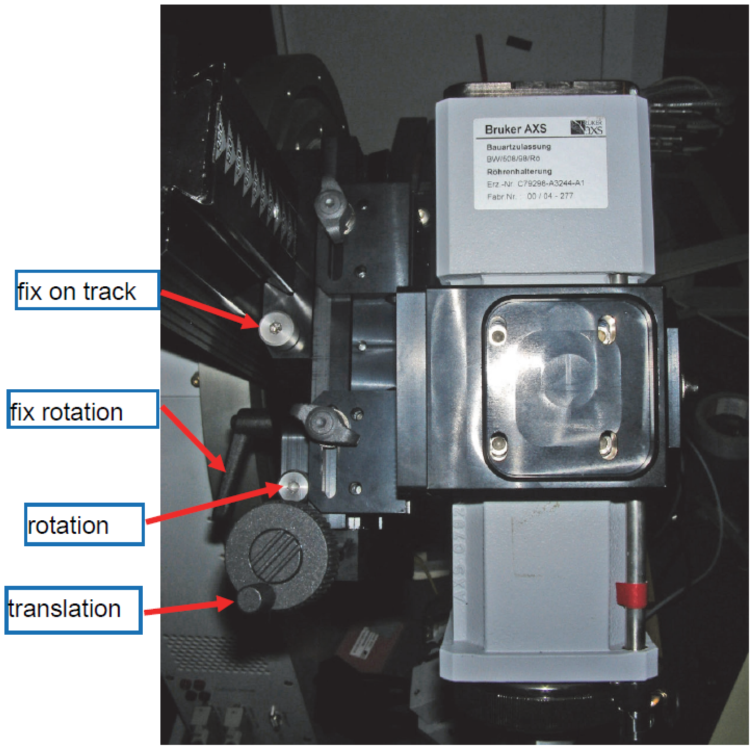


Figure 5.4: Rotation and translation beem-steering plate (non-motorized version)

5.1.3 Takeoff Angle Adaptor Plates

The tube stand is mounted on the beam-steering plate by a takeoff angle adaptor plate. The standard takeoff adaptor plate angle is -7.5° (see figure below). This provides optimum takeoff from the tube focus with an approx. factor 10 decrease of focal dimension. It results in good angular resolution in combination with long fine focus tubes in case of

Bragg-Brentano and beam steering for 500 mm diameter. It is an optimum setting for maximum flux from Göbel mirrors. This plate is used in all cases where the primary beam is close to 0° when taking into account takeoff and deflection by primary optic. This is the case for Göbel mirrors, Bragg-Brentano, TWIN optics, POLYCAP and Montel mirrors. Maximum range is given by the rotation range of the beam-steering plate, i.e. ±4°.The adaptor plate needs to be changed when going beyond the range of ±4°.

Procedure for exchanging the adaptor plate:

- The generator should be switched off when doing this. The safety circuit will take care, if switching off the generator should be forgotten.

1. Remove tube stand, figure 6.2. in section *Rotation Beam-Steering Plate [▸ 64]* shows the four screws which have to be loosened.
2. Remove adaptor plate, four screws in

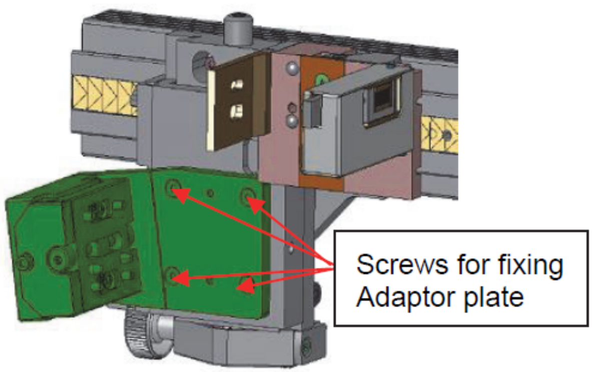


Figure 5.5: Standard takeoff angle adaptor plate (-7.5°). Used for Bragg-Brentano, Göbel mirror, TWIN optics, POLYCAP: all optics with primary beam deflection close to zero

3. Install new adaptor plate. Pins take care of good position. Interlocks check correct installation.
4. Install tube stand. Pins take care of good position. Interlocks check correct installation.
5. Install new optics via SNAP-LOCK. Check primary beam intensity by scanning primary beam. If intensity is too low, move detector in primary beam and optimize intensity with help of ratemeter and focus translation.

Sometimes it might make sense to change between adaptor plates of same type, even when deflection angles of the optics are within the range of ±4°. Most Göbel mirror types have identical deflection angles. These are factory aligned in a way, that they can be switched with SNAP-LOCK and not touching focus translation and beam-steering rotation. However, Mo Göbel mirrors and Phase ID mirrors, as well as various types of Montel mirrors have significant different deflection angles. After a change from e.g. Cu-mirror to Mo-mirror the focus translation needs to be changed by a huge amount for getting back primary intensity. This can be avoided by using dedicated adaptor plates for mirrors with different deflection angles.

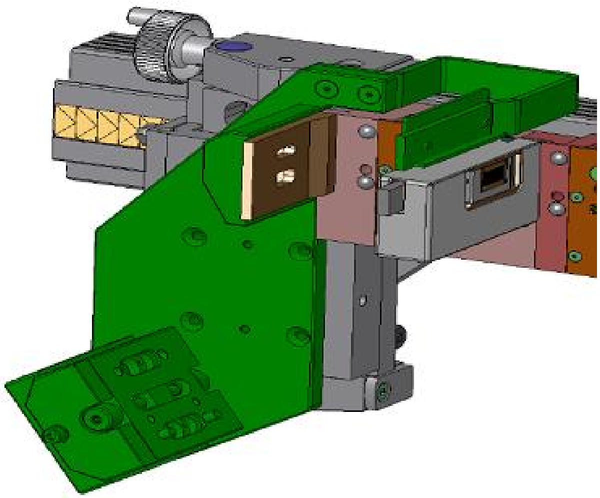


Figure 5.6: Takeoff angle adaptor plate with large negative primary beam deflection angle (-33°). Used for Johansson monochromator. The adapter plate includes a primary beam stop for radiation safety.

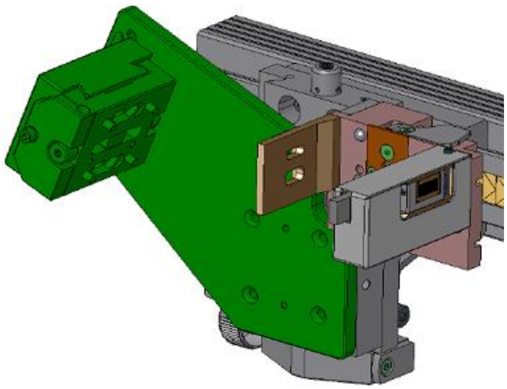


Figure 5.7: Takeoff angle adaptor plate with large positive primary beam deflection angle (+20°)

5.2 Secondary Side: Optics and the Universal Detector Mount

A schematic diagram for mounting components to the secondary track. Typically, position 2 is used for a detector slit and position 3 is used for the rotary absorber. The Universal Detector Mount exists in different versions: Position 2 and 3 are optional. Detectors compatible with the Universal Detector Mount are: LYNXEYE, SOL-XE, Scintillation counter.

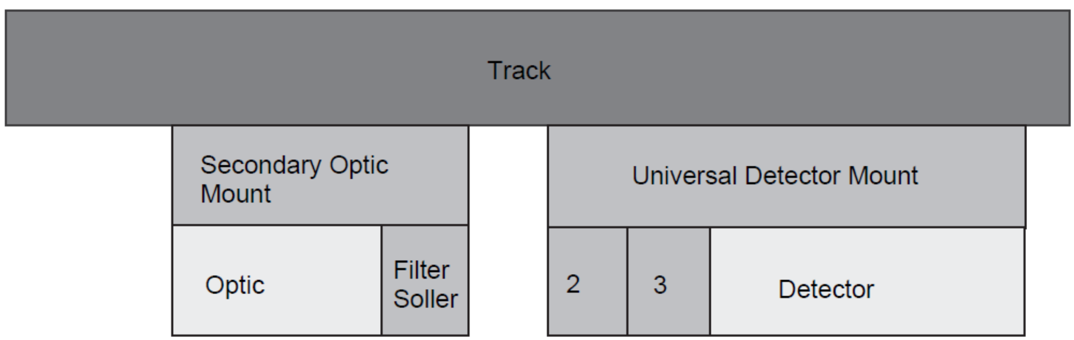


Figure 5.8: Schematic diagram for mounting components to the secondary track

5.3 Automatic Component Recognition

Through the use of identifiers located on the components themselves, it is possible for the DIFFRAC.SUITE software to recognize the presence and position of the most commonly used components. The word “component” refers to the tubes, optics, sample stages, detectors, slits, and filters that are equipped with an identifier. An identifier can either be a holes array, as shown below, or an embedded chip, as shown in figure 6.10. The holes array is used for filters and slits, and the chips for all other components.

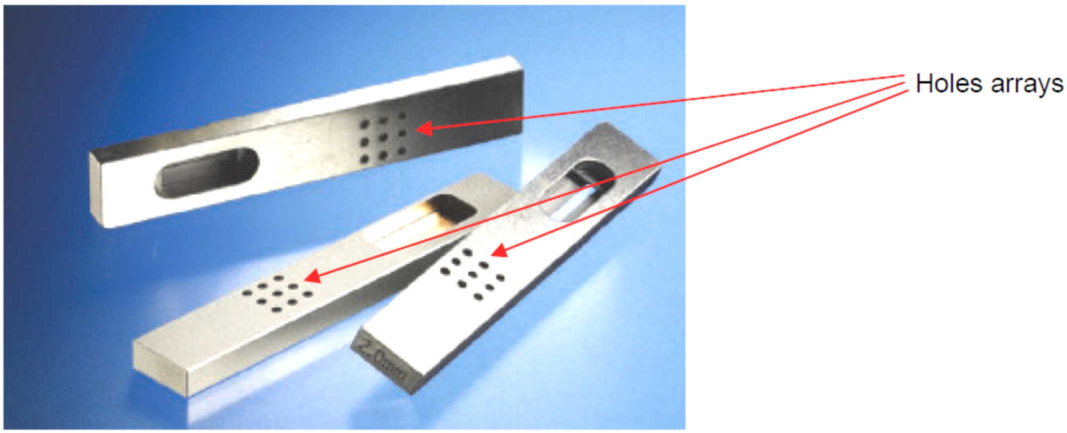


Figure 5.9: Slits with holes arrays, used for automatic component recognition

Automatic component recognition is possible within the framework defined in the Config plug-in, as described in the *DIFFRAC.SUITE User Manual*. In Config, the possible component types, positions and properties are specified. After Config is properly setup, the DAVINCI plug-in then provides an overview of the components currently mounted on the arms and at the center of the goniometer of the connected instrument, as well as their properties. Refer to the *DIFFRAC.SUITE User Manual* for detailed information regarding CONFIG and DAVINCI.

5.3.1 SNAP-LOCK

To change an optics of a Bruker AXS diffractometer in an easy, tool free, and reproducible way is the intention of the SNAP-LOCK concept. The SNAP-LOCK concept, as implemented in the optics mount and optics housing is shown below.

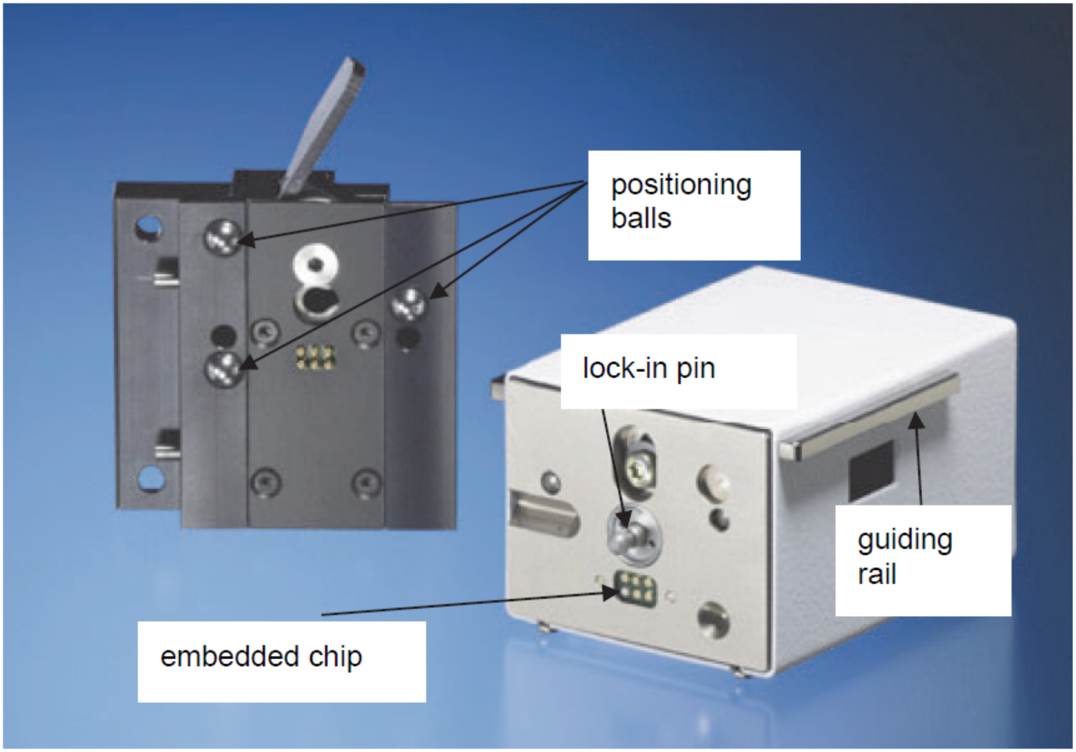


Figure 5.10: SNAP-LOCK and automatic component recognition elements, as shown on an optics mount and optics housing

5.4 Tubes

X-ray tubes with anodes consisting of Cr, Co, Cu and Mo are available as standard products. An X-ray tube with lateral radiation window is used in the D8 diffractometer. This X-ray tube with grounded anode is supplied by a KRISTALLOFLEX 430 X-ray generator which is installed in a console-type housing. The optical focus can be modified by changing the emission angle. A normal emission angle of 6° reduces the projection of the focus to approx. 1/10 of its length.

5.5 Primary Optics

Following optics are available: mirrors, monochromators, capillaries, and slit systems. Additional optics are available for D8 DISCOVER diffractometers. Please refer to Volume 2 of this User Manual for further information regarding these additional optics.

5.5.1 Safety Slit

The D8 ADVANCE / D8 DISCOVER has a new radiation safety concept which is independent of the primary optics mounted. A coarse collimator consisting of a tube side labyrinth and the safety slit defines the maximum primary beam cone. This takes care that the primary beam is always guided towards a lead shielded part of the radiation safety enclosure.

The safety slit fullfils a second purpose besides its safety function. It is used for placing absorber and filters, size limiting slits for Göbel mirrors, axial Sollers.

5.5.2 Mirrors

Mirrors can be used to accept the divergent beam from the X-ray tube and produce a parallel or slightly focusing beam. The background radiation is mostly eliminated, leaving primarily the K characteristic line.

5.5.2.1 Standard Primary D8 Mirrors

- 60 mm Göbel mirrors (curved multilayer construction) for Cr, Co, Cu, and Mo, parallel and focussing. The Mo mirror needs a dedicated adaptor plate for exchanging to other optics without alignment.
- 60 mm Göbel mirror for phase ID, which provides slightly more intensity and a less parallel beam.
- TWIN optics: 40 mm Göbel mirror + variable slit. This enables switching between parallel-beam geometry and Bragg-Brentano geometry.
- TRIO optics: 40 mm Göbel mirror, variable slit, Ge 004 asymetric channel cut monochromator. Enables fast switching between Bragg-Brentano, parallel beam and high resolution/reflectivity configurations.

5.5.3 Monochromators

The focusing primary monochromator of Johansson type generates Kα_1-radiation with high intensity and makes it possible to do X-ray diffraction with high resolution.

The following monochromators are available with the D8:

- Johansson focusing monochromators for Cu Kα_1 radiation, reflection mode. This can be used on theta-2theta systems (D8 ADVANCE / D8 DISCOVER) or theta-theta systems (D8 DISCOVER).
- Johansson focusing monochromators for Cu Kα_1 radiation, transmission and capillary mode. This can be used on theta-theta and theta-2theta systems. In case of a D8 DISCOVER, this monochromator can be used for both, reflection and transmission , in the VARIO setup.

5.5.3.1 Installation of Johansson Monochromator

Changing between the different monochromators is possible via the SNAP-LOCK concept. The optics is prealigned in factory. Only optimizing intensity with the focal adjustment and exact beam centering towards goniometer are required. Switching to other optics requires a different adaptor plate. The procedure is as following:

1. Check if takeoff angle adaptor plate is correct (-33° plate). Install correct plate if necessary.
2. Move beam steering plate to correct distance. Standard measurement circle diameter is 435 mm. The exact tube position depends on the focal length of the monochromator and varies between different pieces. The focal length is saved in the recognition chip of the the optics and can be retrieved by the tools plug-in of the DIFFRAC.SUITE.

5.5.3.1.1 Reflection setup

The distance between tube focus and primary focus slit must be

$$\sqrt{a^2 + b^2 - 2ab\cos(180° - 2\theta_{Johansson})} \approx 332mm \pm \text{ deviation focus lengths}$$

(See figures 5.11, 5.14, and 5.16).

Practically:

- Measurement circle diameter for most sample stages is 435 mm.

1. Therefore, put detector slit, respectively 1D detector to 435 mm diameter.
2. Place primary focus assembly to 435 mm diameter.
3. Install flight tube in front of primary focus assembly and shift tube against it.
4. Move tube against flight tube.
5. Safety slit assembly should touch flight tube assembly.

- detector at 435 mm measurement circle diameter
- primary focus at 435 mm measurement circle diameter
- tube focus 332 mm away from primary focus

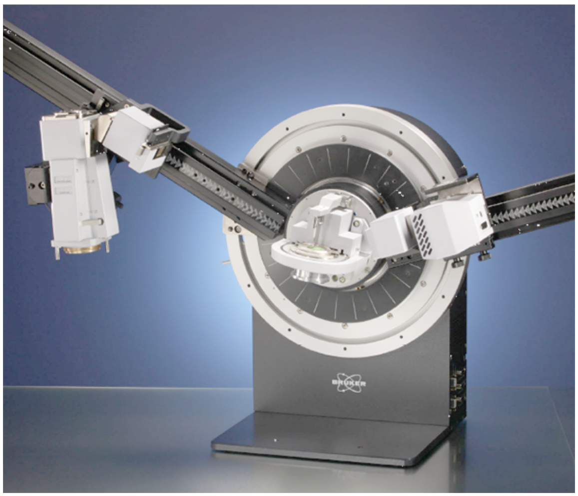


Figure 5.11: Johansson monochromator for reflection mode

5.5.3.1.2 Transmission setup

The distance between tube focus and detector must be primary focal length (a) + secondary focal length (b) of the monochromator (see figures 5.12 and 5.13).

Default value is

$$\sqrt{a^2 + b^2 - 2ab\cos(180° - 2\theta_{Johansson})} \approx 470mm \text{ ± deviation focus lengths}$$

The detector position is defined by the detector slit in case of 0d detectors like a scintillation counter or at the sensor position in case of a 1D detector.

Practically:

- Put detector slit, respectively 1D detector to 435 mm measurement circle diameter. At theta=2theta=0° shift tube towards detector until the nominal focal distance is reached: standard value 470 mm.
- detector at 435 mm measurement circle diameter
- tube focus 470 mm away from detector at theta=2theta=0°

5.5.3.1.3 Check Monochromator Intensity

1. Optimize beam intensity with line focus translation. Remove any samples from sample stage. Move theta and 2theta to 0°. Perform detector scan around 0°. If intensity is not sufficient optimize intensity with help of ratemeter and line focus translation.
2. Steer beam through goniometer centre and redefine optics offset theta. Use standard procedure with glass slit.
3. Redefine optics offset 2theta
4. In case of reflection set up: install beam guide and check that no intensity is lost. Put focal slit (0.2 mm or 0.1 mm) and check that no intensity is lost. Install divergence slit assembly with divergence of you choice.

5.5.3.2 Details on Primary Monochromator in Transmission and Capillary Setup

Figure 5.12 and 5.13 illustrate the beam path for transmission and capillary setup. This setup can be used for theta-2theta and for theta-theta instruments. For theta-theta instruments equipped with other sample stages than the capillary sample stage the goniometer must be installed on a goniometer podium for enabling movements of the detector to negative angles.

Table 5.4: Specification data of Johanssan monochromator for D8 ADVANCE / D8 DISCOVER - transmission and capillary mode

Radiation	**CuKα$_1$**
Crystal	Focusing Ge-crystal (Johansson type), reflection 111
Wavelength	1.54060 Å
Primary focal length a	approx. 120 mm
Secondary focal length b	approx. 360 mm
Incidence angle α	6.722°
Exit angle β	20.559°
2θJohansson	27.281°

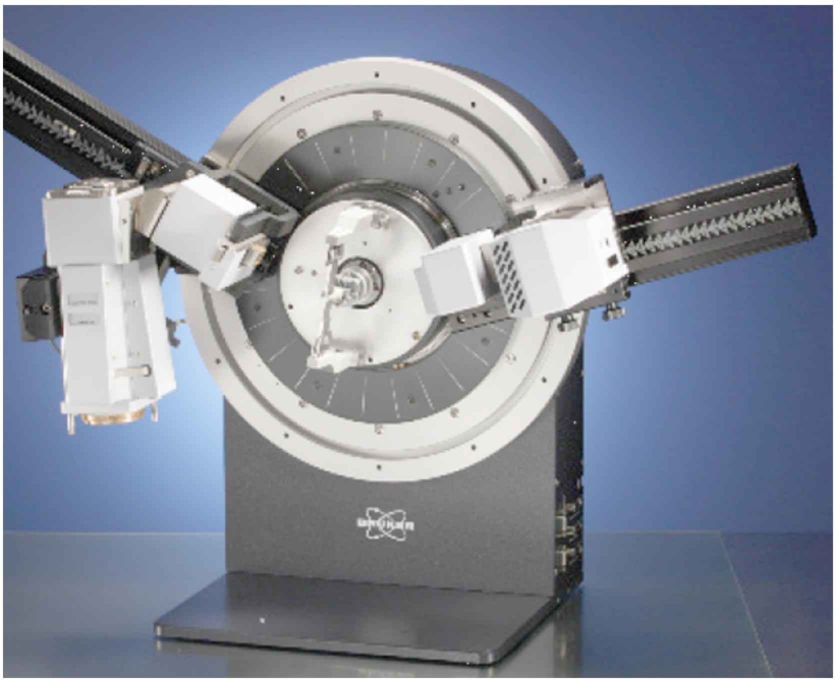


Figure 5.12: Johansson monochromator for transmission/capillary mode

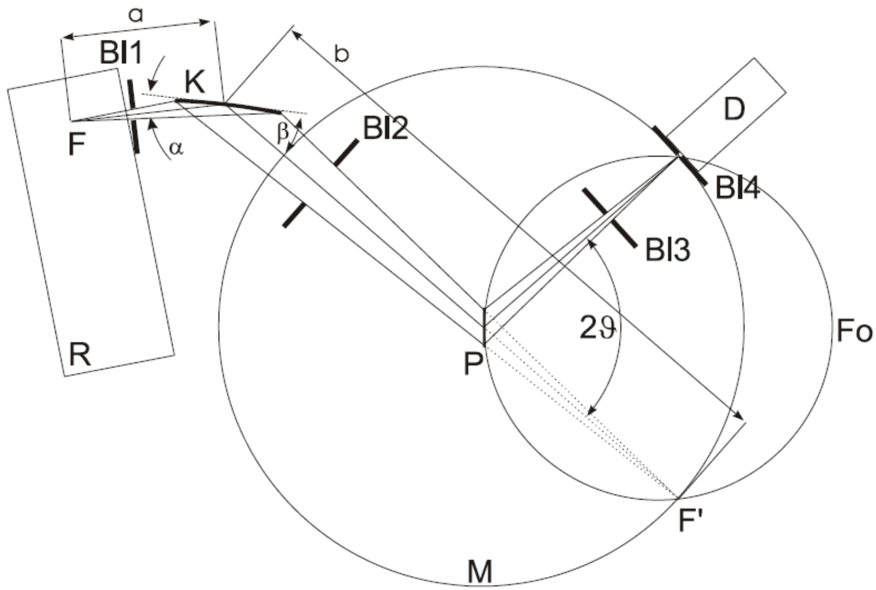


Figure 5.13: Beam path for Johansson monochromator in transmission geometry

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

5.5.3.3 Details on Primary Monochromator in Reflection Setup

Figure 5.16 illustrates the beam path for reflection setup. This setup can be used with theta-2theta instruments (D8 ADVANCE / D8 DISCOVER) or theta-theta systems (D8 DISCOVER). Beam guiding tubes and focus slits are used for optimum back ground and intensity. In front of the goniometer is a dedicated optics mount for putting a divergence slit assembly (figure below).

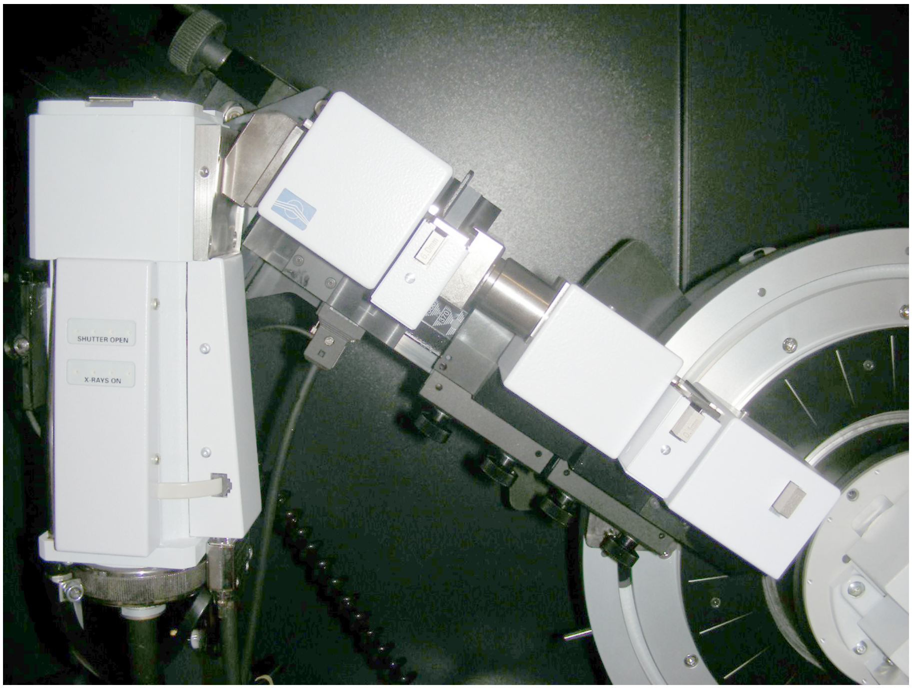


Figure 5.14: Johansson monochromator for reflection mode

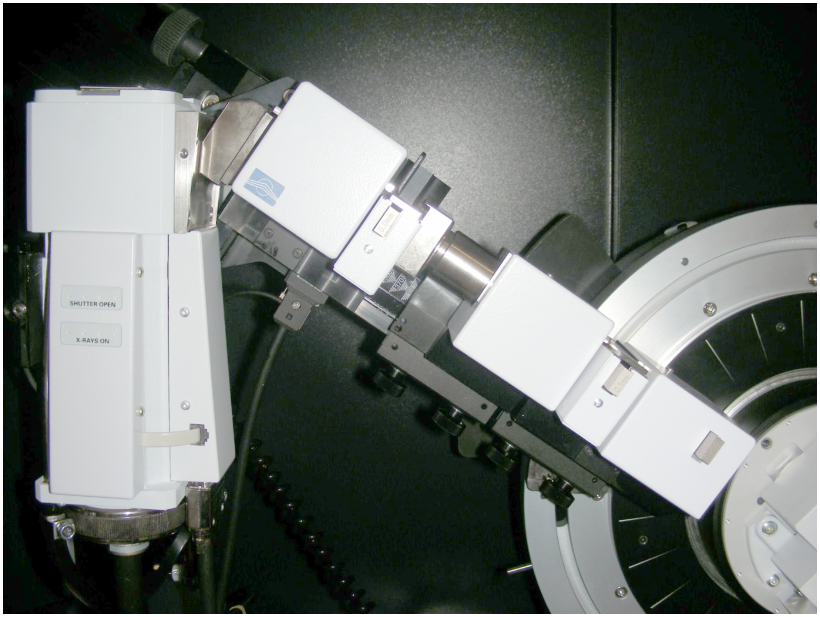


Figure 5.15: Johansson monochromator with reflection setup including guiding tubes, focussing slit, and Bragg-Brentano divergence slit assembly

Table 5.5: Specifications for Johansson monochromator - reflection setup

Radiation	**CuKα_1**
Crystal	Focusing SiO_2-crystal (Johansson type), reflection 101
Wavelength	1.54060 Å
Primary focal length a	approximately 120 mm
Secondary focal length b	approximately 220 mm
Incidence angle α	9.337°
Exit angle β	17.304°
2θJohansson	26.641°

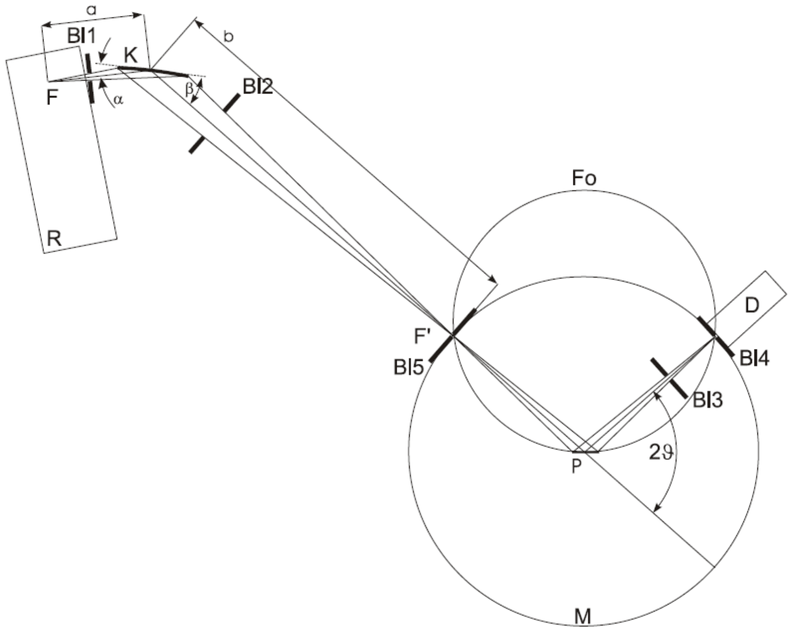


Figure 5.16: Beam path for reflection measurements

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

5.5.3.3.1 Adjustment of the Focus Slit

1. Move beam steering plate and optical bench away from the goniometer center, so that you can mount the focus slit holder and antiscatter tube.
2. Mount focus slit holder at a measurement circle radius of 217.5 mm
3. Mount antiscatter tube. The sample side ending of the antiscatter tube should be flush with the focus slit holder.
4. Move beam steering plate so that the ration safety slit is flush with the tube side ending of the antiscatter tube.
5. Mount fluorescent screen as sample.
6. Set generator to 40 kV and 40 mA.
7. Do not mount focus slit or divergence slit.
8. Check position of the beam on the fluorescent screen. The beam should be in the middle of the fluorescent screen. Check the beam position at a low (approx. 15° Theta) and a high (approx. 90° Theta) incident angle of the beam on the fluorescent screen.
9. Center the beam on the fluorescent screen with the beam steering plate rotation. Check at a low and a high incident angle of the beam on the fluorescents screen.
10. Use 6 mm detector slit and if necessary to prevent saturation 0.1 mm or 0.2 mm Cu absorber.
11. Use decreasing slit sizes as focus slit and optimize the beam intensity through the focus slits with the help of the beam steering plate translation and the rate meter.
12. As a rule of thumb, through a 100 µm focus slit intensity loss of less than 10 % should occurr compared to 1 mm focus slit.

5.5.3.3.2 Adjustment of the Antiscatter Tube

After the adjustment of the focus aperture, the antiscatter tube can be installed. The anti-scatter tube reduces the part of air scattering, which reaches the detector. This leads to an improvement of the signal / background ratio. The length of the antiscatter tube is factory adjusted and should not be changed.

5.6 Secondary Optics

Optics consist of mirrors, monochromators, sollers and slit systems. Additional optics are available for D8 DISCOVER diffractometers. Please refer to Volume 2 of this User Manual for more information regarding these additional optics

5.6.1 Soller Slit

The outer shape of the equatorial Soller slits looks essentially similar as a Göbel mirror. The working principle is the same as for axial Soller slits. They consist of a series of parallel aligned tungsten blades. They are used for parallel beam measurements.

Sizes (divergence in deg):	0.1°	0.2°	0.3°	0.4°

5.6.2 Diffracted Beam Monochromator

The diffracted beam monochromator exist for focussing beam geometry and for parallel beam geometry. Modifications for all standard wavelengths (Cr, Co, Cu, Mo) exist for both versions. The monochromator is prealigned in factory. Standard parameters are inscribed on a recognition chip. After mounting on the system DAVINCI will recognize the monochromator.

Diffracted-Beam Monochromator

The diffracted beam path consists of

- variable slit
- fixed detector slit
- graphite monochromator for CuKα radiation
- scintillation detector

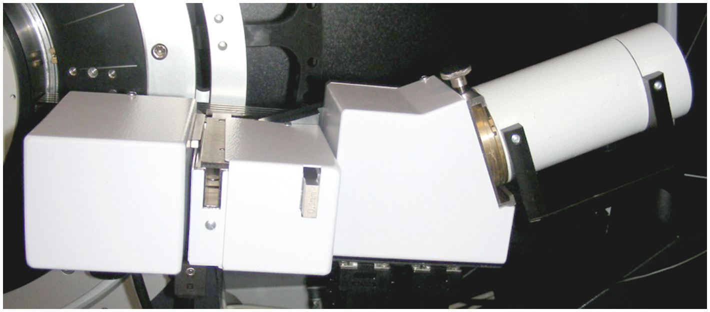


Figure 5.17: Diffracted-beam monochromator

Diffracted-Beam Monochromator for parallel beam

The diffracted beam path for parallel beam consists of

- Soller slit
- variable slit
- slit for limiting diffracted beam width
- flat LiF monochromator for CuKα radiation
- scintillation detector

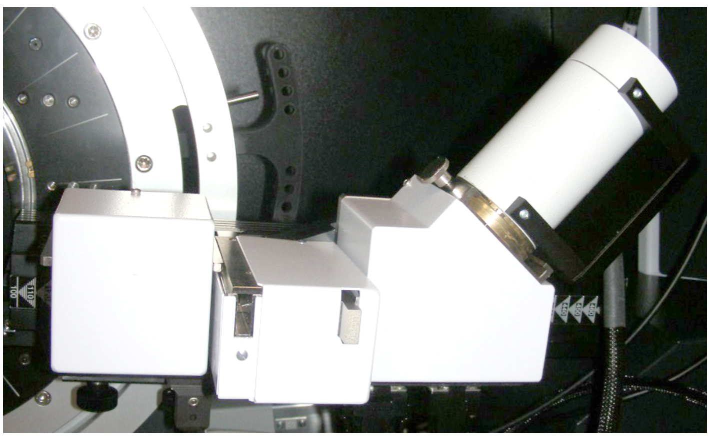


Figure 5.18: Diffracted-beam monochromator for parallel beam applications

5.6.2.1 Application

Diffracted-beam monochromators inserted between the detector slit and the detector suppress fluorescence radiation which may still be excited in the sample in addition to the white spectrum and Kβ radiation.

The $K\alpha_1$ and $K\alpha_2$ peaks cannot be separated because of the mosaic structure of the graphite monochromator crystal.

5.6.2.2 Design and Mode of Operation for Focussing Geometry (Bragg-Brentano)

The diffracted-beam monochromator can be simply mounted onto already adjusted diffractometers, if a detector mount without rotary absorber is installed.

- The diffracted-beam monochromator is prealigned in the factory.
- Further adjustment is not necessary if the diffracted-beam monochromator is delivered together with the diffractometer.
- The diffracted-beam monochromator is fastened to the universal detector mount instead of the detector. The scintillation detector is mounted onto the exit of the monochromator.

The figure below shows the beam path with sample and diffracted-beam monochromator in detail.

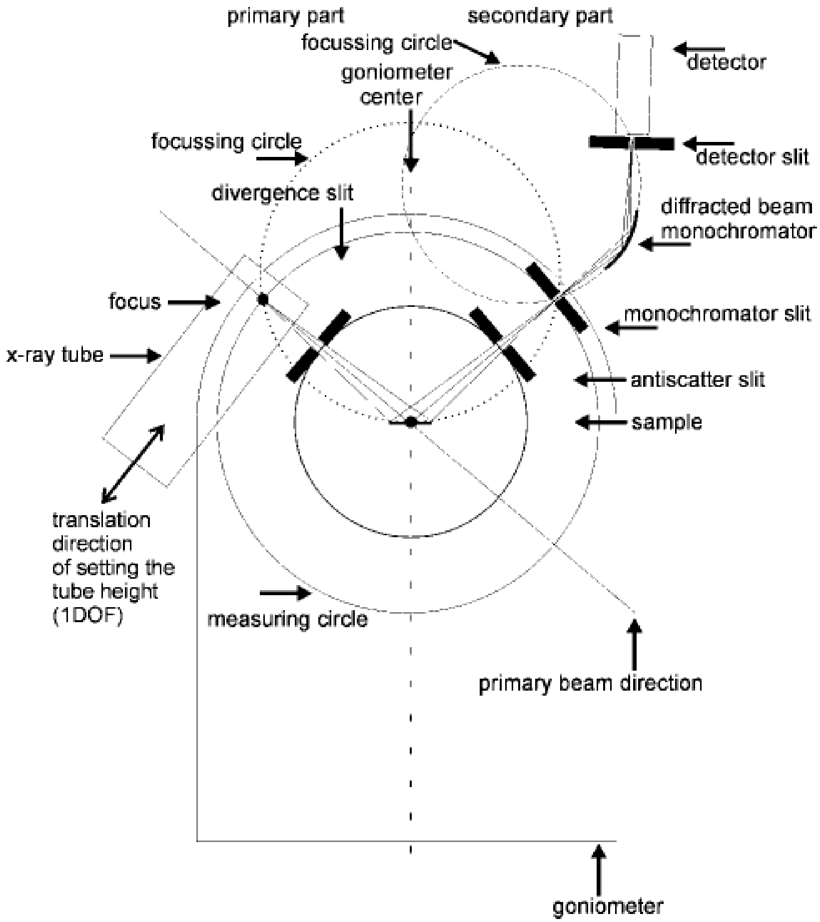


Figure 5.19: Schematic beam path of the D8 with diffracted-beam monochromator

Elimination of the Kβ reflections results in greater clarity of the recorded diffraction pattern. The ratio between the peak and the background is also improved, in case of a strong fluorescence radiation by a factor of up to 1000. The intensity of the signal due to Cu radiation is reduced to approx. one quarter in the process; compared to the wanted signal with a Kβ filter, the loss in intensity is only approx. 50 %, however.

Table 5.6: Intensity yield with a diffracted-beam monochromator compared to a measurement with unfiltered radiation

Anode	E (kV)	Intensity compared to Standard Bragg Brentano without Kβ-Filter
Cr	5.415	1/8
Co	6.930	1/5
Cu	8.048	1/4
Mo	17.479	1/3

5.6.2.3 Technical Data

Table 5.7: Technical Data

Radiation:	Cr, Co, Cu, Mo
Graphite crystal	2d = 0.6714 nm, .002 reflection
Fixed diffracted-beam monochromator	
For scintillation counters	
Mosaic spread approx. 0.4°	

Table 5.8: Wavelenghts as used in the DIFFRAC.SUITE

Anode Material	$K\alpha_{mean}$	$K\alpha_1$	$K\alpha_2$
Mo	0,71073	0,7093	0,71359
Cu	1,54184	1,5406	1,54439
Co	1,79026	1,78897	1,79285
Cr	2,291	2,2897	2,29361

5.6.2.4 Installation

The assembly is shown below.

1. Loosen locking screw (5) on the backside of universal detector mount.
2. Remove detector (1).
3. Fit diffracted-beam monochromator on universal detector mount and fix locking screw (5).
4. Fit scintillation detector on exit flange of diffracted beam monochromator.
5. Tighten locking screw (2).

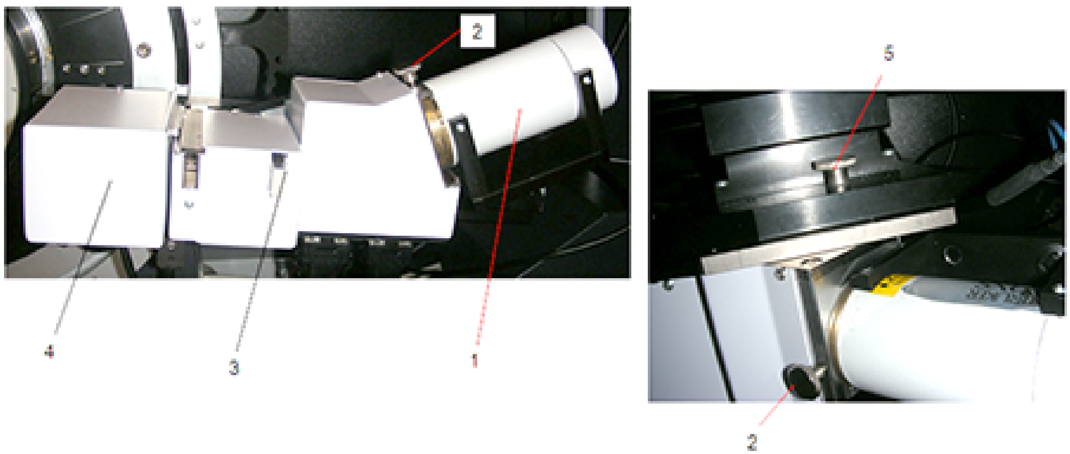


Figure 5.20: Secondary monochromator

1	Detector
2	Locking screw for detector
3	Detector slit
4	Secondary slit assembly
5	Locking screw for monochromator

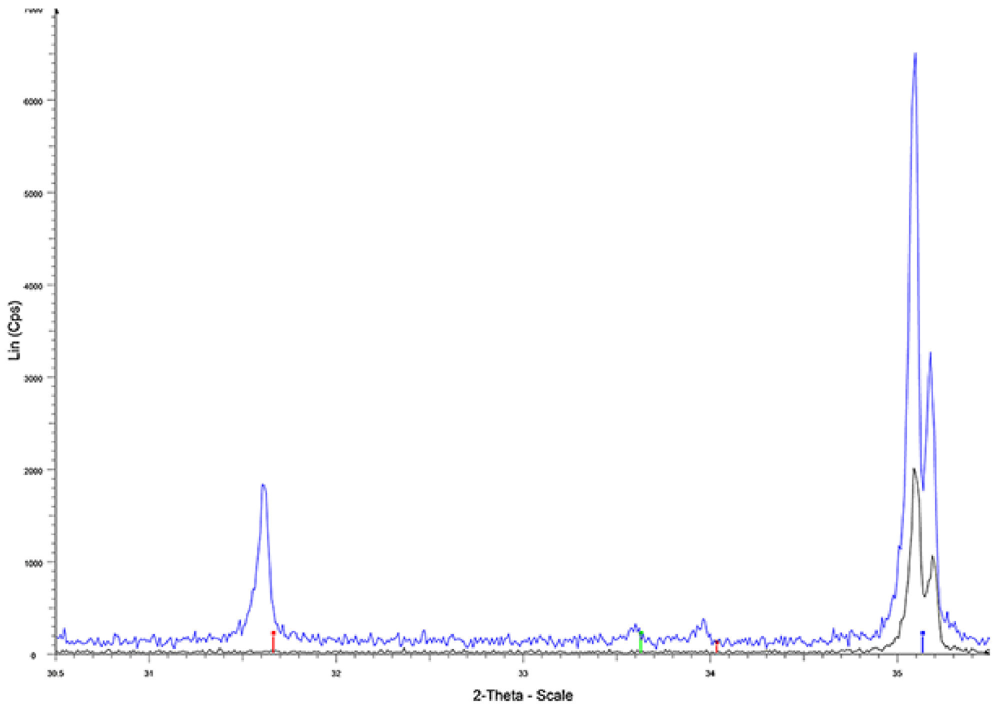


Figure 5.21: SRM 1976A measured with (black) and without (blue) graphite secondary monochromator

5.6.2.5 Application

The flat LiF diffracted beam monochromator is most commonly used in grazing incidence applications. It shall remove e.g. fluorescent radiation during measurement of thin films, surfaces and multilayers. X-ray diffraction using Bragg-Brentano geometry is only partially suitable for this type of application due to the unfavourable peak-to-background ratio. Applying small incidence angles (0.1° to 3°) of the X-ray beam leads to strongly reduced penetration depths and an increased size of the irradiated area. The diffracted beam is paralleled in the secondary Soller slit and can be monochromatized using a flat monochromator. In this case the flat monochromator is LiF.

5.6.2.6 Operation

The picture below shows the basic principle of operation. It should be noted that the incidence angle α of the X-ray beam on the specimen is very small (0.1° < α < 3°)

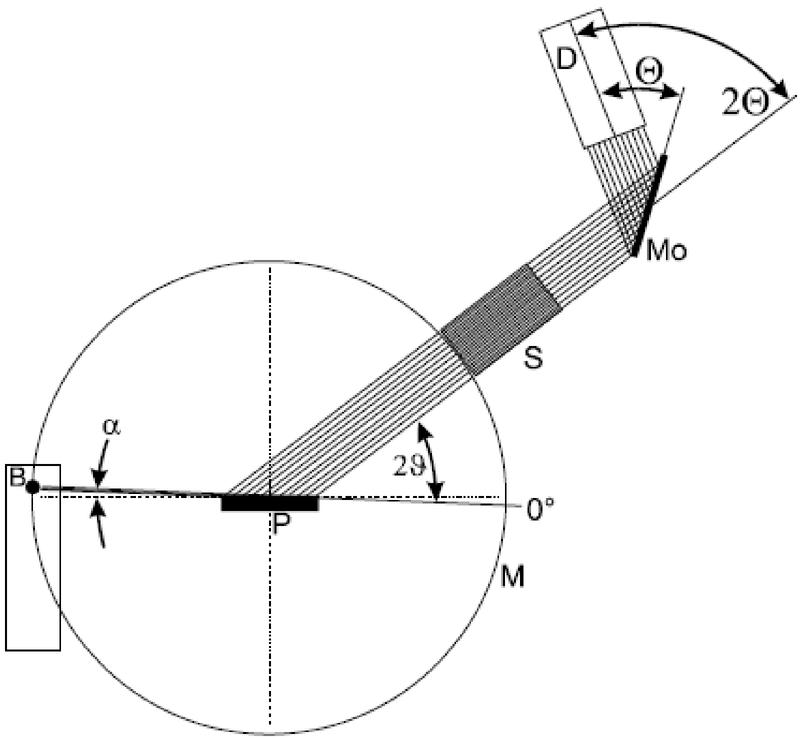


Figure 5.22: Monochromator in parallel beam setup with grazing incidence

5.6.2.7 Technical Data

Radiation Crystal	Mo	Cu	Co	Cr
LiF (100)	20.32°	44.99	52.75°	69.29°

5.7 Slits, Filters, and Absorbers

The D8 ADVANCE / D8 DISCOVER provides several positions where manual slits, filters, and absorbers can be inserted. The main properties will be automatically recognized with help of an array of light barriers. For slits this is the size, for filters the absorbtion for the different characteristic wavelengths. Magnets inside the slit boxes take care that the position of a slit is precise.

Available sizes of plug-in slits (mm):	0.05, 0.1, 0.2, 0.4, 0.6, 0.8, 1, 1.2, 1.5, 2, 6
Micro plug-in slits (Ø in mm):	0.3, 0.5, 1
Available absorbers:	
Cu absorber (µm):	50, 100, 150, 200, 250
Fe absorber (µm):	100
V absorber (µm): 100	100
Available filters and use:	
Ni	as Kβ filter for Cu radiation
Zr	as Kβ filter for Mo radiation
Mn	as Kβ filter for Fe radiation
Fe	as Kβ filter for Co radiation
V	as Kβ filter for Cr radiation

Figure 5.23: Plug-in slits, filters, absorbers

5.7.1 Axial Soller slits

The axial Soller slits are used for reducing the axial divergence in case of line focus applications. They can be mounted without tools. Their properties are retrieved by the D8 ADVANCE / D8 DISCOVER with help of recognition chips. Mounting position is at the sample side of the safety slit and on the filter/Soller position of the secondary optics mount.

Sizes (divergence in deg):	1.6°	2.5°	4.1°	5.1°

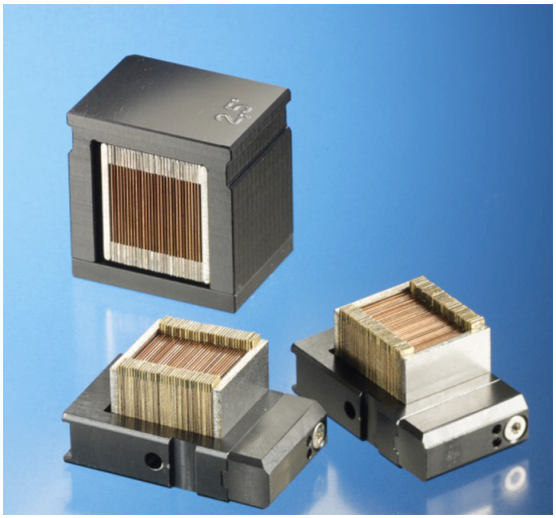


Figure 5.24: Axial Soller slits

5.7.2 UBC collimator

UBC collimators can be mounted at end of the optical bench. In case of the D8 DISCOVER there is an additional version where the UBC collimator base is mounted from the track. They are used for providing a round spot on the sample. In connection with a plugin pinhole they can also be used for getting a round beam shape in case of a line focus tube. The UBC collimator is mounted by a prepositioned magnet holder. The pinhole size is recognized by the system reading the data on the chip placed in the UBC collimator.

Sizes:	50 µm	100 µm	300 µm	500 µm	1 mm	2 mm

The UBC collimator base must be removed from the optical bench if a primary axial Soller shall be inserted.

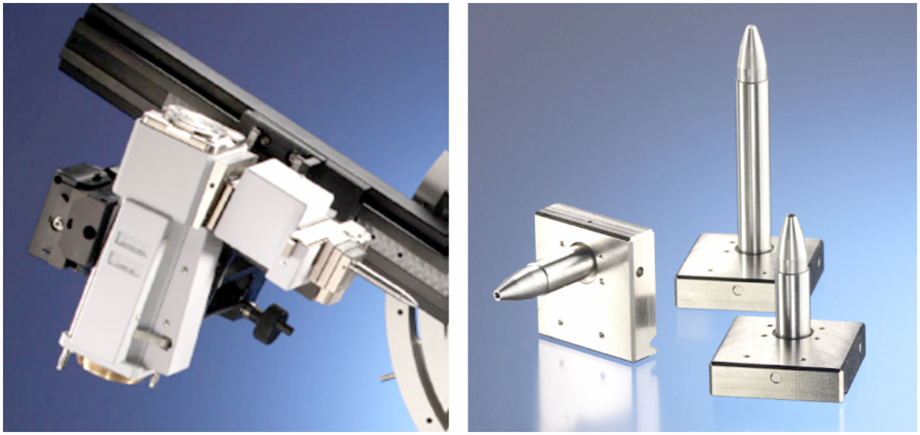


Figure 5.25: UBC collimator

5.7.3 Air Scatter Screen

Especially at small incidence angles and when using 1D detectors like LYNXEYE and VÅNTEC-1 X-rays scattered by air create high background in the data. The air scatter screen takes care of reducing this background significantly. The various sample stages are prepared for inserting air scatter screens closely above the sample surface. The height can be precisely adjusted by fine pitch screws. The air scatter screen can be easily removed without tools when not needed.

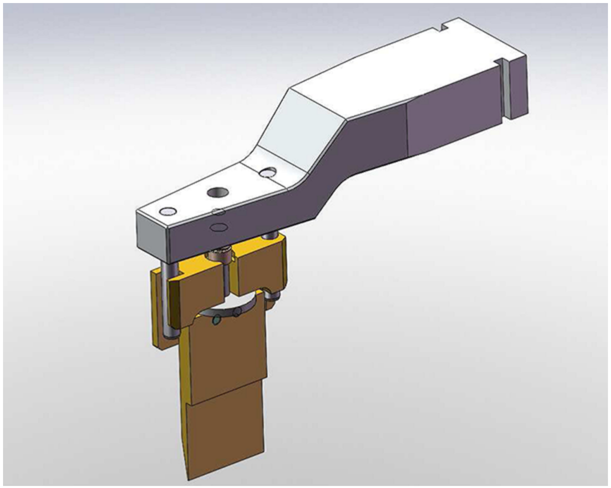


Figure 5.26: Air scatter screen

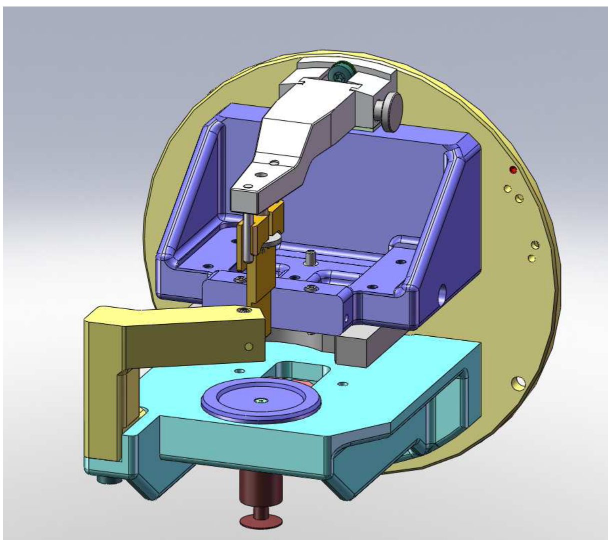


Figure 5.27: Air scatter screen mounted on standard sample stage

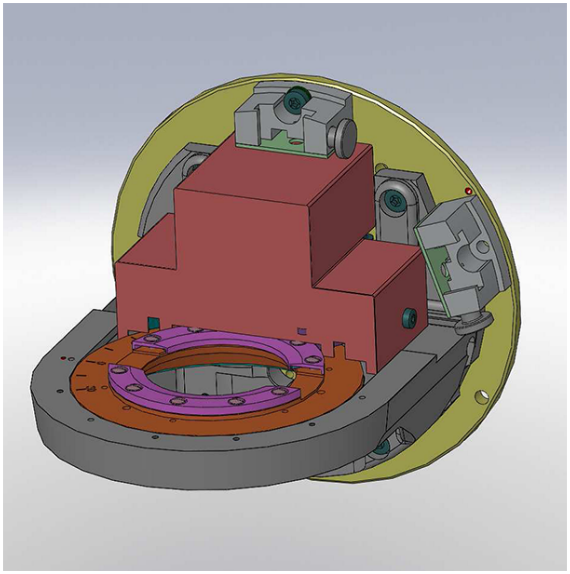


Figure 5.28: The Rotary sample stage provides two insertion positions for the air screen. One is used for reflection measurements and the second in case of transmission.

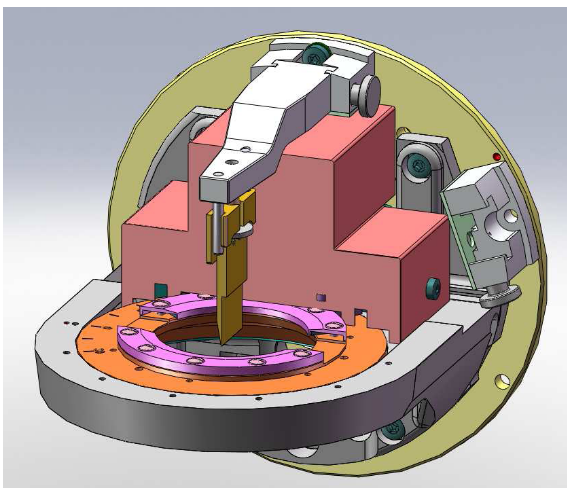


Figure 5.29: Rotary sample stage setup for reflection

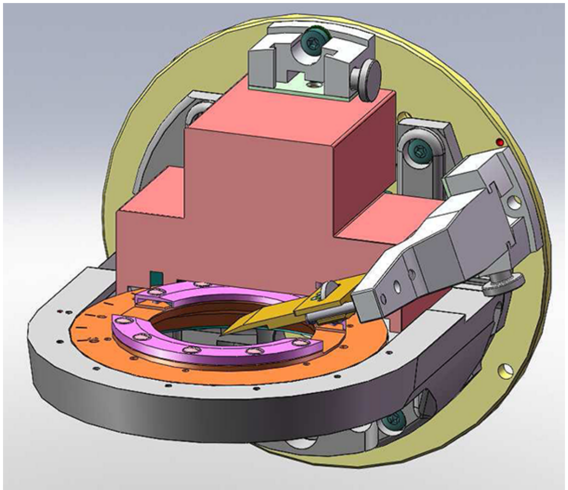


Figure 5.30: Rotary sample stage setup for transmission

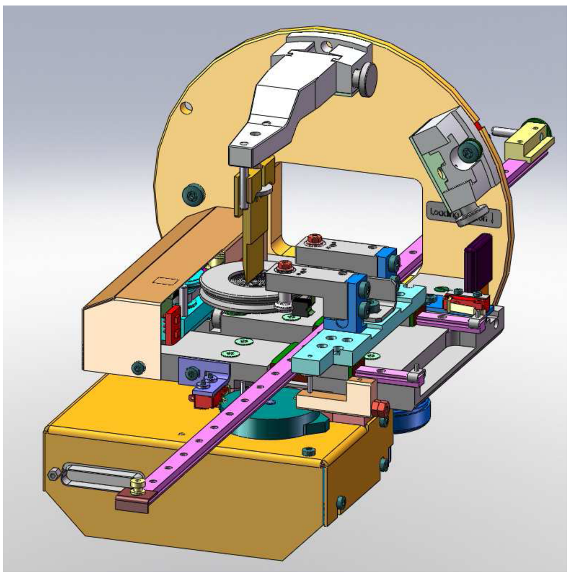


Figure 5.31: FLIP-STICK setup for reflection

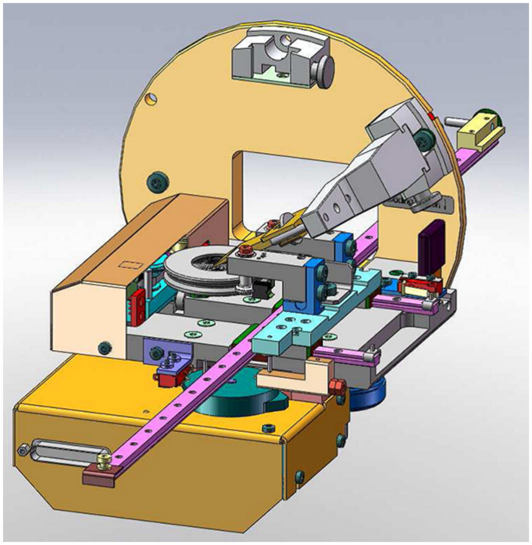


Figure 5.32: FLIP-STICK setup for transmission.

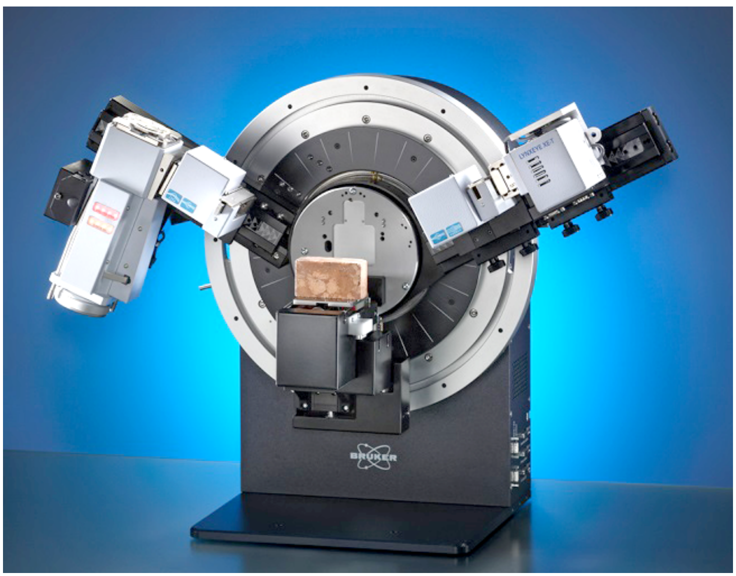


Figure 5.33: Measurement of a large sample with the compact UMC stage

5.7.4 Motorized Anti-Scatter Screen

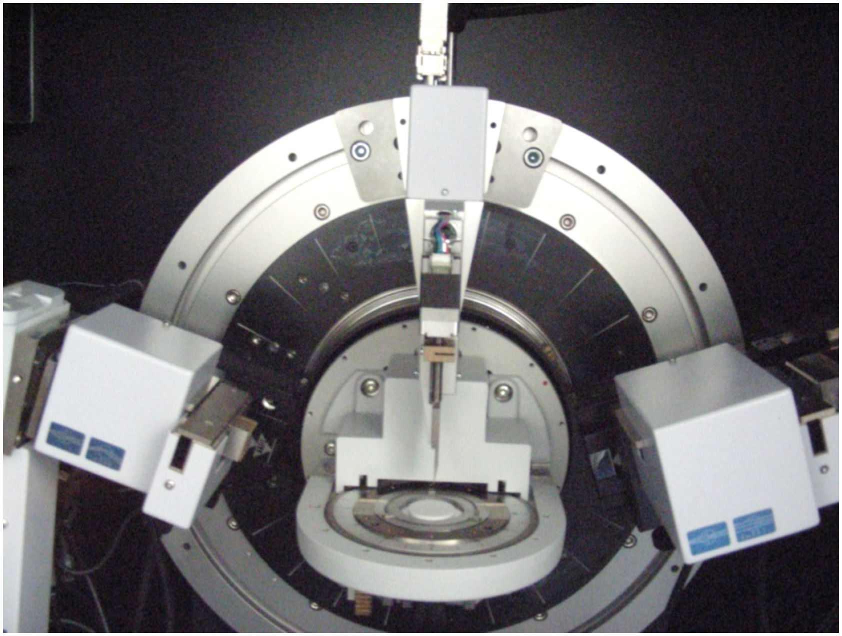


Figure 5.34: Motorized anti-scatter screen mounted on a rotary sample stage

The motorized anti-scatter screen offers the possibility to automatically change the position of the knife during a single measurement or serial measurement.

To achieve the best data quality based on the measurement setup the user can chose between different operation modes of the motorized knife edge.

Fixed sample distance

The knife is moved to a certain defined distance away from the sample surface. The knife will keep this position during the whole measurement. For a second measurement another position can be defined. After choosing **Fixed Sample Distance** as operation mode for the motorized anti-scatter screen the distance between sample surface and knife can be entered as a measurement parameter.

Automatic

The **Automatic** mode only works in combination with a motorized divergence slit. The mode and parameter settings of the motorized anti-scatter knife will always corresponded to the mode and setting of the motorized divergence slit. The **Automatic** mode will automatically chose between **Fixed divergence** and **Variable divergence** mode as described below.

Any fixed divergence opening must be entered in degree.

Fixed divergence angle

For a defined divergence angle of the incident beam the position of the knife edge of the motorized anti-scatter screen is corrected automatically during a measurement when the Theta angle is changing. After choosing **Fixed Divergence Angle** as operation mode for the motorized anti-scatter screen the divergence angle can be entered as a measurement parameter for the movement of the anti-scatter screen.

Variable divergence (V-mode)

For a defined constant illuminated sample area the position of the knife edge of the motorized anti-scatter screen is corrected automatically during a measurement when the Theta angle is changing. After choosing **Variable Divergence** mode as operation mode for the motorized anti-scatter screen the illuminated sample length can be entered as a measurement parameter for the movement of the anti-scatter screen.

5.8 Stages and Sample Holders

5.8.1 Standard Sample Stage

The standard sample stage is adjusted in the factory such that the goniometer rotary axis is in the sample surface. The play compensating disk permits sample stage replacement without play and thus ensures reproducibility. Tightening the centering screw (2) reduces the play between play compensating disk and sample stage ring to zero. Once the play compensating disk has been tightened to the goniometer, the centering screw should be loosened and only re-tightened slightly.

Figure 5.35: Standard sample stage mounted on goniometer

Attention

The centering screw should not be tightened once the equipment has been removed.

5.8.1.1 Insertion of Samples

With help of a quick-release lock the sample is pressed in the standard sample stage. The stop screw can be adjusted without play. The three stop screws (5) are factory-adjusted and secured. The screw (6) may be carefully tightened and adjusted without play.

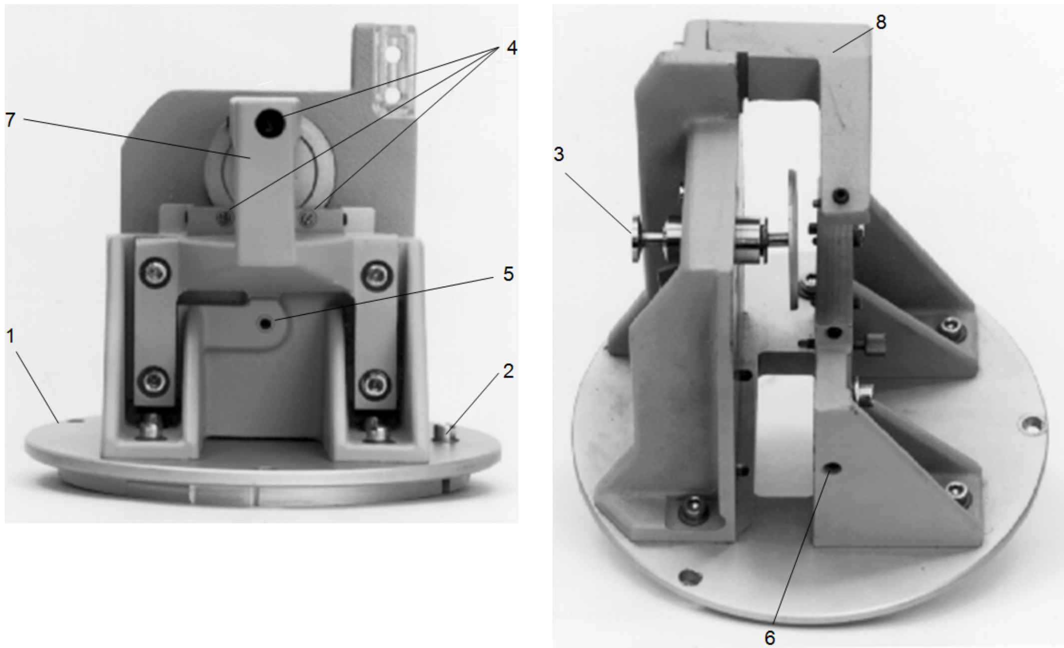


Figure 5.36: Standard sample stage

1	Flange	3	Pressure unit
2	Centering screw	6	Lock Screw for [5]
4	Set screws, factory-adjusted	8	Front stop bracket
5	Set screw		
7	Standard bracket		

Samples up to a thickness of 20 mm may be inserted. An insertion aid (see figure below) is available for samples of 50 x 50 mm and with 50 or 60 mm diameter.

The pressure disk (figure below) permits unshaped samples to be held tight. It may be removed from the pressure pin and replaced by a sample-specific disk shape. Setting the pressure unit to the lower position enables alignment of large and uneven samples to the goniometer centre using the fourth stop screw. The insertion aid must be removed in this case.

The front stop bracket permits large-surface insertion of samples; angle 2Θ is then limited to 150°.

Standard bracket and front stop bracket may be removed and reinstalled in a reproducible manner. Figure 6.39 shows these two possible installations for the standard sample stage.

5.8.1.2 Accessories

Sample holders for powder measurements, Corundum sample for test measurements, calibration slit for 0° adjustment and a silicon single crystal holder for examination of very small samples (see table 6.6) are available for use with the standard sample stage. These accessories are also used with the standard cup for the rotary sample stage.

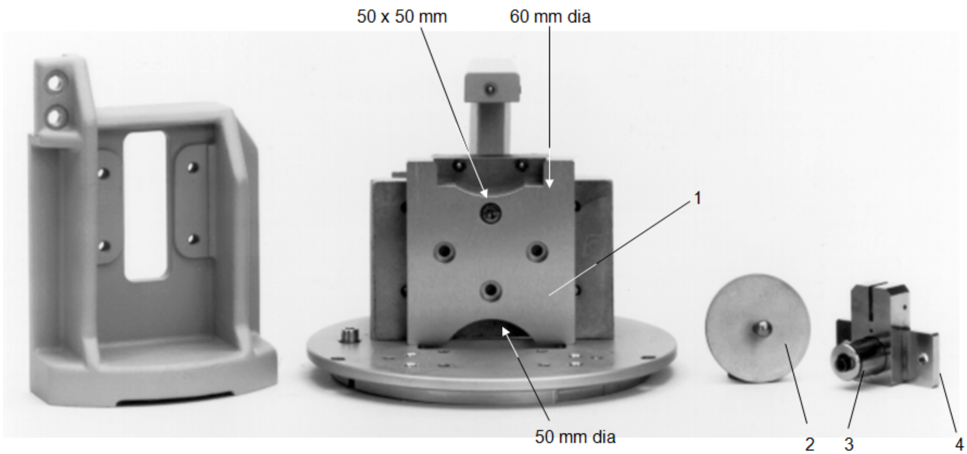


Figure 5.37: Insertion aid and pressure unit

1	Insertion aid	3	Pressure pin
2	Pressure disk	4	Pressure unit

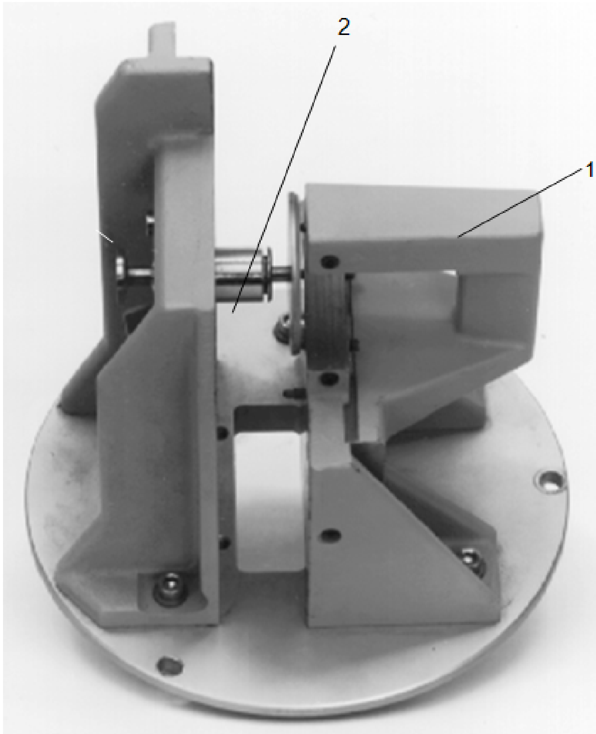
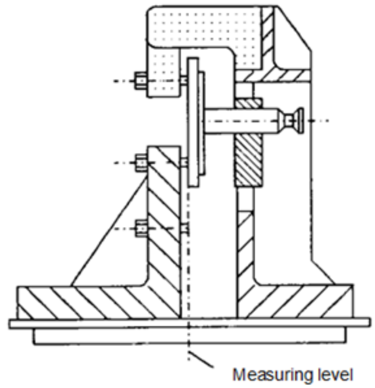


Figure 5.38: Standard sample stage with front stop bracket

1	Front stop bracket
2	Pressure unit

With standard bracket

Preferably for samples:
50 mm diameter, 60 mm diameter or
50 x 50 mm,
max. 20 mm thick



With front stop bracket
(optional)

Preferably for big samples,
max. 20 mm thick

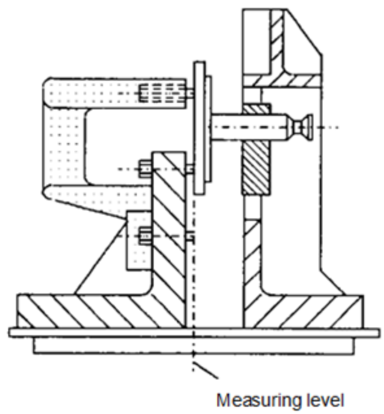
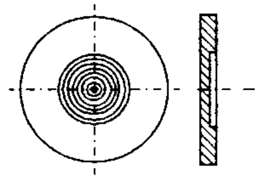
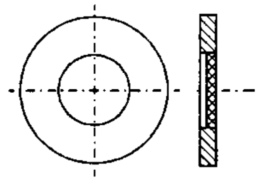
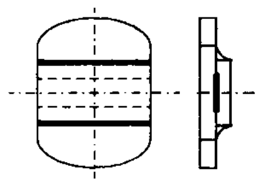
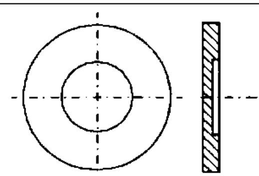


Figure 5.39: Standard sample stage

Table 5.9: Accessories for the standard sample stage

		Used with	**Application**
Sample holder 50 mm diameter		Standard sample stage Rotary sample stage	Powder measurement
Corundum sample 50 mm diameter		Standard sample stage Rotary sample stage	Test measurement
Calibration slit 50 mm diameter 38 mm long		Standard sample stage Standard cup (with mask cap 20°/0° or mask ring 20°/0°) for rotary sample stage	0° adjustment
Silicon single crystal holder 50 mm diameter		Standard sample stage Standard cup (for rotary sample stage	Very small samples, low background

5.8.2 Rotary Sample Stage

Instead of the standard sample stage, the sample stage with stepper motor drive may be used for rotating measurements in reflection and transmission geometry.

Figure 5.40: D8 ADVANCE with rotary sample stage

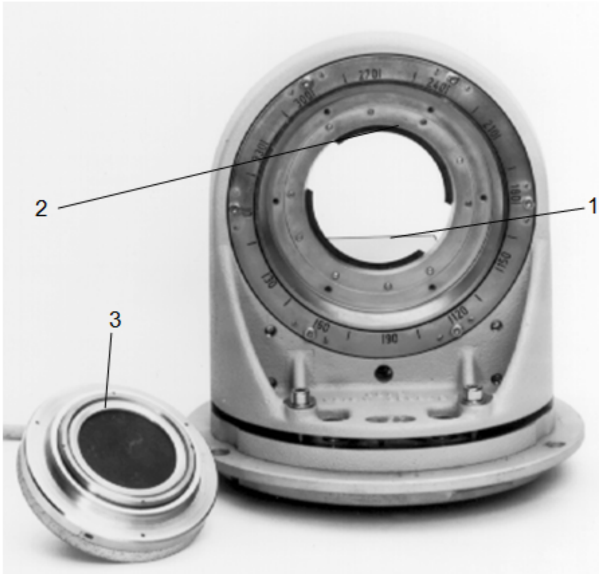


Figure 5.41: Rotary sample stage

1	Drive motor
2	Seating ring for sample cup
3	Transmission sample cup

- The sample is inserted in a sample cup which is held by permanent magnets on a pivotable seating ring. A spring in the cup lid presses the sample against the cup mask.
- The sample cup's design guarantees that the sample surface is in the cup seat level; different mask thicknesses are thus not significant for the measuring result. Masks of 42 mm diameter are available.
- The maximum sample thickness is 50 mm. Plastic centering rings may be used for centering small samples.
- The maximum sample thickness for rotary sample stages is 40 mm (reflection geometry).
- The requirements for preparation are drastically reduced if the sample stage is used as rotating sample stage. Rotating the sample around its surface normal eliminates the influence of particle size and orientation to a great extent.

- The sample stage is used as a transmission sample stage if the crystallite orientation of transmittable objects is to be obtained.
- The sample may be rotated around its surface normal.
- A scale ring with 15° division is applied to the rotary seat and to the sample cup.
- In reflection measurements, measurement of a rotating sample is possible from $2\theta = 0°$.Transmission technique permits measurement up to $2\theta = 110°$ (in case of Bragg-Brentano geometry). Rotational speed can be adjusted continuously between 15 and 120 rpm.
- The angular positions can be selected in steps of 0.28°. Reference marks at 0° (mechanical) permit electronic angle correction.
- For measurements in reflection and transmission geometry specific sample cups must be used. Otherwise the sample surface is not positioned in the goniometer axis.

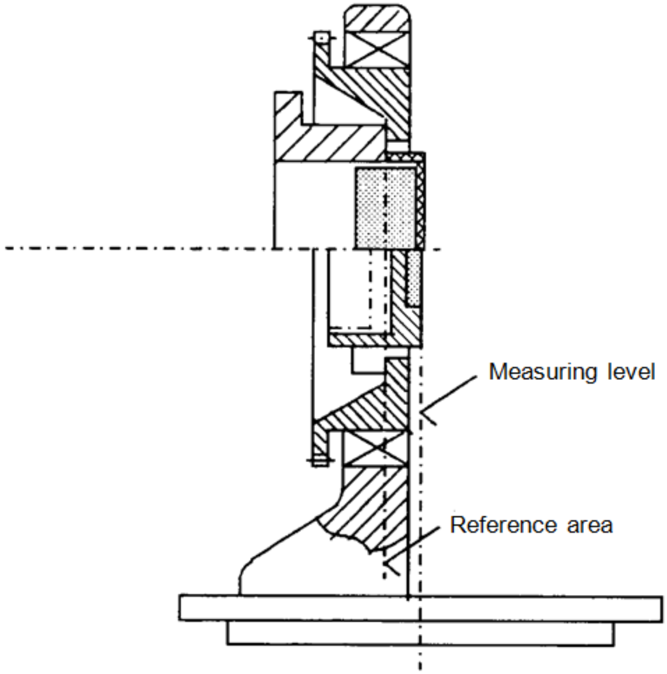


Figure 5.42: Design of the rotary sample stage

5.8.2.1 Accessories

Universal cup

A universal cup permits measurement of samples up to 50 mm diameter and 40 mm thickness.

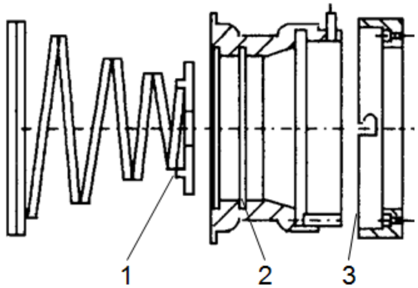


Figure 5.43: Universal cup

1	Pressure unit
2	Upper part
3	Lower part

Intermediate ring

Smaller samples may be supported using an intermediate ring in the universal cup.

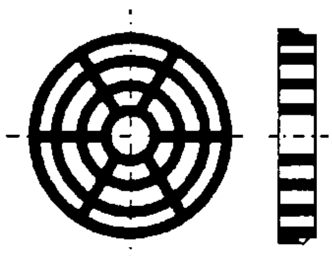


Figure 5.44: Intermediate ring, 50 mm diameter.

Sample holder

The accessories for the standard sample stage (sample holders, Corundum sample, calibration slit and silicon single crystal sample) may also be used with the universal cup.

Transmission cup

A transmission cup for samples of up to 50 mm diameter and 4 mm thickness is provided for transmission measurements.

Figure 5.45: Transmission cup

Sample holder

A sample holder which may be used at both sides permits measurements with rotating samples from $2\theta = 0°$. Powder samples can be inserted in one side, while samples of up to 50 mm diameter and 15 mm thickness can be accepted in the other side. A rupture joint permits the sample holder to be broken easily in order to remove the thick sample. The sample holder may be reused for thick samples.

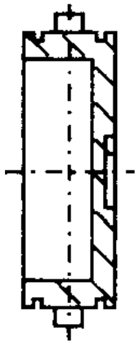
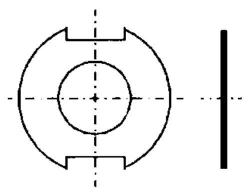


Figure 5.46: Sample holder

Further accessories are sample holders for measuring fibres and threads:

		Used with	Application
Sample holder, 50 mm diameter		Universal cup for rotary sample stage Transmission cup for rotary sample stage	Examination of fibres and threads

5.8.3 FLIP-STICK

The FLIP-STICK is meant for routine reflection and/or transmission measurement of up to 9 samples of different types.

Figure 5.47: FLIP-STICK in reflection mode

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 FLIP-STICK is equipped with a bayonet; therefore the installation is similar to the rotary stage (cf. *Mounting Sample Stages [▸ 155]*). DAVINCI takes care that the stage is recognized after mounting. Switching off the D8 is usually not necessary, as normally all motor cables are already connected. It is compatible with vertical θ−θ and θ−2θ goniometers.

5.8.3.1 General Features

Table 5.10: Features

Reproducibility in 2θ of identical sample (σ)	better ± 0.005°
Reproducibility of standard Corundum (σ)	better ± 0.01°
Precision of surface position of sample rings	better ± 0.025 mm
Measurement height 150 mm	up to 9 samples
Rotation	0 or 30 rpm
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, 500 mm diameter, goniometer podium needed for raising height of goniometer

5.8.3.2 D8 Standby

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

5.8.3.3 Change of Sample Rings

1. Push sample release.
2. Push sample ring against support rings.
3. Release actuator.

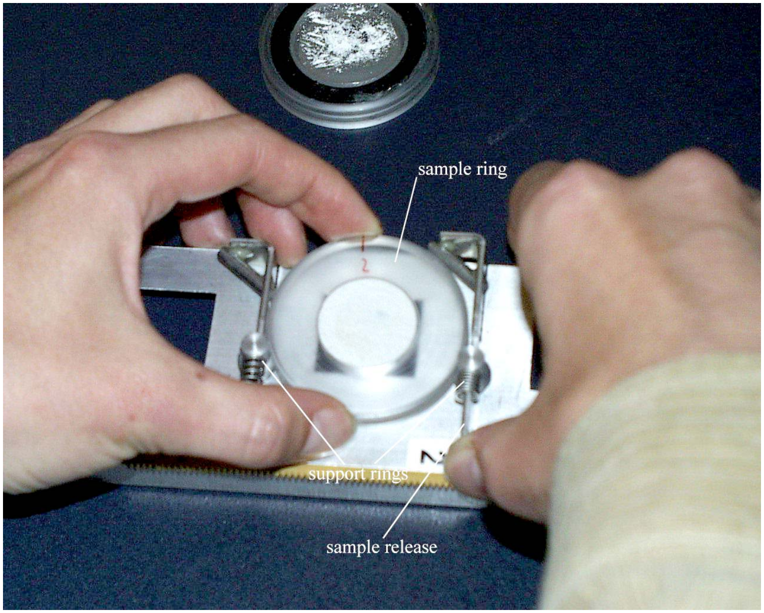


Figure 5.48: Changing of sample rings

5.8.3.4 Release of Magazine

1. Move magazine to release position, if necessary: Sample magazine horizontal and magazine at position of sample 7, use **Magazine release** button in DIFFRAC.TOOLS).

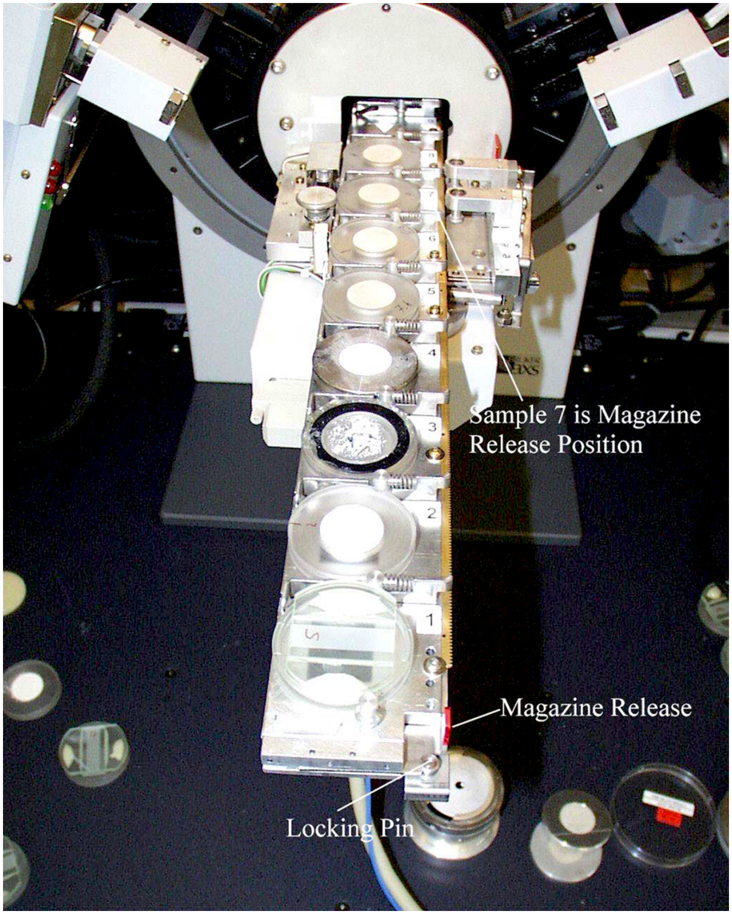


Figure 5.49: FLIP-STICK in release position

2. Push red **translation release** actuator to the right on 9 Position Sample Stage to give free translation of magazine.

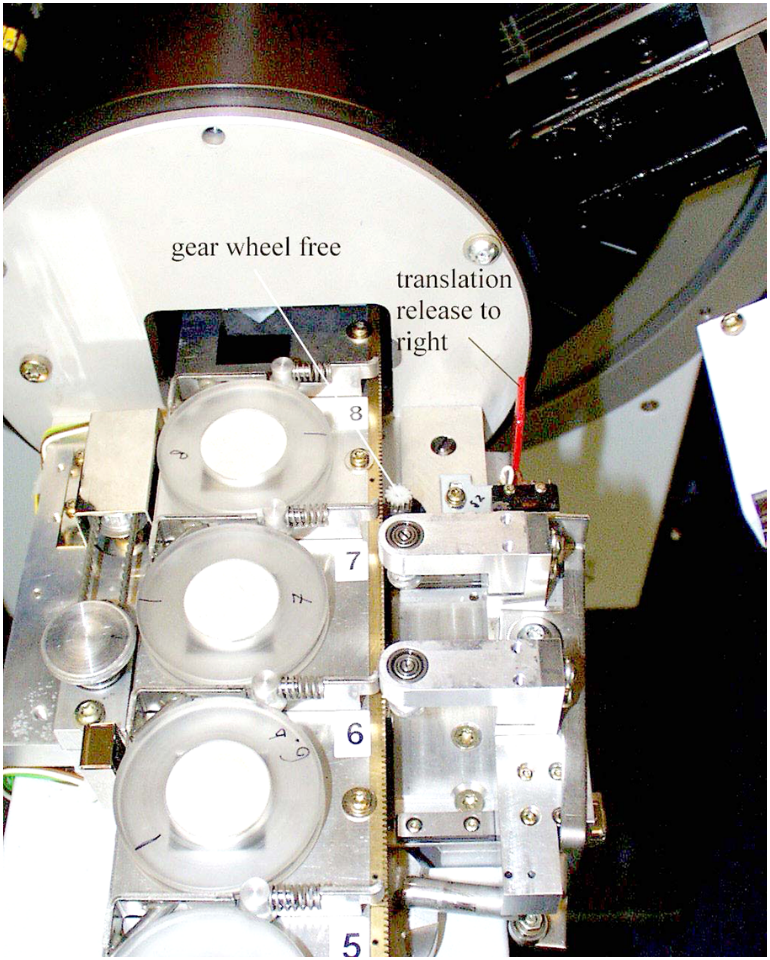


Figure 5.50: Magazine translation released

3. Move out magazine as far as possible. Use red **Magazine release** actuator on magazine to take out magazine.

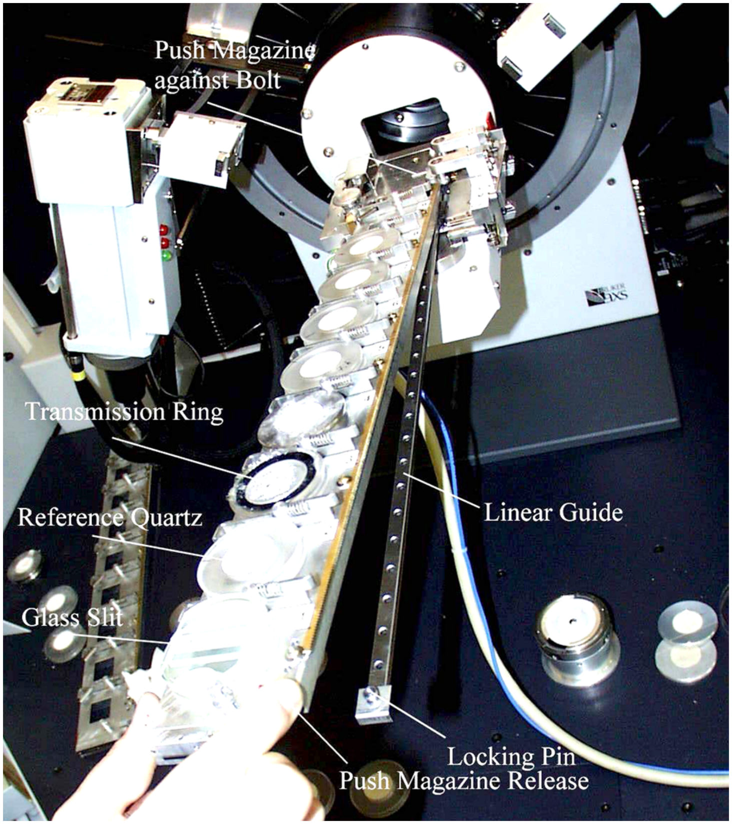


Figure 5.51: Changing of sample magazine

NOTICE
Attention
Do not bend linear guide of 9 Position Sample Stage!

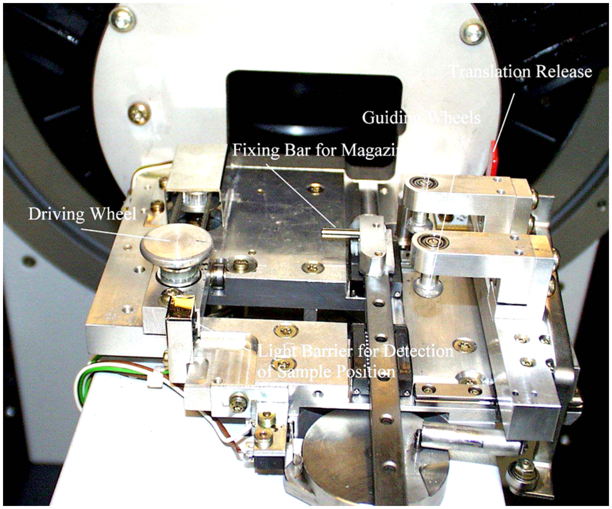


Figure 5.52: FLIP-STICK without magazine

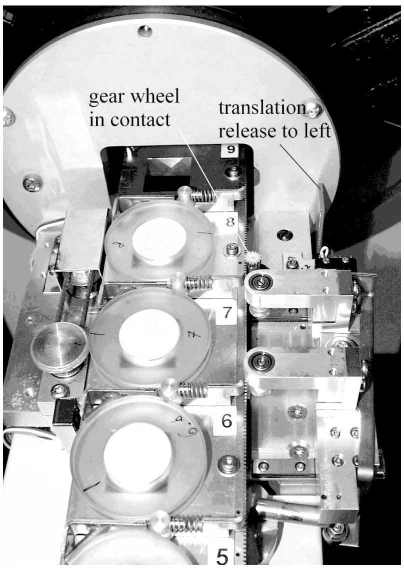


Figure 5.53: FLIP-STICK with magazine translation in working condition

5.8.3.5 Insertion of Magazine

NOTICE

Attention

Do not touch light barrier!

1. Red “translation release” actuator of sample translation must be in out position (right, Figure 5.50).
2. Move out linear guide of 9 Position Sample Stage as far as possible – **do not bend!**
3. Shift magazine against bolt (Figure 5.51, Figure 5.52).
4. 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 (Figure 5.49).
5. Translate magazine roughly to position of sample 7
6. Push red **Translation release** actuator of sample stage back to working position (left, Figure 5.53).
7. Initialise 9 Position Sample Stage.

5.8.3.6 Transmission Measurements

The figue below shows the D8 with FLIP-STICK in case of a transmission measurement. T ake sample rings for transmission measurement (C79298-A3244-D81). Instead of performing **Coupled Theta/2Theta scans** use **Offset coupled Theta/2Theta scan** scans with start value of theta increased by 90° compared to the **Coupled Theta/2Theta scan**. Reflection and transmission measurements might be mixed. The accessible 2θ range is larger than 0° - 90°.

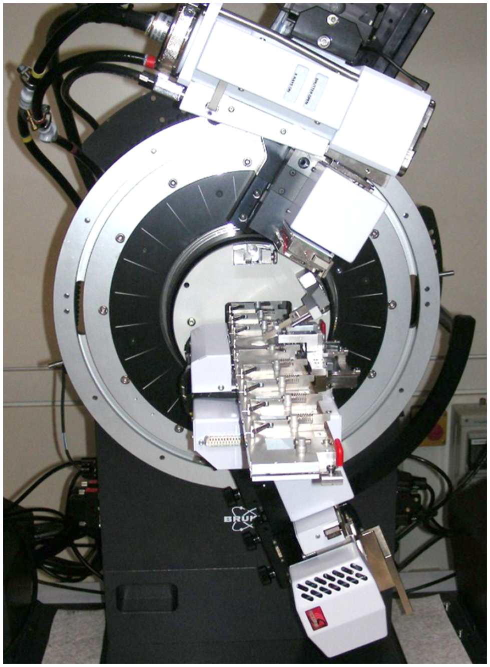


Figure 5.54: FLIP-STICK in transmission mode

5.8.3.7 Accessories

Basic Set Sample Rings (C79298-A3244-D80)

- 1 glass slit 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

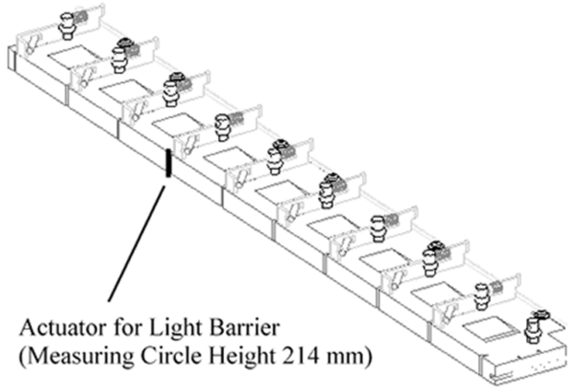


Figure 5.55: Standard magazine for reflection and transmission

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

C79298-A3244-B252,

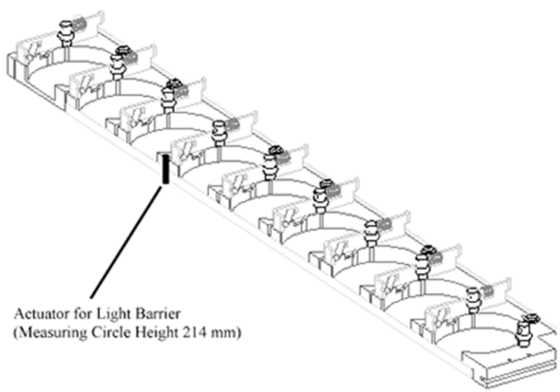
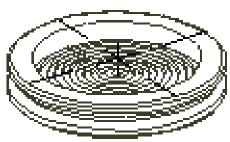


Figure 5.56: Magazine for reflection measurements on thick samples

5.8.3.8 Sample Rings

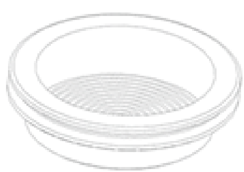
5.8.3.8.1 Standard Sample Ring

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



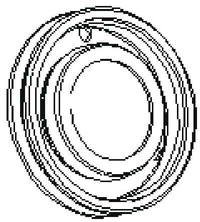
5.8.3.8.2 Thick Samples

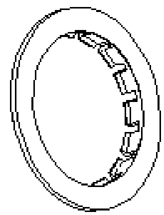
Part Number 9 Sample Rings	Ø Sample	Sample Height	Ring Height	Material
C79298-A3244-C254	40	17	20	Steel



5.8.3.8.3 Air Sealed

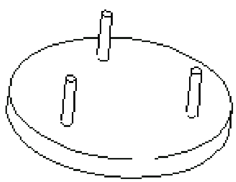
Part Number 9 Sample Rings	Ø Sample	Sample Height	Ring Height	Material
C79298-A3244-D97	25	1	8.5	PMMA
C79298-A3244-D98	25	4	8.5	PMMA



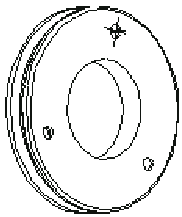
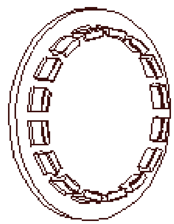


5.8.3.8.4 Preparation Tool

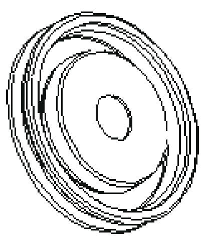
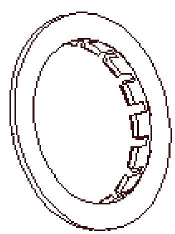
Part Number	
C79298-A3244-B259	Preparation of back loading, air sealed, transmission, side loading sample rings)



5.8.3.8.5 Back Loading Sample Ring

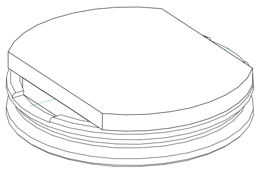
Part Number 9 Sample Rings	Ø Sample	Sample Height	Ring Height	Material
C79298-A3244-D88	25	6.5	8.5	PMMA
 				

5.8.3.8.6 Transmission Sample Ring

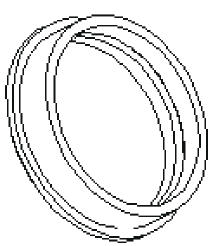
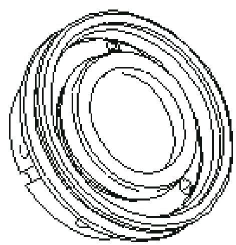
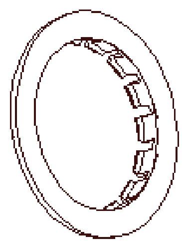
Part Number 9 Sample Rings	Ø Sample	Sample Height	Ring Height	Material
C79298-A3244-D81	11		8.5	PMMA
Transmission measurements (e.g. Polymers), monochromator recommended (e.g. Göbel mirror, $K\alpha_1$ Transmission)				
 				

5.8.3.8.7 Glas Slit

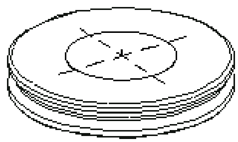
Part Number	
C79298-A3244-B255	**Alignment**



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

5.8.3.8.8 Silicon Single Crystal Sample Ring (Low Background Sample Ring)

Part Number	
C79298-A3244-B249	Silicon Single Crystal Sample Ring
	

5.8.4 AUTO-CHANGER

- The AUTO-CHANGER sample stage is designed for routine powder diffraction in reflection and transmission geometry of up to 90 or 54 different samples. The number differs depending on sample thickness chosen. Standard sample rings as described in the FLIP-STICK section are compatible with the AUTO-CHANGER.
- Sliding away the robot unit to the right or left side allows an easy access to the goniometer centre. This opens, together with the bayonet ring, an easy, reproducible way to exchange the rotary sample stage of the AUTO-CHANGER by e.g. a heating stage on the goniometer.
- A detailed description of all features can be found in the dedicated *AUTO-CHANGER User Manual* (DOC-M88-EXX101).

Possible measurements:

- Reflection measurements
- Transmission measurements, goniometer podium needed for raising the goniometer.

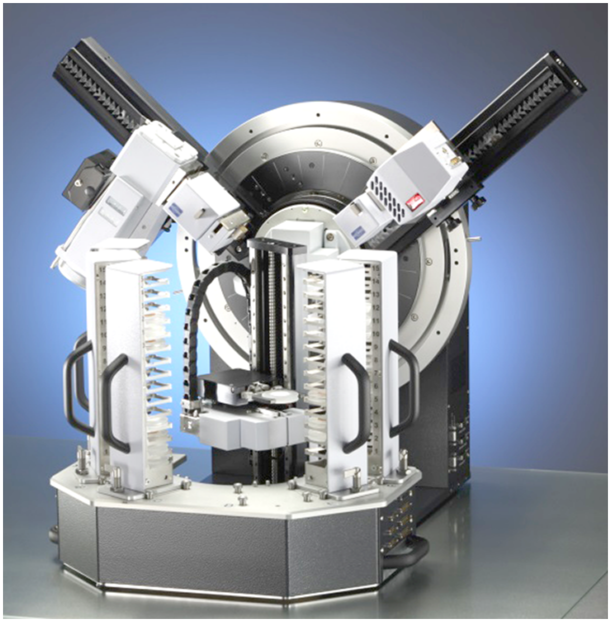


Figure 5.57: AUTO-CHANGER - sample stage for automatic exchange of many samples

5.8.5 Compact UMC Stage

- The motorized UMC Sample Stage (Part-Nr.: A24D298) was designed for accurate alignment and/or mapping of a sample in the X-ray beam of vertical D8 diffractometer. Completely implemented in the measurement software, the stage provides 25 mm travel in X-direction, 70 mm in Y- and 50 mm in Z-directions which can be used for scanning or for alignment purposes.
- The size of the support plane is 80 x 80 mm; if required additional sample holders can be mounted using the available tap holes.
- Three free connection ports on the 4-axis motor driver board of the D8 controller are required.

- For a simple and accurate adjustment of the instrument, which shall run the XYZ Sample Stage, it is recommended to equip the D8 System with a standard / rotation sample stage.

Included in delivery are:

- XYZ-Stage with the mounting base for D8 (measuring height 150 mm)
- three motor cables
- holder for flat samples up to 50 x 50 mm size
- glass slit
- Corundum Standard sample.
- fluorescence sample

Figure 5.58: Compact UMC stage

NOTICE

Substantial damages to the system

PRIOR MOUNTING: In order to prevent substantial damages to the system you must turn off the power of the diffraction system completely before connecting or disconnecting any cables to the X-ray tube, the radiation detector, motors or to the various accessory components.

- ▶ Be aware, wrong settings of the motors phase current will damage the motors!
- ▶ Turn off the power of the diffraction system completely

The XYZ stage is mounted to the sample stage adapter ring in the same way as the standard or rotating sample stage. The bayonet locking permits the sample stage replacement without play and thus ensures reproducible mounting. Tightening the centering screw (see also *Motorized UMC-Sample Stage User Manual,* DOC-M88-EXX221) reduces the fit play between the centering disk and the sample carrier ring to zero. Once the centering disk has been tightened to the goniometer and the stage fixed to the sample carrier ring by the three fixing screws, the centering screw should be loosened and only re-tightened slightly.

Attention

The centering screw should not be tightened once the equipment has been removed.

Install the UMC Stage and connect X, Y and Z cables to the 4-axis Indexer Board B232 (series 2 version). Add the stage to the instrument configuration, following the Installation and Configuration of the *Motorized UMC-Sample Stage User Manual*.

5.8.6 Compact Eulerian Cradle

The Compact Eulerian Cradle with motorized Z drive (Part no. A24D385) integrates Chi and Phi rotations and Z translation into one sample stage with minimum space requirements. Common texture or residual stress samples, powder samples, as well as thin films and small wafers can be mounted by selecting an appropriate sample fixture. The compact design of the Eulerian Cradle allows short sample-to-detector distances advantageous for many applications. The Chi- and Phi-rotations, and the Z translation are motorized and can be used for positioning as well as scanning.

For a simple and accurate adjustment of the instrument it is recommended to equip the D8 ADVANCE, which shall run the Compact Eulerian Cradle with Motorized Z, with a standard / rotation sample stage.

Technical specifications

- for D8 ADVANCE type instruments Theta/Theta or Theta/2Theta, 150 mm measurement height, 500 mm or larger measurement circle diameter (depending on the space requirements of the optical components), three free ports on a 4-axis Indexer Board (B232 or B105) are required.
- Bayonet mounting base for an alignment-free exchange of sample stages
- Chi circle: -5° to 95°, step size 0.02°, max. speed 20°/s
- Phi circle: unlimited, step size 0.001°, max. speed 3 rpm
- Motorized Z-translation: max. 2 mm, min. step size: 0.0005 mm
- Max. sample weight, depending on sample holder: 250 g
- Max. sample height, depending on sample holder: 25 mm
- Max. sample diameter: 70 mm

Included in delivery are:

- Transport Box
- *Compact Eulerian Cradle with Motorized Z User Manual* (DOC-M88-EXX220)
- Three motor cables
- Sample fixture for flat samples (for Compact Eulerian Cradle).
- Fluorescence screen with holder and integrated pinhole, for Compact Eulerian Cradle
- Glass slit
- Corundum sample
- Fixed diagonal slit, 1.0 mm opening
- Fixed pinhole micro slit, 0.3 mm diameter
- Bearing and gearing grease

Optional accessories

- XY-sample stage, (Part-Nr.: A19B53). For maximal 2 mm thick samples, since the top of the XY-sample stage is 1 mm lower than the reference surface of the sample fixture for flat samples.
- Vacuum sample holder (Part-Nr.: A19B56)
- Clamp sample holder (Part-Nr.: A19B57)
- Air scatter screen (Part-Nr.: A24B295).
- Knife edge collimator for the XY-sample stage (Part-Nr.: A24B296)
- Dial indicator for D8 (Part-Nr.: A100B99), for aligning the sample surface to the correct height in the center of the goniometer.
- Double-laser sample alignment assembly (Part-Nr.: A100D43), for positioning the sample in the goniometer center.

⚠ CAUTION

Risk of crushing of fingers and hand

If the motor power is switched off during the alignment / manipulation or sample fixation, the Phi-Stage can slide down freely along the cradles C-Bow and may crush your fingers or hand!

▶ Be cautious when handling the Phi-Stage!

- The Compact Eulerian Cradle is mounted to the sample carrier ring in the same way as the standard or rotating / transmission sample stage. The bayonet locking permits the sample stage replacement without play and thus ensures reproducible mounting.

5.8.6.1 Mounting the Compact Eulerian Cradle

1. Set the Compact Eulerian Cradle onto the goniometer.
2. Partially screw in the three fastening screws into the mounting plate so that the cradle can still be shifted to the theta bearing.
3. Tighten the centering / play compensation screw, in order to center the cradle in the theta bearing. Tightening this screw reduces the fit play between the play compensation disk and the sample carrier ring to zero.
4. Tighten the three fastening screws.
5. Once the centering disk has been tightened to the goniometer and the stage fixed to the sample carrier ring by three fastening screws, the centering screw should be loosened and only re-tightened slightly.
6. Remove two screws of the transportation lock, which fixes the Phi-stage to the C-bow.
   ⚠ **CAUTION! Be aware that if the cradle motors are not powered and the transportation lock is removed, the Phi-Stage moves freely on the cradles C-bow.**

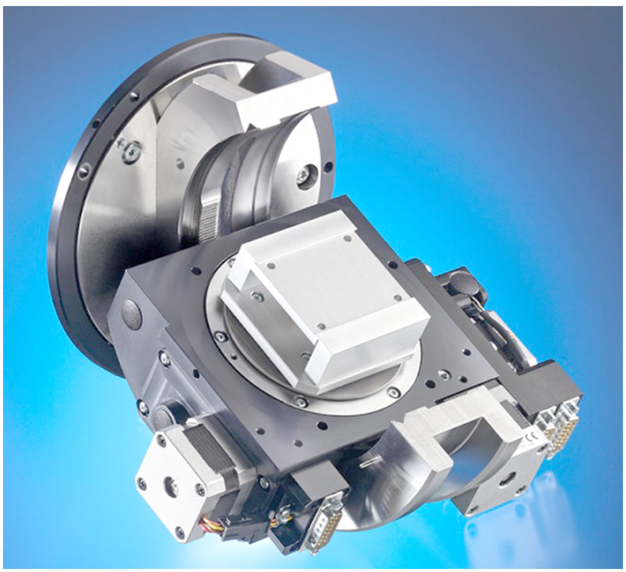


Figure 5.59: Compact Eulerian Cradle

5.8.7 Capillary Stage

Powder samples can be investigated by putting them into capillaries. These capillaries are then mounted in a goniometer head for centering on the goniometer axis. For good averaging on powder grains the capillary can be rotated. An example for a complete system setup is shown below.

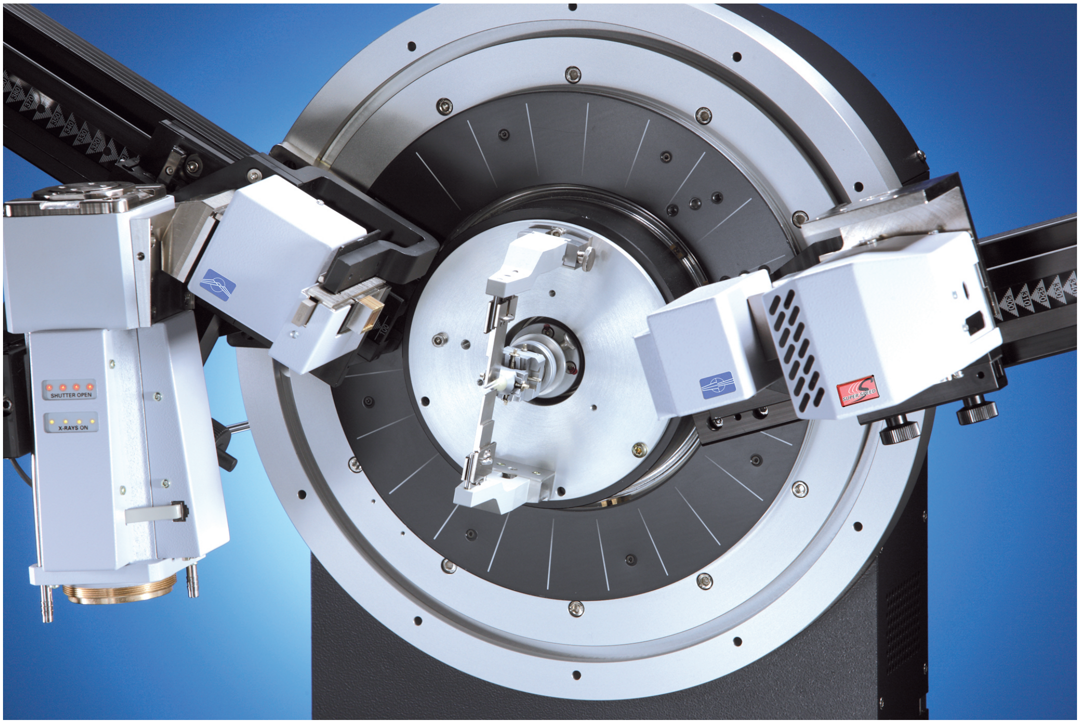


Figure 5.60: Transmission measurement with powder sample in capillary. A Johansson monochromator for transmission geometry is used on primary side in connection with a LYNXEYE detector on secondary side.

Two knife edges can be mounted on the capillary stage. The upper one reduces air scattering and is positioned slightly above the capillary. The lower one blocks the primary beam and is therefore placed behind the capillary.

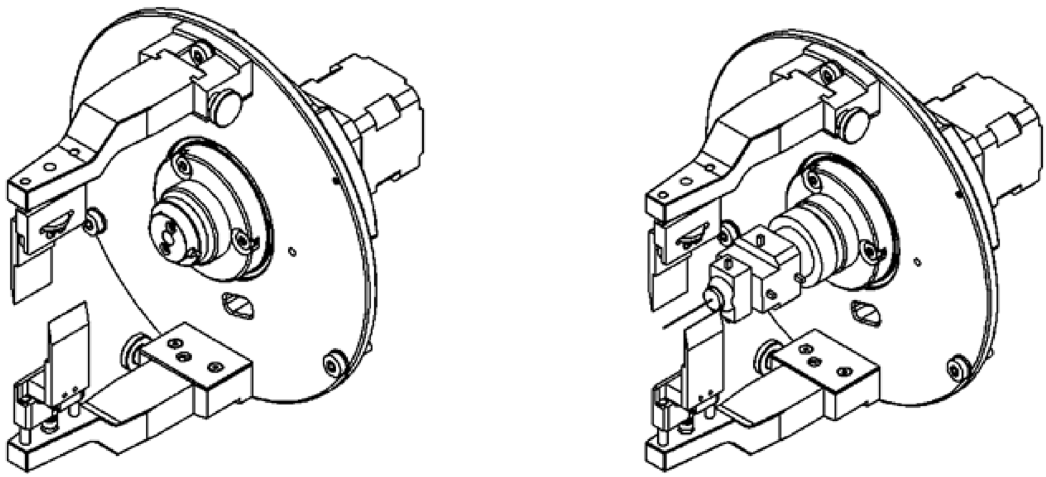


Figure 5.61: Capillary stage without goniometer head (left) and with goniometer head (right)

5.8.8 Non Ambient

A wide variety of heating and cooling devices are available to change the temperature (-263 to 2300° Centigrade), humidity (0 to 95% rel. humidity) and pressure (up to 20 bar) conditions for the sample. The heating chamber is operated by a temperature controller, which is integrated in the controlling software. Communication via RS232 port allows complex non ambient sequences for temperature and humidity if setup by using the XRD WIZARD. A wide variety of controllers is fully software integrated.

Different heating principles for powders and bulk samples are available. Fast and easy heating is achieved by direct heated strip heaters, Highest temperatures are achievable. Oven and environmental heaters allow heating of bulk samples (some mm^3) or capillaries, partly with sample rotation or in gas atmospheres. Cooling to lowest temperatures requires liquid N_2. Thermocouples and Pt100 sensors are used for temperature control. In some cases, vacuum is required for sample heating and cooling. Suitable vacuum systems are available.

Mounting and recognition are working in the same way as for other sample stages. Manual and motorized alignment bases (software controlled) are used for sample height adjustment.

A detailed description of temperature chambers can be found in the dedicated User Manuals from Bruker and in the user manuals of the suppliers.

Documentation	**Order number**
MTC- Modular Temperature Chambers for D8 ADVANCE / D8 DISCOVER	DOC-M88-EXX250
TC - Temperature Chambers for D8 ADVANCE / D8 DISCOVER	DOC-M88-EXX269
Anton Paar Temperature Chambers for D8 ADVANCE / D8 DISCOVER	DOC-M88-EXX268

Figure 5.62: Non ambient setup with Hot Humidity System and VÅNTEC-1

Figure 5.63: Non ambient setup TC-Dome chamber on a centric Eulerian cradle

5.9 Detectors

5.9.1 Scintillation Detector

A scintillation counter is used as the most basic X-ray detector. It enables X-ray measurement in the wavelength range between 0.05 and 0.3 nm (4 – 25 keV)

Detector cable – Tensile stress relief

The cable is fixed to the detector mounting base with a tensile stress relief to release the plugs from their tensile load. Be careful, the detector has a beryllium window. Do not touch this window!

Figure 5.64: Scintillation counter

WARNING

Beryllium

Beryllium is carcinogenic and causes diseases of the skin, splenic, liver and lungs.

- ▶ Never touch, inhale or incorporate fumes, dust or pieces of beryllium.
- ▶ Wear personal protective equipment (PPE).
- ▶ Do not touch any part of the instrument which contains beryllium, e.g. X-ray tube radiation outlet window, the detector front window or the heating chamber windows of some systems.
- ▶ Do not cut, machine, or handle beryllium in any way.
- ▶ It must not escape into the environment.
- ▶ Disposal of beryllium must comply with all applicable national, state, and local regulations.
- ▶ Strictly obey the applicable national, state, and local safety regulations.

5.9.2 LYNXEYE

The LYNXEYE is a 1D detector for X-ray powder diffraction, based on Bruker AXS' compound silicon strip technology. Compared to a simple point detector the LYNXEYE dramatically increases measured intensities – without sacrificing resolution and peak shape. A Diffraction Solution equipped with the LYNXEYE records a typical powder pattern in approximately 1/200th of the time required using a point detector, with identical data quality.

The active area of the detector is 14.4 mm by 16 mm (along the scattering plane respectively perpendicular). The 192 strips of the sensor act as 192 individual detectors. This technology allows operation at count rates much higher than those typically possible with gaseous detectors while maintaining all benefits. Together with the innovative front-end electronics, optimum tuning of the silicon strip sensor to the requirements of the X-ray energy from 6 keV to 15 keV is provided. The factory settings are optimized for Cu-Kα.

- LYNXEYE can be used in scanning and fixed 1D mode or in the integrating 0D mode where a definable number of the 192 channels are added prior to display and saving of data.

- LYNXEYE is mounted on the universal detector mount (cf. section on universal detector mount). LYNXEYE can be mounted in 2 orientations: standard for 1Dimensional Bragg-Brentano measurements and rotated by 90° for out of plane measurements or for increasing the dynamical range in the integrated 0D mode.

A detailed description of LYNXEYE can be found in the dedicated manual DOC-M88-EXX095.

Figure 5.65: LYNXEYE detector

5.9.3 LYNXEYE XE

A second generation LYNXEYE detector offers improved energy resolution, enabling better fluorescence and K-beta filtering. Operational X-ray energy range has been extended in comparison to the first generation detector. Measurements with the LYNXEYE XE can be performed with radiation from 4 keV to 25 keV.

With the 2013 release of DIFFRAC.MEASUREMENT CENTER, the 1D LYNXEYE XE detector is fully supported (0- and 1- dimensional measurement mode). The 0D mode means coupling of several neighboring channels of the detector to one counting channel. All events collected by the selected channels are counted as one single rate – equal to how a scintillation counter works. The LYNXEYE XE in 0D mode completely replaces the scintillation counter for many applications, even the basic alignment of the diffractometer.

Depending on the operation mode, one strip can detect up to ~20,000 cps (high energy resolution) or ~500,000 cps (high count rate), resulting in ~100,000,000 cps in 0D mode and 90° detector orientation, count rates are not dead time corrected.

A detailed description of LYNXEYE XE can be found in the dedicated manual DOC-M88-EXX240.

Figure 5.66: LYNXEYE XE detector

5.9.4 LYNXEYE XE-T

The third member of the LYNXEYE family, the LYNXEYE XE-T offers <380 eV FWHM energy resolution for Cu-radiation at 298 K, allowing for practically complete K-beta removal without the need for a Ni filter. Operational X-ray energy range has been further extended. Measurements witht the LYNXEYE XE -T can be performed with radiation from 4 keV to 35 keV.

With the 2015 release of DIFFRAC.MEASUREMENT CENTER, the 1D LYNXEYE XE-T detector is fully supported (0- and 1- dimensional measurement mode). The 0D mode means coupling of several neighboring channels of the detector to one counting channel. All events collected by the selected channels are counted as one single rate – equal to how a scintillation counter works. The LYNXEYE XE in 0D mode completely replaces the scintillation counter for many applications, even the basic alignment of the diffractometer.

Depending on the operation mode, one strip can detect up to ~20,000 cps (high energy resolution) or ~1000,000 cps (high count rate), resulting in ~4,000,000 cps resp. 100,000,000 cps in 0D mode and 90° detector orientation. Count rates are not dead time corrected.

A detailed description of LYNXEYE XE-T can be found in the dedicated manual DOC-M88-EXX239.

5.9.5 VÅNTEC-1

VÅNTEC-1 is a 1D detector for fast and simultaneous detection for X-ray diffraction in a large 2theta range. The detector can be used as well in fixed 2theta position as in scanning mode for 2theta. In scanning mode the results are quite comparable to measurements done with a 0-dimensional detector (e.g. Scintiallation counter), but typically the measurement times for a standard powder diffraction pattern are reduced up to a factor 100 with similar angular resolution. Moreover, VÅNTEC-1 enables in situ experiments with measurement times down to 100 ms. These experiments can cover 2theta ranges up to 10°. Using this mode allows X-ray movies from kinetic processes to be recorded.

By using the patented mikrogap technology VÅNTEC-1 provides all known advantages of gas based detectors like high signal amplification with low noise, i.e. very good peak to background ratio, high sensitivity for a large X-ray energy range, and relatively good energy resolution.

VÅNTEC-1 can be used with all typical standard wavelengths common in X-ray diffraction. These are the characteristic Kα lines of Cr, Co, Cu, and Mo. In factory VÅNTEC-1 is optimized for Cu Kα. The user can optimize the electronic discrimination of unwanted X-rays. This reduces background and to some extent fluorescence radiation.

The active length of the detector is 50 mm within the 2theta plane. The active height is 16 mm. Measurement diameter, sample, wavelength, and optics define angular resolution and simultaneously detected 2theta range. The D8 enables optimization to the diffraction needs by using the flexibility of the primary and secondary tracks of the goniometer. In general focussing Bragg-Brentano geometry and larger measurement circle diameter provide best angular resolution.

Specifications:

- Active window: 50 mm x 16 mm (scattering plane x othogonal to scattering plane)
- Maximum simultaneous 2theta range: approx. 10° at 500 mm measurement circle diameter
- Usable wavelength range: Cr-Kα…Mo-Kα, standard optimization for Cu-Kα.
- Energy resolution: <25 %
- Spatial resolution: < 65 µm, >1600 single channels
- Gas filling: Xe-CO_2, no regassing needed

For details refer to dedicated *VÅNTEC-1 User Manual* (DOC-M88-EXX072).

Figure 5.67: VÅNTEC-1 detector

5.9.6 VÅNTEC-500

The VÅNTEC-500 area detector is based on the patented MikroGap™ technology, which offers all the benefits of gas detectors, such as high signal amplification resulting in high peak-to-background ratio, allowing operation at count rates much higher than those typically possible with standard gas detectors. The active area of the detector is approximately 154 cm^2. The single frame angular range, as well as the achievable angular resolution, is influenced by the sample properties, the applied X-ray wavelength and the sample-detector distance.

Detailed information about the VÅNTEC-500 detector can be found in the dedicated user manual, DOC-M88-EXX177.

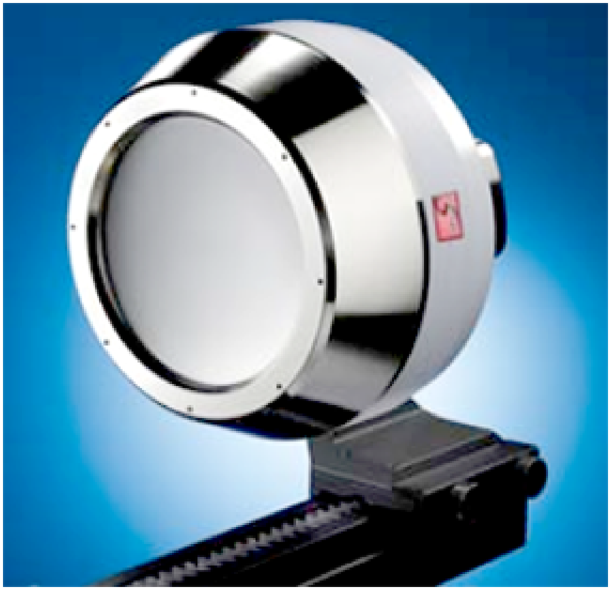


Figure 5.68: VÅNTEC-500 detector unit

5.9.7 PILATUS3 R 100K A

The DECTRIS PILATUS3 R 100K A hybrid pixel detector offers 20 bit dynamic range with single photon sensitivity. Fully integrated into the D8 platform, the PILATUS3 is an ideal solution for measurements of weakly and/or strongly scattering samples, supporting Co, Cu, Mo and Ag radiation. The patented HPC™ technology is based on the direct conversion of X-ray photons to electrons via the photoelectric effect.

Please consult the dedicated *PILATUS3 R 100K A User Manual* (DOC-M88-EXX280) for a detailed description, installation and usage instructions.

5.9.8 EIGER2 R 500K

The EIGER2 R 500K (hereafter abbreviated as EIGER2) is a multi-mode (0D/1D/2D) detector designed specifically for laboratory instruments. It is based on the next generation of Hybrid Photon Counting (HPC) technology developed by Dectris Ltd.

The active area of the sensor is about 77 x 39 mm^2 (approximately 30 cm^2), covered by 529 420 pixels, 75 x 75 μm^2 each. The simultaneously-recorded angular range, as well as the achievable angular resolution, are influenced by the sample properties, the applied X-ray wavelength, and the sample to detector distance.

The detector is based on the patented Hybrid Photon Counting (HPC™) technology. HPC silicon X-ray detector technology is employing the direct conversion of photons to electrons via the photoelectric effect. Due to the direct nature of this conversion, the position of the photon interaction is accurately measured, which results in an almost ideal point spread function. The fast conversion also allows for highest count rates and dynamic range.

The factory settings are optimized for Cu Kα radiation but can be adapted to all standard X-ray wavelengths (Cr, Co, Cu, Mo, Ag). The EIGER2 consists of three parts: the detector unit, the detector controller and the secondary instrument control system (ICS).

A detailed description of EIGER2 R 500K can be found in the dedicated *User Manual* (DOC-M88-EXX293).

Figure 5.69: EIGER2 R 500K

6 Operating the Instruments

6.1 Instrument Status

6.1.1 Checking and Optimizing the Primary Beam Intensity

The mirrors and monochromators are factory-adjusted to deliver adequate intensity and resolution. A decrease in intensity due to tube aging is normal and expected. If additional intensity decrease is observed, it could be time to check the focus translation. The focus translation moves the tube focus relative to the optics, so that the intensity can be tuned without changing the beam alignment with respect to the goniometer.

To check the focus translation, refer to the figure below and perform the following steps:

1. Loosen the focus translation locking screw.
2. Translate the tube focus using the screw.
3. Monitor the intensity using the ratemeter of DIFFRAC.SUITE.
4. When the optimal intensity is reached, tighten the translation locking screw.

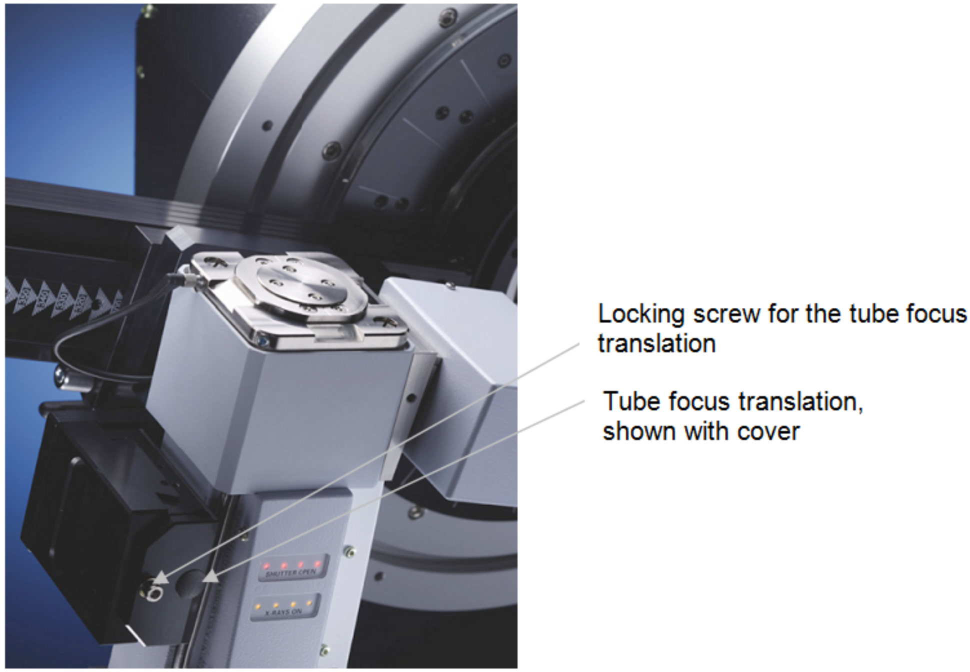


Figure 6.1: Locations of the screws

6.2 Tube

6.2.1 Conditioning

The operation software will automatically read out the recognition chip of the tube when switching on system or generator. Conditioning of the tube will be started depending on operation history of the tube.

The conditioning of the tube might be interrupted by the user, if urgent measurements must be done. However, this will have negative impact on the life time of the tube. The tube chip will memorize that conditioning has not been done.

6.2.2 Changing Tubes

6.2.3 Switching Between Point and Line Focus

The following figure shows the tube supporting plate for the “TWIST-TUBE” housing. Screws A are the fixing screws for the tube twist. The engraved line marking on the plate shows the orientation of the filament focus.

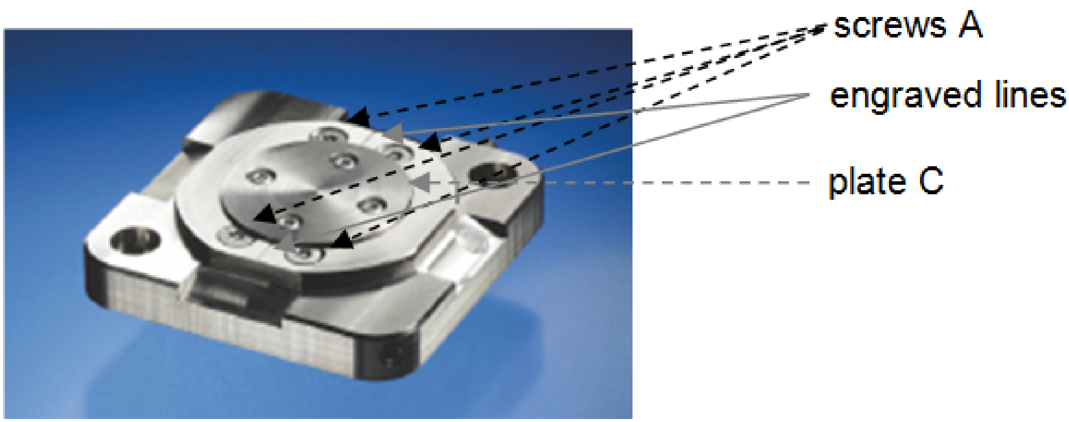


Figure 6.2: TWIST-TUBE supporting plate

The next figure shows the plate in the point and line focus positions.

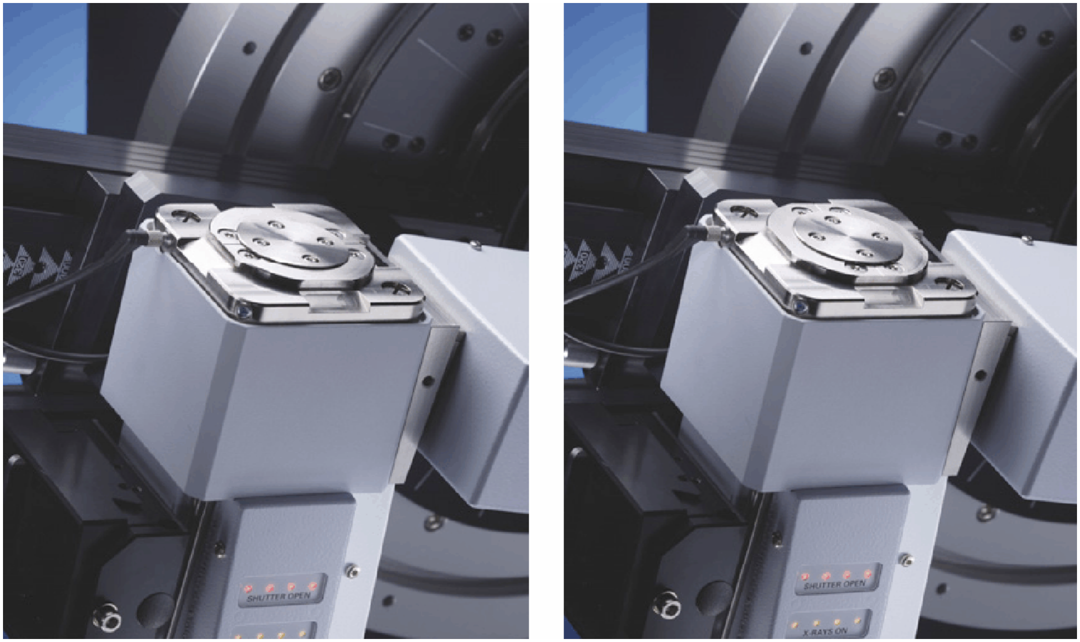


Figure 6.3: Supporting plates as shown on the tube housing, in the point focus (left) and line focus (right) configurations

The X-ray tube of D8 diffractometers is operated with electrical voltages up to 50 kV DC (in special applications even up to 55 kV).

For electrical safety reasons, before switching the X-ray tube between line and point focus, the electrical high voltage must be turned off completely by all means using the so-called “Generator screen-key” at the front panel of the instrument. This must be done before touching and loosening any of the screws labelled as “A” within this section!

I

The high voltage applied to the X-ray tube is turned off if the display of the screen-key indicates the **Switch-on** symbol.

If the screen-key displays any of the symbols **Heating on**, **X-rays on, generator ramping up/down** or **X-rays on, generator ready** you must press once the screen-key switch and wait until it is showing the **Switch-on** symbol.

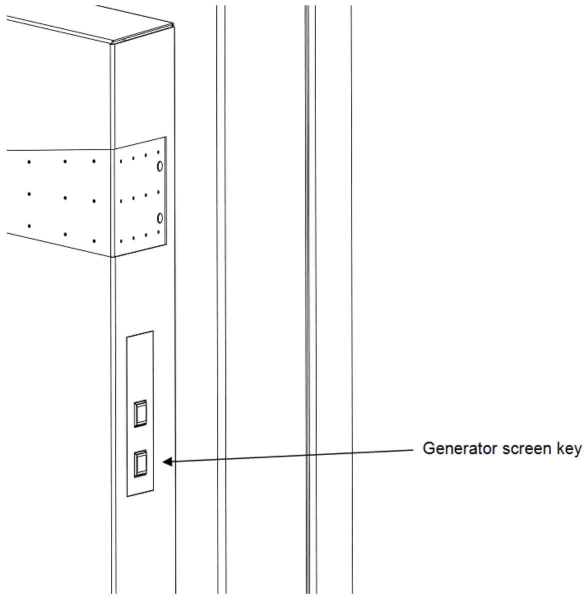


Figure 6.4: Location of the Generator screen-key (Turn on/off X-rays)

To know the meaning of the illumination of the generator screen key please refer to chapter *Starting the Instrument [▸ 13]*.

⚠ WARNING

High voltages, up to several tens of thousands Volts DC in:

high-voltage generator | X-ray tube | radiation detectors | high-voltage cables

High risk of lethal electrical shock when components are damaged!

- Switch off the instrument using the Generator screen-key.
- Disconnect the system **completely** from the mains supply.

To perform a switch between point and line focus:

Attention

The screws should be loosened only far enough to allow the rotation (approximately one turn). Too much loosening could cause the screw heads to get jammed in plate C.

1. Turn off the high voltage (X-ray generator HV).
2. Loosen the four screws A shown in figure 7.2.
3. Using the special tool provided, perform the rotation as shown below.
4. Tighten screws A, to move the filament back into the proper focus position.
5. Turn on the generator high voltage.

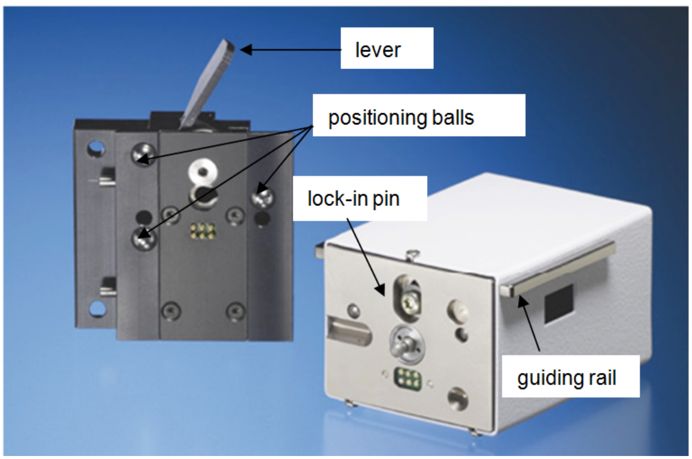
Figure 6.5: Performing the filament rotation

6.3 Mounting Optics

6.3.1 Optics Mount (SNAP-LOCK)

The optics mount is shown below. The positioning balls and lock-in pin are used for centering and fixing the optics on the optics mount. Moving the lever down secures the optics into position.

On the back of the optics are indents for centering the optics over the positioning balls. Note that one of the indents is elongated and the other two are round. The conical round indent should be placed exactly on the center of its respective positioning ball. Therefore, when mounting an optics, guide the optics onto the optics mount by lightly pressing on the housing at the point on the housing across from this indent. This procedure is described in detail in the next section.



Detailed view of an optics mount and optic

Mounting and Changing Optics

The tool-free way of changing optics is displayed in the pictures below. To achieve the intended reproducibility some simple rules must be followed. These rules are also described in the following.

1. Open lever

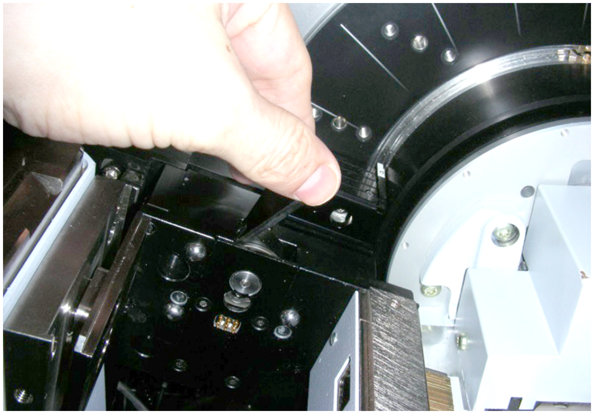


Figure 6.6: Lever

2. Put in the optics module. The guiding rails (shown below) at the side of the optics module slot, also called optical bench position, will guide you. You have to lift the optics module a little bit before the three pins of the optics module catch the three holes of the optics module slot. Only when the three pins have caught the hole, it is possible to push down the lever to fix the optics.

Figure 6.7: Guiding rail (a)

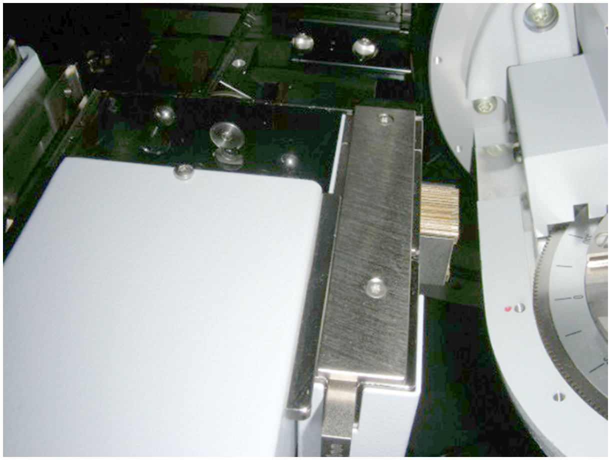


Figure 6.8: Guiding rails (b)

3. Please press with your thumb against the lower left front corner of the optics module. This corner is usually marked with a blue sticker. While pressing against this spot push down the lever to fasten the optics module.

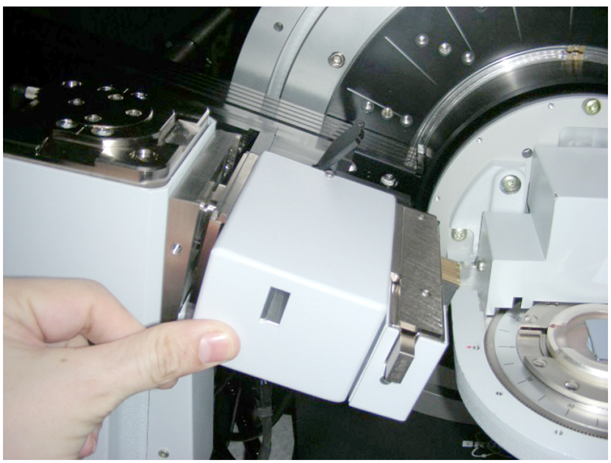


Figure 6.9: Press at this spot if you mount any optics

The pins and the rails are only supports for the optics module mounting. The positioning accuracy of the optics module is given by the three positioning balls on the surface of the optics module slot and counter surfaces of the optics module.

Please keep the positioning balls and the counter surfaces of the optics module always clean. You can clean them with a soft tissue, do not damage or scratch any of these surfaces or positioning balls.

6.3.2 Optics mounting status displayed in DAVINCI plug-in

The DAVINVI plug-in displays if an optics is mounted and which optics is mounted. Therefore, the optics must be correctly configured and component recognition must be activated (cf. *DIFFRAC.SUITE User Manual* DOC-M88-EXX191). The change in DAVINCI is displayed below:

- Left: No optics is mounted on the primary beam path
- Right: An optics called TestGM is mounted) on the primary side beam path

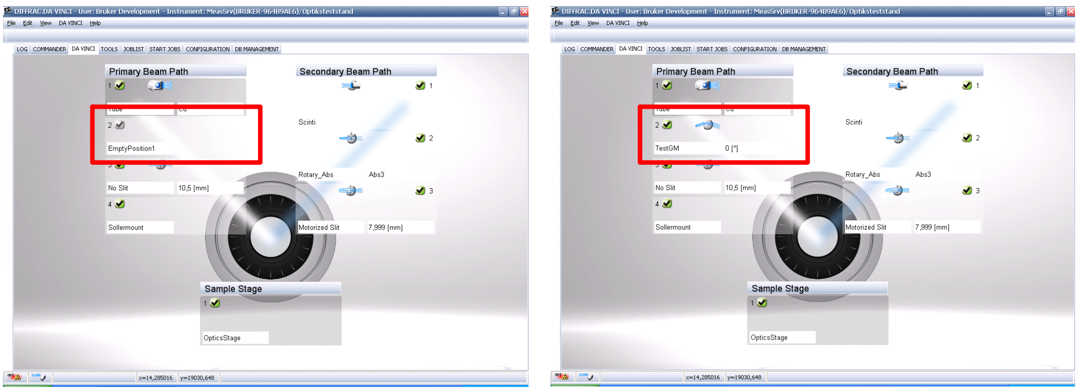


Figure 6.10: DAVINCI plug-in

6.4 Mounting Sample Stages

Most of the stages are equipped with a bayonet and a recognition chip.

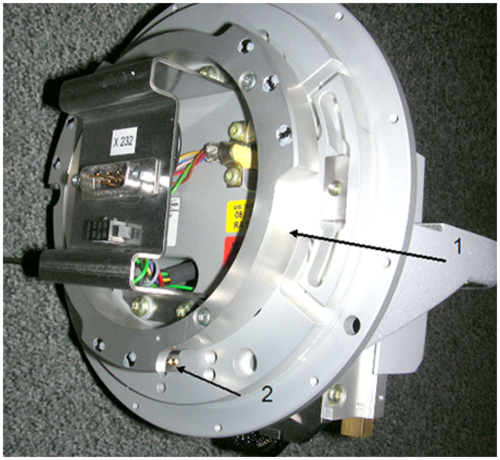


Figure 6.11: Stage with bayonet (1) and recognition chip (2)

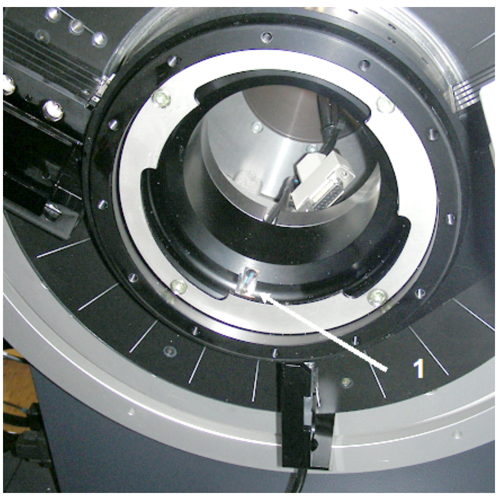


Figure 6.12: Goniometer with bayonet and recognition readout (1)

Do not mount the stage turned by 120° or 240°. In these cases the recognition interface in the goniometer will be damaged.

1. Put the stage with bayonet into the goniometer as shown in the picture below. The red mounting marker at the stage must match the marker position on the goniometer.

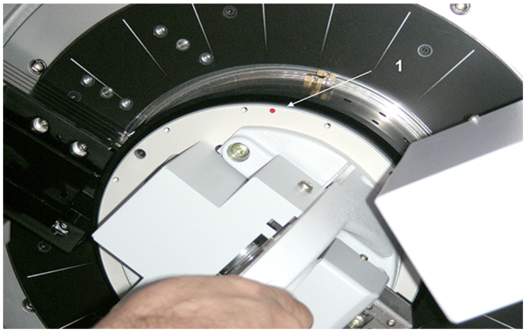


Figure 6.13: Stage with red mounting marker (1)

2. Then turn the stage clockwise to fix it with the bayonet mechanism.
3. Use the centering screw to align the stage position and then firmly fix the stage with the three provided screws. The marker on the stage must match the marker position on the goniometer.

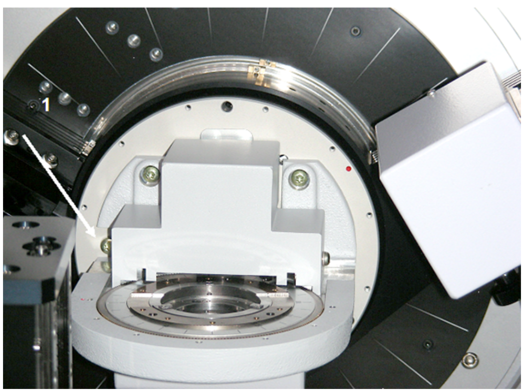


Figure 6.14: Centering screw (1) of the stage

Every stage with a bayonet is subject to the same mounting restrictions as described above. Please refer to the related manual how to adjust and securely fix the stage.

6.4.1 Stage status displayed in DAVINCI plug-in

The mount status of a stage (if configured correctly) is displayed in the DAVINCI plug-in. A situation with and without mounted stage is shown below. For more detail refer to the *DIFFRAC.SUITE User Manual* (DOC-M88-EXX191).

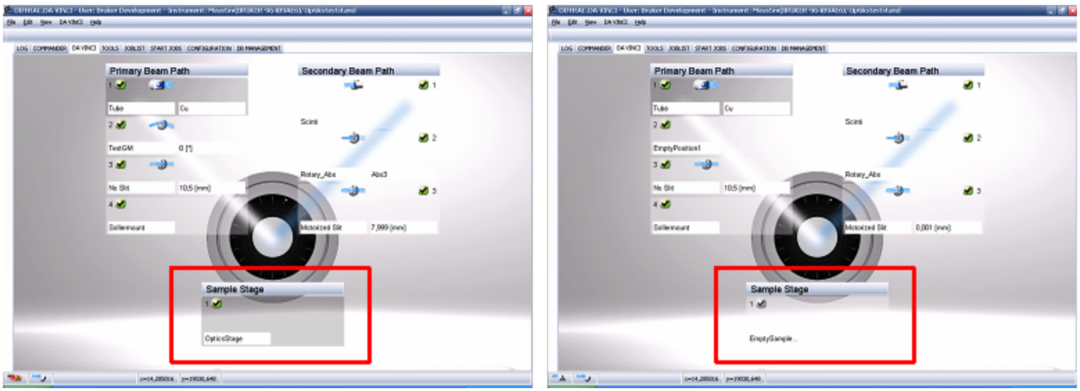


Figure 6.15: DAVINCI plug-in (Left: A stage is mounted; Right: No stage is mounted).

6.5 Mounting Detectors

The most common 0D and 1D detectors can be mounted without the help of tools on the universal detector mount (UDM). All detectors mounted on a universal detector mount are recognized by the system and can be used in combination with most standard secondary optics.

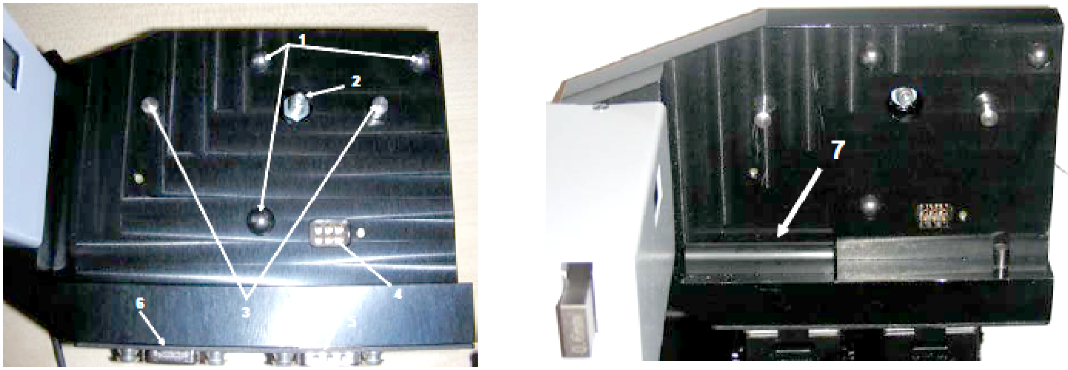


Figure 6.16: Universal Detector Mount (UDM)

1	Three positioning balls mainly used for LYNXEYE mounting	5	Socket for cable between UDM and Goniometer
2	Fixing screw	6	Socket for cable UDM and secondary optical bench
3	Guiding pins	7	Positioning rail UDM
4	Chip-Readout interface		

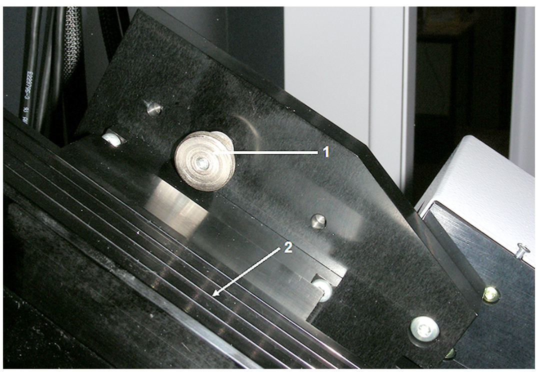


Figure 6.17: Backside of the universal detector mount

1	Fixing screw	2	Track

The detectors which can be mounted this way are:

- Scintillation counter
- SOL-XE
- LYNXEYE

The VÅNTEC-1 and all 2D detectors are not mounted on this UDM. For mounting the VÅNTEC-1 and 2D detectors the detector mount must be exchanged together with the detector.

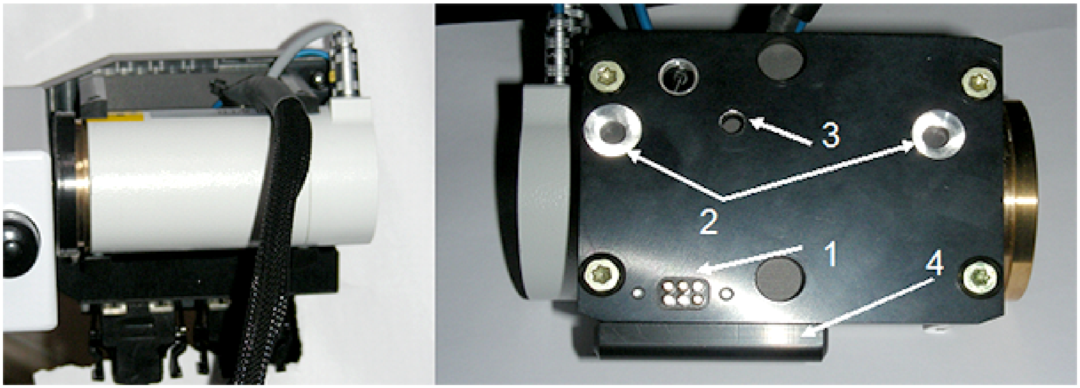


Figure 6.18: Scintillation detector is fixed on a detector holder which is mounted on the UDM; backside of the detector holder

1	Recognition Chip	3	Thread for fixing screw
2	Holes for guiding pins	4	Positioning rail detector holder

Figure 6.19: Left: LYNYEYE detector holder; Mittle/Right: LYNXEYE detector holder mounted on the universal detector mount, (right: 0° position, left: 90° position)

1	Reference surfaces	4	Recognition Chip
2	Thread for fixing screw	5	Positioning rail
3	Hole for guiding pin		

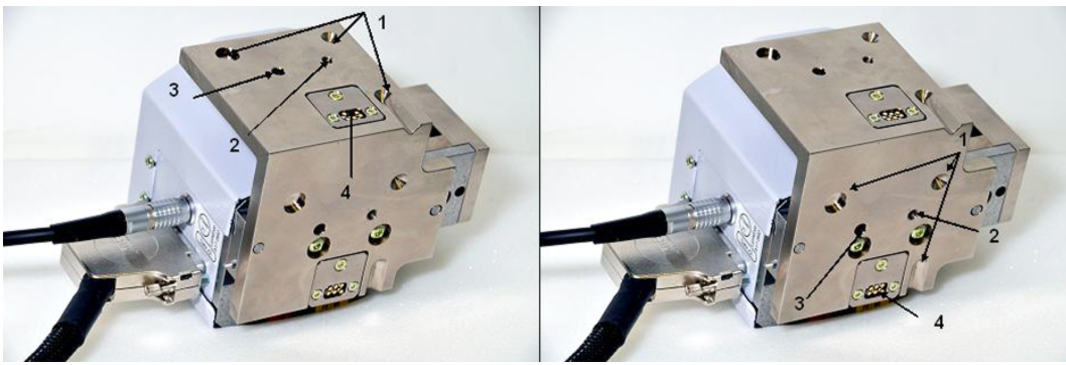


Figure 6.20: LYNXEYE with detector holder

1	Reference surfaces	3	Hole for guiding pin
2	Thread for fixing screw	4	Recognition Chip

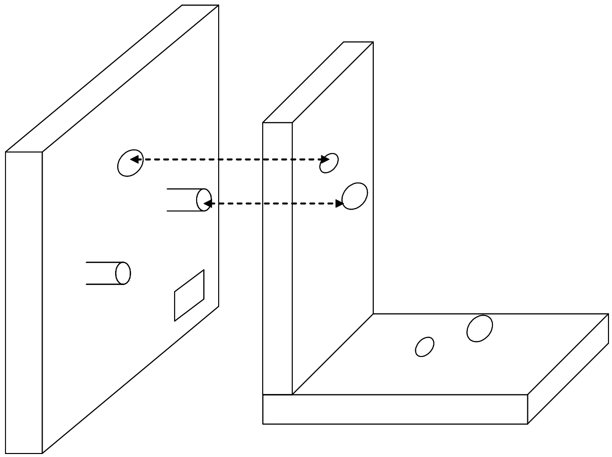


Figure 6.21: Mounting of the detector holder on the universal detector mount (LYNXEYE).

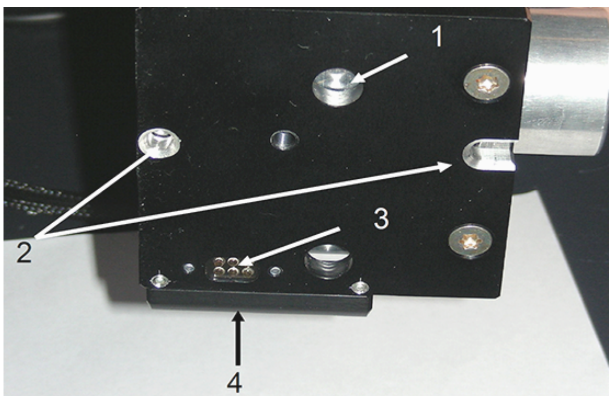


Figure 6.22: SOL-XE with detector holder

1	Thread for fixing screw	3	Recognition Chip
2	Holes for guiding pins	4	Positioning rail detector holder

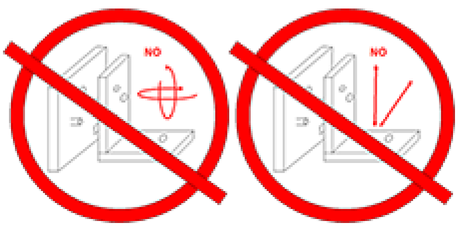
1. When mounting the detector holder keep the detector holder parallel to the surface of the universal detector mount.

2. Place the positioning rails on the detector holder into the corresponding position on the universal detector mount (UDM).
3. The guiding pins of the universal detector mount must fit into the corresponding holes in the detector holder as shown in schematic drawing above.
4. Also the threads for the fixing screw must be at the right position.
5. If the detector holder is properly placed fix the holder with the fixing screw at the backside of the universal detector mount.

NOTICE

Chip interface can be damaged

Do not rotate or translate the detector holder relative to the surface of the universal detector mount while the detector is nearly in place. If you turn or translate the detector holder the chip interface can be damaged



The LYNXEYE detector holder only uses the rear guiding pin.

6.5.1 Detector status display in DAVINCI plugin

The mounting status of all detectors (if configured correctly) is displayed in the DAVINCI plug-in. The relevant change in The DAVINCI display is marked with a square in. For more detail refer to *DIFFRAC.SUITE User Manual*.

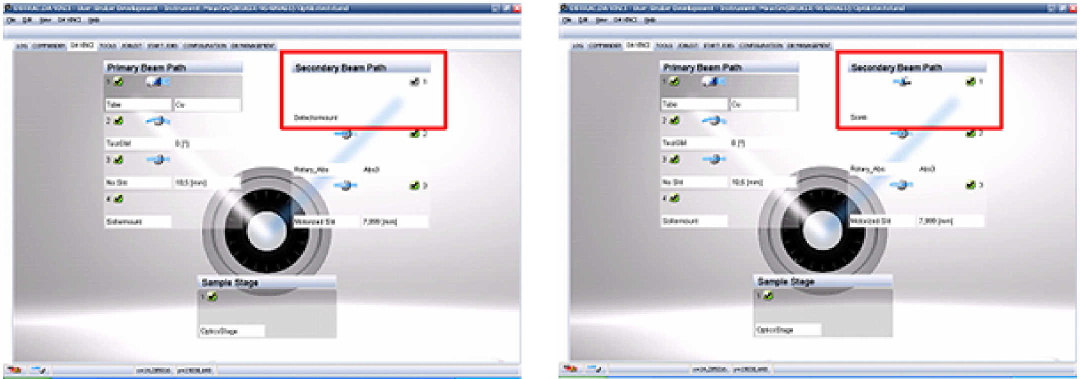


Figure 6.23: DAVINCI plug-in (Left: No detector is mounted; Right: A scintillation counter is mounted)

6.6 Typical Scans

6.6.1 Axes vs. Motorized Drives

The Theta (θ) axis, which is used to change the angle between the X-ray source arm and the surface of the sample, involves two different movements depending on the system used. In θ/θ systems, the angle is changed by rotation of the X-ray source arm around the goniometer axis, while in θ/2θ systems it is changed by rotation of the sample around this axis while the X-ray source arm is stationary.

Motorized drives are the devices by means of which the movements are realized in practice. They can take various forms depending on the design of the diffractometer, but they usually involve the use of one or several electric motors.

Axis	Motorized Drive	Movement	Reference point
Theta (θ) (Omega)	θ/θ: Source arm of goniometer θ/2θ: Sample	Around horizontal axis of goniometer	θ/θ: Surface of sample (horizontal line through centre of goniometer) θ/2θ: direction of beam path

Axis	Motorized Drive	Movement	Reference point
TwoTheta (2θ)	θ/θ: Source arm and/or Detector arm θ/2θ: Detector arm	Around axis of goniometer	direction of primary Beam
Detector	θ/θ: Detector arm of goniometer	Around axis of goniometer	Surface of sample (sample horizon)

6.6.1.1 Theta (θ) or Omega (ω) Axis

In some applications, for example high resolution XRD and reflectometry, the theta (θ) axis is called omega (ω). This is, because in these applications the angle formed by the X-ray primary beam and the surface of the sample is not always equal to half the diffraction angle (2θ), as in powder diffraction. Applications other than powder diffraction therefore require the use of another notation for this axis. In these cases one speaks of the omega (ω) axis.

6.6.1.2 TwoTheta and Detector Axis

The TwoTheta (2 θ) axis is used to position the detector with respect to the primary beam path and is available independent of the goniometer geometry.

In θ/θ systems additionally a detector axis is available. Its movement is identical to the TwoTheta axis except that the zero position of this axis is at the sample horizon. Thus, the Detector axis is independent from the primary beam direction whereas TwoTheta changes if the primary beam direction is changed (see also *Deflection Angle [▸ 165]*).

6.6.1.3 Offset

All axes positions can get temporary offsets for adjusting angles according to optimum values for an actual measurement. In case the position of an axis is not optimal for an actual measurement, then the **Offset and Reference Postion Determination** tool in the COMMANDER plug-in can be used for redefining the position temporarily.

For example, the sample surface might not be parallel to the direct beam at theta=0° and the user wants to change this during measurements on this sample. Move the TwoTheta axis to zero and perform a rocking scan of the sample. Using the **Offset Determination** on the peak maximum (define an offset to have the peak maximum at 0°) will solve this problem.

Offset determination will not be saved permanently. The offset will be reset to zero after next restart of the instrument.

6.6.1.4 Deflection Angle

If an axis position shall be redefined permanently for a specific setup, then deflection angles can be defined for each optics responsible. Deflection angles can be defined with the CONFIGURATION plug-in. Changing the deflection angle will only affect setups where that specific optics is used.

6.6.1.5 Reference Position

The basic reference positions for all drives are defined in factory. Resetting is only necessary when a major adjustment of the system is done. Changing of reference positions will affect all optics setups and not only the optics which are actually installed on the system. Additionally a change of a reference position will have a permanent effect after saving to the database of the DIFFRAC.SUITE and/or activating that configuration on the instrument. Therefore, reference positions should only be changed for serious reasons.

6.6.2 General procedure for optimizing reference positions and deflection angles

The D8 ADVANCE / D8 DISCOVER allows easy change between different optics setups. This is either done by push-button with TWIN/TWIN or without readjustment by the SNAP-LOCK mechanism. DAVINCI.MODE monitors the instrument and takes care that the correct reference positions are used for the actual setup.

This requires a correct and consistent setting of all setups during factory test, installation on site, and when adjusting the instrument during maintenance. The general procedure is as described below: Define Basic Setup, then define additional setups.

6.6.3 Beam Path and Deflection Angle Concept

The SNAP-LOCK mechanicsm and the TWIN optics allows a quick and alignment free change between Bragg-Brentano and Goebel Mirror setups. To assure that such a change is possible, all optics must be adjusted and configured following strictly some simple rules. Be aware that all setups of your instrument were thoroughly aligned in factory and that this alignment was verified during first installation on customer side.

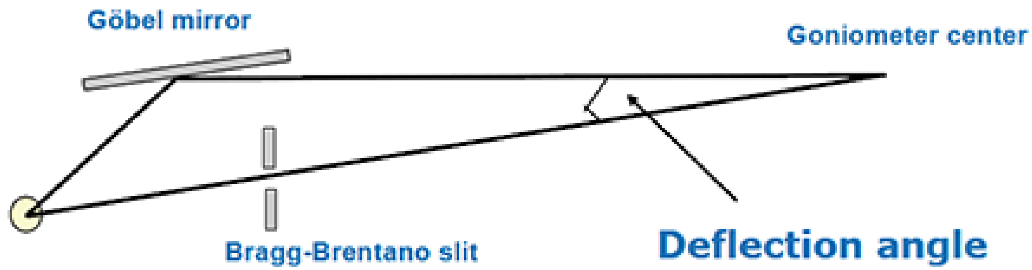


Figure 6.24: Beam path and deflection agle

6.6.4 Basic Setup

The basic reference position of the D8 is set in Bragg-Brentano geometry at factory. The reference positions for Theta and TwoTheta are determined with the Bruker AXS glass slit alignment procedure. The Commander plug-in provides an interactive tool for finding peak maxima and using the positions for saving optimized reference values to the database.

Most exact results are achieved by following the procedure described in the Bruker AXS verification booklet. Final reference positions are saved in the CONFIGURATION plug-in of the DIFFRAC.SUITE.

The original factory setting is defined in the configuration **Instrument Verification Factory**.

6.6.5 Additional Setups

Additional setups like different parallel beam setups with Göbel mirrors or the parallel beam path of the TWIN optics have beam directions different from the basic Bragg-Brentano setup. Accordingly, this information must be added to the database. The system takes this into account by the deflection angles of the optics. The value of the deflection angle is identical for theta-theta and theta-2theta systems. Deflection angles can be redefined in the Configuration plug-in of the DIFFRAC.SUITE, e.g.

- **Göbel mirror:** Goniometer/PrimaryTrack/Göbel Mirror
- **TWIN optics:** Goniometer/PrimaryTrack/TWIN OPTICS/Göbel Mirror.
- **TRIO optics:** Goniometer/PrimaryTrack/TRIO OPTICS/Göbel Mirror.

The position of any primary and secondary optics can be fine tuned by refining the deflection angle of the specific optic. The default value (usually in the Bragg-Brentano setup) is always 0°.

For becoming permanent, the deflection angle must be saved to data base of the DIFFRAC.SUITE by the **Save configuration to database** function of the CONFIGURATION plug-in. The software will ask for a name for this configuration. When installing the system first time, the default name is **Instrument Verification Customer**. However, any other convenient name may be chosen by the user. Save and activate this configuration.

6.6.6 Focus Position Alignment

The beam path concept requires a fixed position of the X-ray tube focus. The X-ray tube focus position must be the same for all optics mounted on the first position of the optical bench if these optics require the same take-off angle adapter plate and tube type.

The procedure of the focus alignment depends on the type of optics which are delivered with the system. Two main groups of systems can be classified in this case. These are diffractometers with and without monochromatic optic. In this content monochromatic optics are: Göbel mirrors (incl. the Göbel mirror in the primary TWIN optics, TRIO optics and the focusing Göbel mirror), monochromators and Johansson monochromators. For standard Bragg Brentano setups this alignment is usually not necessary.

An alignment of the X-ray tube focus position may be necessary under the following conditions:

- First set up of the diffractometer at customer side
- Change of the adapter plate
- Change of the x-ray tube
- Change from point focus to line focus

6.6.6.1 Alignment of the Focus Position

Mount adapter plate, tube housing and tube necessary for the monochromatic optics which will be used for adjustment.

Monochromatic optics are Göbel mirrors (incl. Göbel mirror in the primary TWIN and TRIO optics), Montel mirrors and primary side monochromators (eg. Johansson monochromator, TRIO chanel cut monochromator).

For the first set up of the diffractometer at customer side please remember:

Before shipping, each diffractometer system was carefully put into operation and was completely aligned. Therefore, it is not necessary to change the settings of the alignment screws on any optics if the control values obtained during the alignment steps are within the tolerances.

If an alignment of the focus position is necessary follow the procedure described below.

1. Move Theta and 2Theta to 0°.
2. Make sure that the beam is not blocked by the sample holder.
3. Use suitable absorbers and Kß filter.
4. The measured countrate should be in the range of 103 cps to 105 cps (counts per second).
5. Use no detector slit and no anti scatter slit.
6. Open fixing screw of the focus position.
7. Use suitable detector settings (for Cu radiation usually 40 kV / 40 mA).
8. Start the ratemeter in DIFFRAC.COMMANDER and open the shutter.
9. Adjust focus position in steps of ¼ turns to achieve maximum intensity recorded with the ratemeter.
10. Check focus position with all other monochromatic optics.
11. One focus position should fit for all monochromatic optics which are used with this tube and focus type (point/line).

6.6.7 Glass slit alignment

In order to ensure reliable operation of the diffractometer, it is recommended to check from time to time the zero position of the x-ray beam on primary and secondary side. This is done with the here described glass slit alignment procedure. Also an exchange of certain components (e.g. stages) requires to check the angular position of primary and secondary beam relative to the sample position.

6.6.7.1 Zero Point Definition of the θ Scale

Measurement

1. Execute a rocking scan measurement or a tube scan $-1° < \theta < +1°$, step size 0.01° or smaller, 0.1 sec/step, using DIFFRAC.COMMANDER.
2. Determine the angular position θ_{Max} of the intensity maximum.

Consequence

The basic alignment of the θ scale is o.k. if the angle θ_{Max} lies within the range $-1° < \theta < +1°$; if not, check sample stage and its installation.

Result

The exact zero angle of the beam passing through the center of the goniometer on the uncorrected θ scale is θMax. This alignment step is done if $|\theta - \theta_{Max}| < 0.004°$. Depending on the installed optics setup greater deviations of the zero beam position will either affect the reference angle of the theta scale or the deflection angle of the installed optics as described above. Please also refer to the *Instrument Verification Booklet* for example values for the different setups.

6.6.7.2 Zero Point Definition of the 2θ Scale

Measurement

1. Execute a 2Theta scan measurement –1°< 2θ < +1°, step size 0.01° or smaller, 0.1 sec/step, using DIFFRAC.COMMANDER.
2. Determine the angular position $2\theta_{Max}$ of the intensity maximum.

Consequences

The basic alignment of the 2θ scale is o.k. if the angle $2\theta_{Max}$ lies within the range –1.0° < 2θ < +1.0°, if not, check sample stage and its installation.

Both above mentioned alignment steps must be repeated if the sample stage is changed.

Result

The exact zero angle of the beam passing the centre of the goniometer on the uncorrected 2θ scale is 2θMax. This alignment step is done if $|2\theta_{Max} - 2\theta_{Ref}| < 0.004°$. Depending on the installed optics setup greater deviations of the zero beam position will either affect the reference angle of the TwoTheta scale or the deflection angle of the installed optics as described above. Please also refer to the *Instrument Verification Booklet* for example values for the different setups.

6.7 Standard and TWIN (TRIO)/TWIN Configurations

The configurations described in this section serve as examples of setups for measurements that can be performed with the D8 ADVANCE. The following two basic configurations are possible with the D8 ADVANCE:

- **Standard configuration**: SNAP-LOCK change switching between different beam paths
- **TWIN (TRIO)/TWIN configuration**: fully automated, software-controlled switching between different beam paths. Note that the primary TWIN (TRIO) optics requires a 560 mm measurement diameter.

NOTICE

Attention

Each of the two configurations, standard and TWIN (TRIO)/TWIN, is designed for user friendliness within the setup. Switching between these two basic configurations, or mixing parts of one configuration with another, will most likely lead to configuration misalignment that requires manual realignment.

When planning a configuration, check the space requirements of the components involved. For example, a small measurement radius could pose problems with detector optics or monochromators

6.7.1 Standard Configuration

Characteristics:

- Measurement diameter 500 mm, sometimes 560 mm
- Switching application with help of SNAP-LOCK, Sample Stage Bayonet, and Universal Detector Mount.
- Dedicated Optics: all SNAP-LOCK optics modules (e.g. Plug-In Slits, Göbel mirrors, POLYCAP, etc.)
- Sample stages: Standard, Rotary, FLIP-STICK, AUTO-CHANGER, Capillary, Compact XYZ stage, Non Ambient

Setups:

- Bragg-Brentano, grazing incidence diffraction, reflectometry, stress, transmission, texture and others

6.7.1.1 Dedicated Bragg-Brentano Setup (Standard)

Characteristics:

- Fixed measurement circle, usually with 500 mm diameter
- Beam is slit-collimated to achieve focusing on the detector. Motorized as well as plug-in slits are available with the D8 ADVANCE / D8 DISCOVER.
- Typically, no primary monochromator

Applications:

- Phase ID and quantification, structure quantification on powders

Setups:

- Examples with 0D and 1D detectors are shown in the figures below.

See also special setup with Johansson monochromator in chapter *Installation of Johansson Monochromator [▷ 73]* .

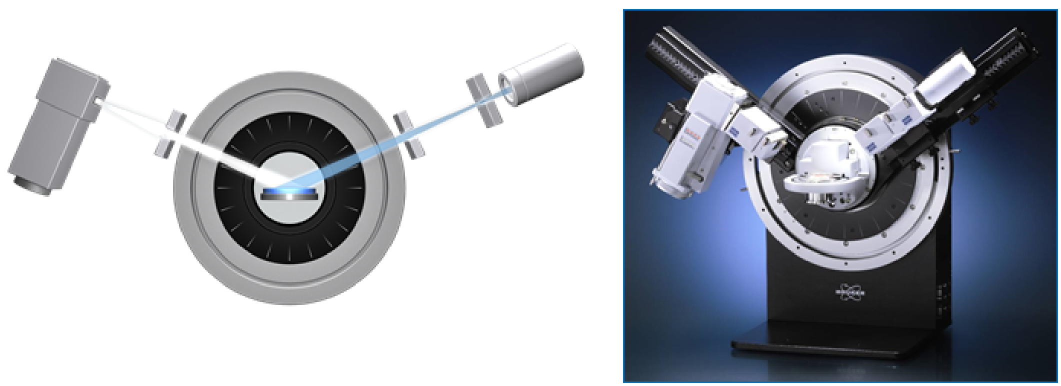


Figure 6.25: Basic Bragg-Brentano setup for phase identification and structure determination. Plug-in slits and Scintillation counter are mounted in this example.

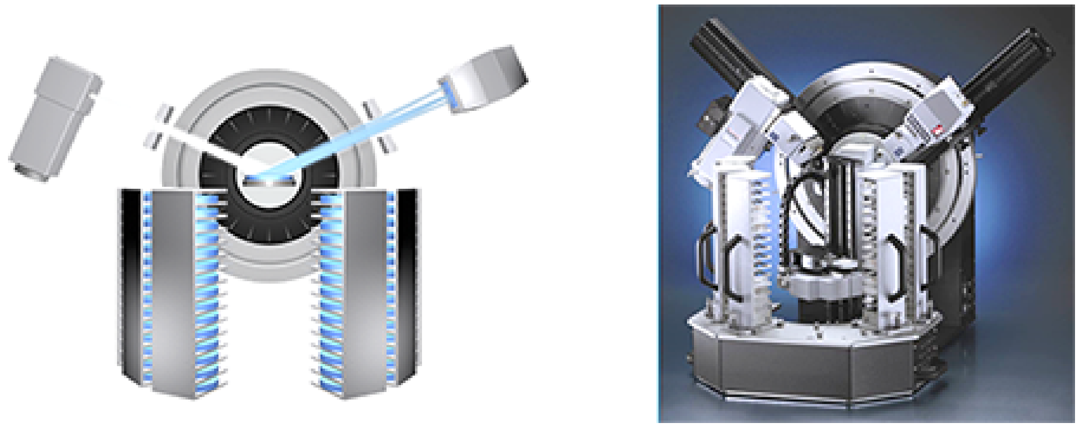


Figure 6.26: Figure 132: Bragg-Brentano setup for high throughput – AUTO-CHANGER and LYNXEYE

6.7.1.2 Transmission Setup (Standard)

Characteristics:

- Fixed measurement circle 500 mm sometimes 435 mm, and 560 mm
- Goniometer podium necessary
- Often used for smaller samples, hence a focusing optic.
- In the transmission geometry, investigation in the low-angle region (TwoTheta less than 5 degrees) requires low-angle background-scatter reduction.
- Be aware that transmission setups with flat samples are realized by steering the beam downwards on a vertical goniometer. When trying to mount the sample stage other than horizontal, the goniometer and/or the stage might be damaged.

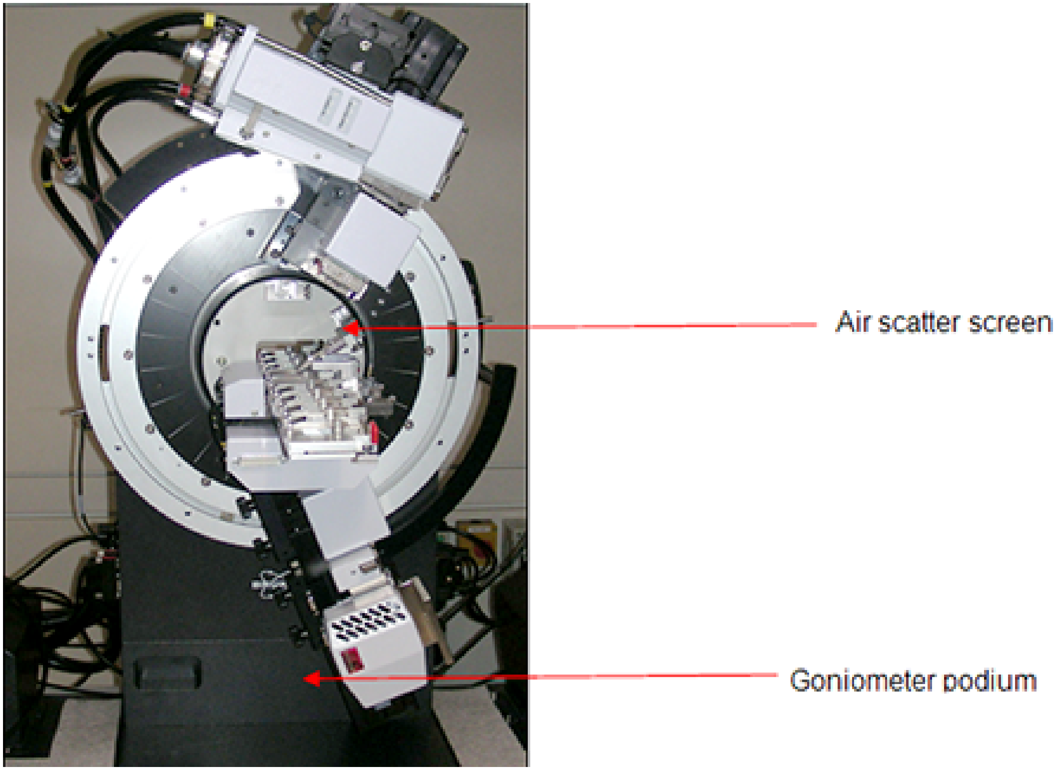
Applications:

- Phase ID, structure determination, high-throughput screening on powders.

Setups:

- As an example, a setup with primary side focussing Göbel mirror, FLIP-STICK sample changer and LYNXEYE detetector is shown below. Note the mounted goniometer podium and the position of the air-scatter screen.

See also special setup with Johansson monochromator in chapter *Installation of Johansson Monochromator [▸ 73]*.



Typical setup for transmission measurement

6.7.1.3 X-ray Reflectometry Setup (Standard)

Characteristics:

- Beam is usually parallel, hence a fixed measurement circle diameter is not mandatory. Typical measurement circle radii are 250 mm and 280 mm
- Layer thicknesses in sub-micron range, depending on composition, can be determined.
- Knife-edges can be used to restrict the measured area.

Applications:

- Layer thicknesses, roughnesses and densities

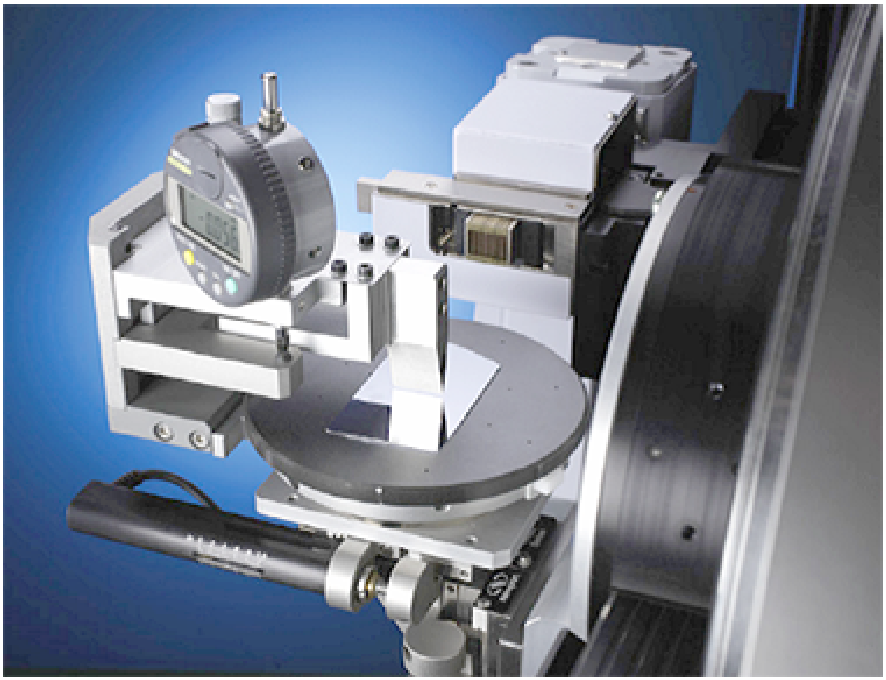


Figure 6.27: Sample area of a reflectometry setup, showing a compact XYZ stage and a knife edge

6.7.1.4 Non-Ambient Setup (Standard)

Characteristics:

- Measurement diameter starting from 500 mm.
- Sample space requirements approximately the same as a standard sample carrier.

Figure 6.28: Non ambient setup with VÅNTEC-1

6.7.1.5 Stress and Texture Setup (Standard)

Characteristics:

- Measurement diameter starting from 500 mm (depends on sample stage)
- Sample Stages: Compact Eulerian Cradle, compact UMC, Standard, Rotary, FLIP-STICK, AUTO-CHANGER, Capillary, Non Ambient chambers
- UBC collimator (50 µm - 2 mm)

- Optics: Bragg-Brentano, POLYCAP, Göbel mirror
- Detector: Typically with Scintillation counter sometimes LYNXEYE or VÅNTEC-1

Stress:

- In many cases with Cr-radiation and point focus X-ray tube.
- Side inclination mode requires a Eulerian cradle and a point spot on the sample. This is achieved either with a point focus tube or by universal beam concept: Göbel mirror combined with micro slit and UBC collimator.
- Iso inclination mode, also called omega mode, which is more flexible regarding sample stages and optics.

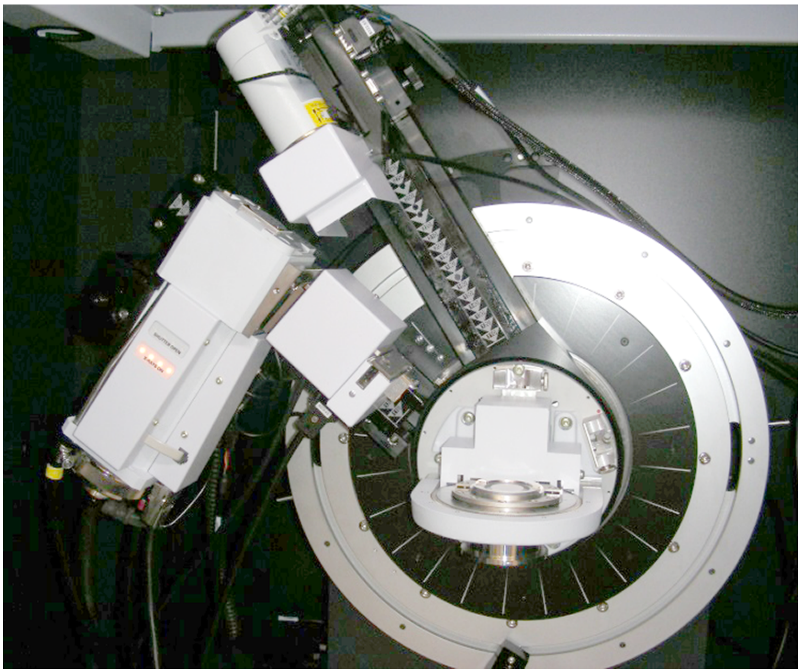


Figure 6.29: Residual stress detemination in isoclination mode

Texture:

- Always requires a compact Eulerian cradle and a point shaped X-ray spot on the sample.

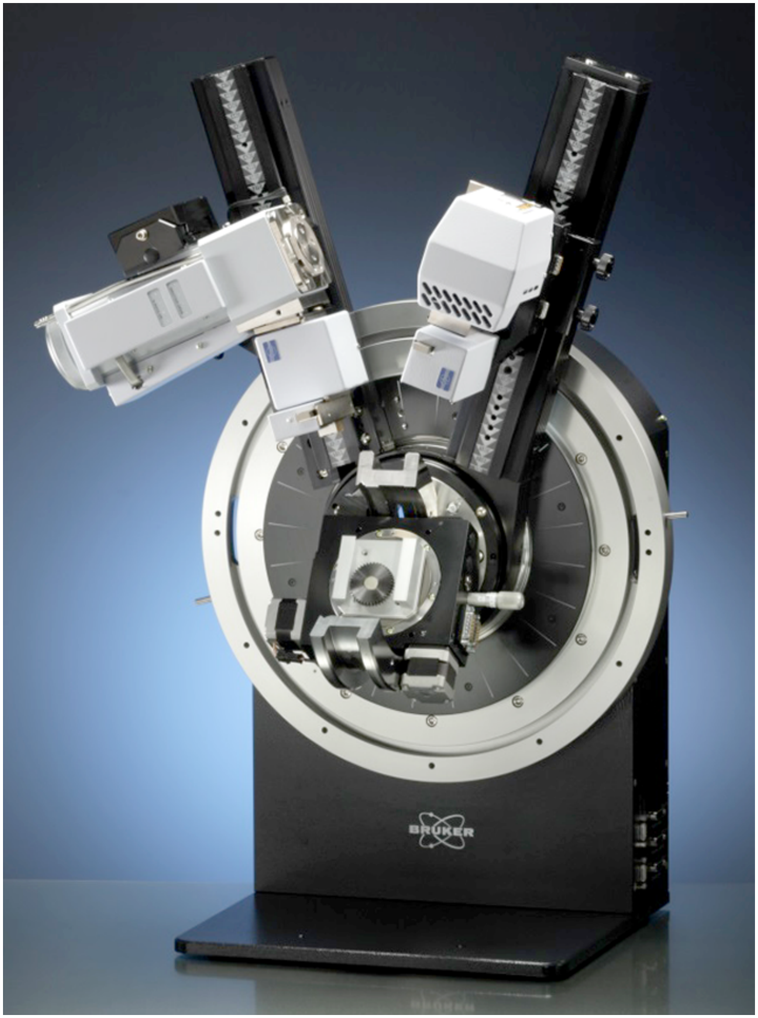


Figure 6.30: Example for Texture setup

6.7.2 TWIN (TRIO)/TWIN Configuration

Characteristics:

- Primary TWIN optics provide automated switching between parallel-beam (with the Göbel mirror optic) and Bragg-Brentano (with a motorized slit) geometries.
- Secondary TWIN optics provides automated switching between an equatorial Soller slit and a motorized slit.
- 560 mm diameter only
- software-controlled changing of setups
- Free choice of sample stages: Standard, Rotary, FLIP-STICK, AUTO-CHANGER, Capillary, Compact XYZ stage, Compact Eulerian Cradle, Non Ambient
- In addition to the primary TWIN components (Göbel mirror, motorized slits), the TRIO optics offers an **asymmetric channel cut Ge 004 2-bounce monochromator**. The monochromator can be inserted into the beam path after the Göbel mirror. As the TWIN optics, the TRIO is motorized, allowing for software controlled switching between the motorized slit, Göbel mirror and Göbel mirror + monochromator beam path.

The channel cut introduces a translational offset to the beam. The offset is measured during installation and recorded in the system configuration. This allows for automated offset correction with the motorized beam translation, which is triggered by inserting the channel cut into the beam. However, since the offset beam is no longer centered in the slit mount, measurements with the TRIO in channel cut configuration require special, off-centered beam defining slits. Just like the standard slits, the off-centerd slits are manually mounted and recognized by the Optical Component recognition system.

- Inserting the channel cut into the beam path will add an offset to the displayed beam translation position. The offset is corrected by moving the beam translation to zero.

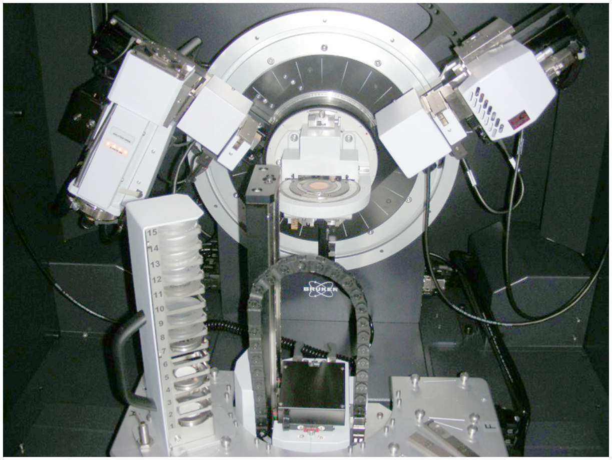


Figure 6.31: TWIN/TWIN setup, which allows pure software switching between different applications (Bragg-Brentano, parallel beam, Grazing Incidence Diffraction, X-ray reflectivity, Microdiffraction). In this example combined with an AUTO-CHANGER sample stage.

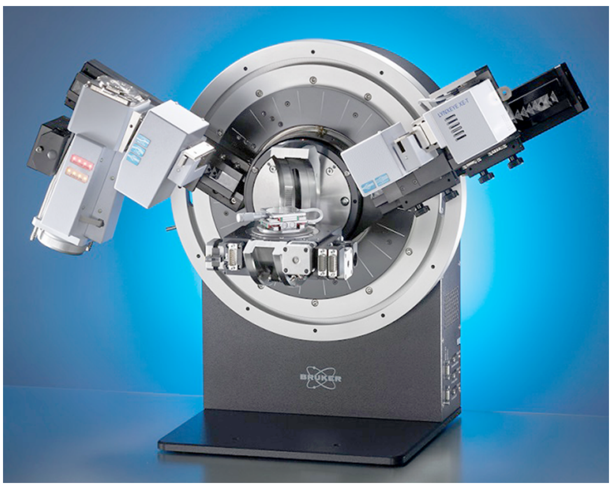


Figure 6.32: TRIO/TWIN setup, in addition to the TWIN/TWIN functionality, allowing for high resolution diffraction measurements with the primary side channel cut Ge 004 monochromator. In this example combined with the Compact Cradle^{plus} and a manual XY sample stage.

6.7.2.1 X-ray Reflectometry Setup (TWIN/TWIN)

The [▷ 181] following figure shows a reflectometry configuration as realized with TWIN/TWIN optics. The primary TWIN optics is in the Göbel mirror configuration. A knife edge above the sample is shown. The secondary TWIN optics is in the motorized slit configuration with a 90° rotated LYNXEYE detector for achieving a high dynamic range in 0D mode.

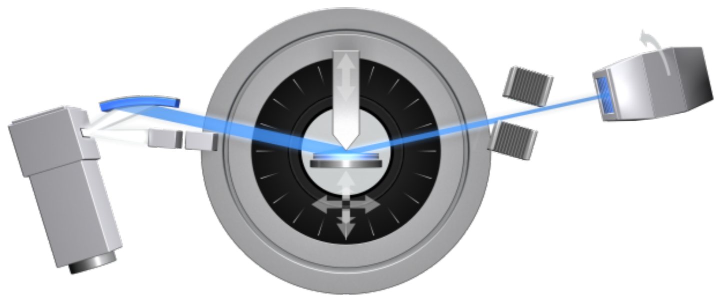


Figure 6.33: X-ray Reflectometry Setup (TWIN/TWIN; TRIO/TWIN)

6.7.2.2 X-ray Reflectometry Setup (TRIO/TWIN)

The figures below show a reflectometry configuration as realized with TRIO/TWIN optics. The primary TRIO optics is in the Göbel mirror + channel cut configuration. A knife edge above the sample is shown. The secondary TWIN optics is in the motorized slit configuration with a 90° rotated LYNXEYE detector for achieving a high dynamic range in 0D mode.

Figure 6.34: X-ray Reflectometry setup with the TRIO/TWIN optics

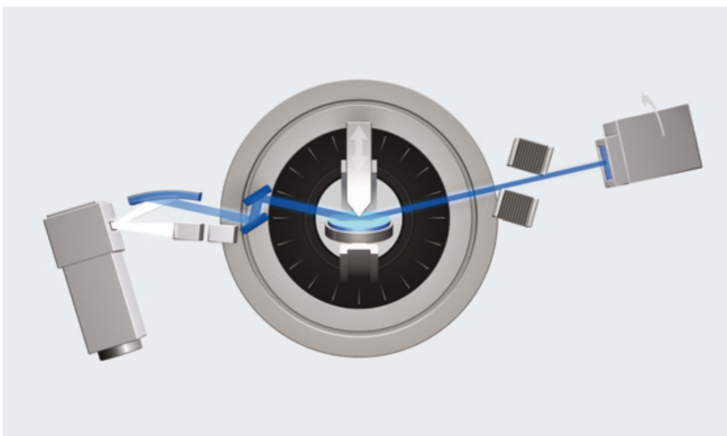


Figure 6.35: Schematic drawing showing the X-ray Reflectometry configuration using the TRIO/TWIN optics.Bragg-Brentano Setup (TWIN/TWIN)

6.7.2.3 Bragg-Brentano Setup (TWIN/TWIN)

TWIN/TWIN Bragg-Brentano mode. Primary and secondary side TWIN set to motorized slit (TRIO/TWIN setup also possible).

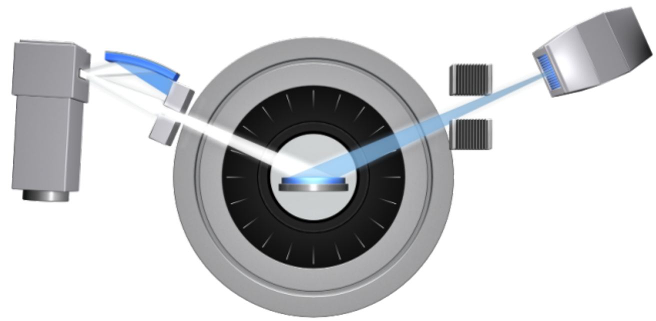


Figure 6.36: FTWIN/TWIN Bragg-Brentano mode

6.7.2.4 Grazing Incidence Setup (TWIN/TWIN)

TWIN/TWIN - Grazing Incidence Diffraction: Primary TWIN set to Göbel mirror and secondary side TWIN set to equatorial Soller (TRIO/TWIN setup also possible)

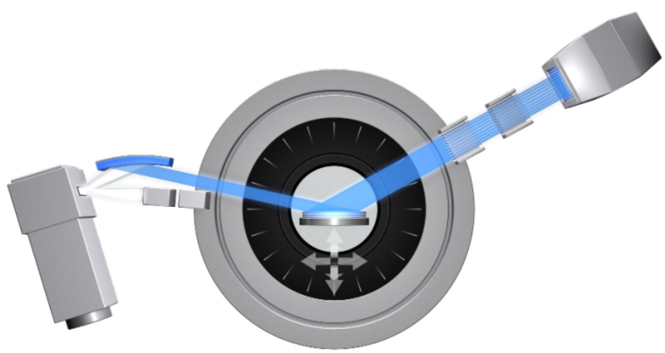


Figure 6.37: TWIN/TWIN - Grazing Incidence Diffraction

6.7.2.5 Microdiffraction Setup (TWIN/TWIN)

TWIN/TWIN – Microdiffraction: Primary TWIN set to Göbel mirror, Microslit and secondary side TWIN set to slit (TRIO/TWIN setup also possible)

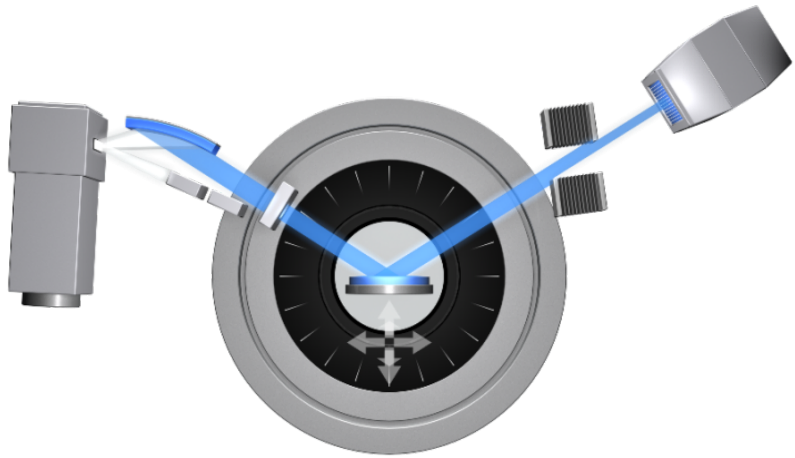


Figure 6.38: TWIN/TWIN – Microdiffraction

6.7.2.6 High Resolution Diffraction Setup (TRIO/TWIN)

The figures below show the TRIO/TWIN HRXRD setup. The primary TRIO optics is in the Göbel mirror and channel cut configuration. The sample is placed on the Compact Cradle-plus with the manual X,Y table attachment.

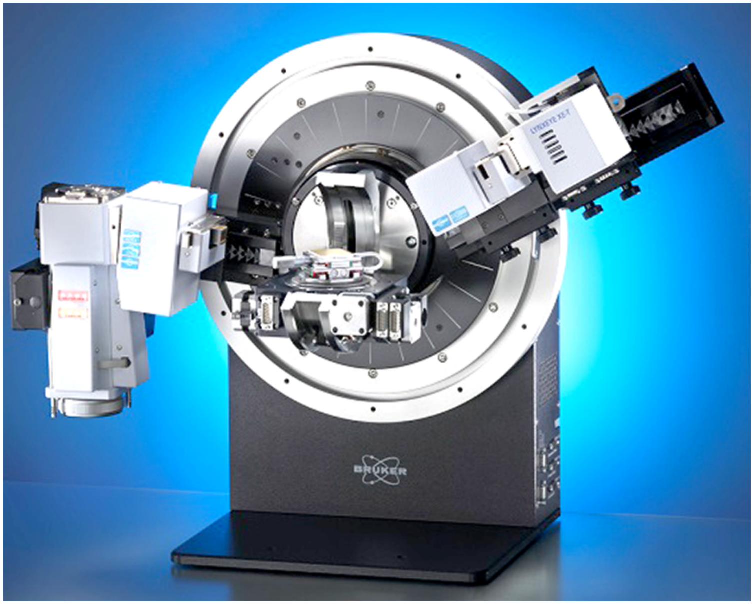


Figure 6.39: TRIO/TWIN HRXRD configuration

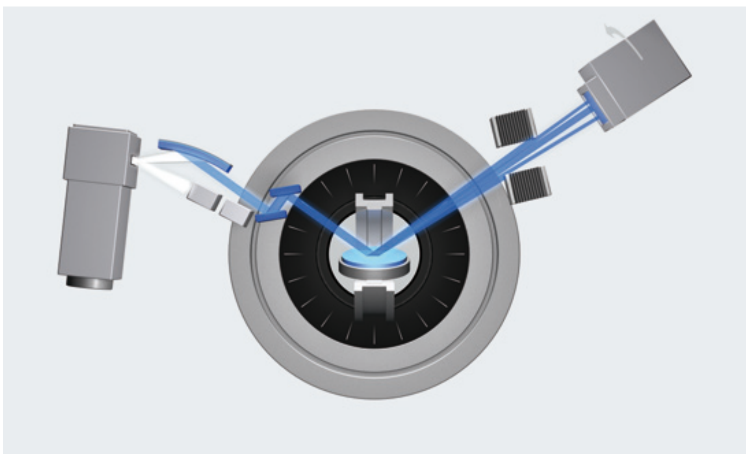


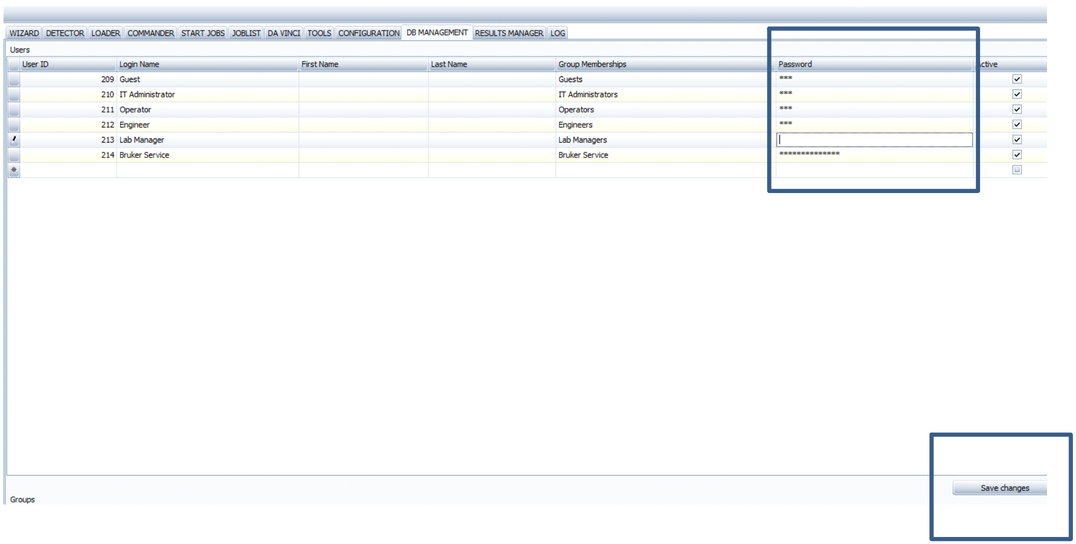
Figure 6.40: Schematic drawing of the TRIO/TWIN HRXRD configuration

6.8 How to Change User Profile Passwords

Reset the password

▶ You need to login as a user with sufficient rights to change the passwords. The **Lab Manger** and **IT Administrator** have the right to set and change passwords for different user profiles.

1. Open the **MAINTAINANCE** plugin.
2. Click on the **DB MANAGEMENT** plugin.



3. Enter the new password in the password column, then
4. click on the **Save Changes** button.
5. Close the Software and restart it again.

⇨ Password changes will be effectiv after this software restart.

NOTICE

Reset the password after installation

After installation the passwords for the different user profiles must be changed by the operator of the system.

- ▶ It is the responsibility of the operator of the system to make sure that a trained person gets access to a profile with the rights to reset the safety module.
- ▶ To prevent unauthorized use we highly recommend changing the password for **Lab Manager** and **IT Administrator**.
- ▶ It may be also recommended by national regulations that you must define a specific password for each user.

7 References

Documentation	Order number
D8 ADVANCE Introductory User Manual (Preinstallation, Safety, Specifications)	DOC-M88-ZXX146
D8 DISCOVER Introductory User Manual (Preinstallation, Safety, Specifications)	DOC-M88-ZXX151
D8 ADVANCE / D8 DISCOVER User Manual Volume 1	DOC-M88-EXX153
D8 DISCOVER User Manual Volume 2	DOC-M88-EXX162
D8 ADVANCE Supplement Folder	DOC-M88-ZXX152
D8 DISCOVER Supplement Folder	DOC-M88-ZXX163
AUTO-CHANGER User Manual	DOC-M88-EXX101
Compact UMC stage User Manual	DOC-M88-EXX221
Compact Eulerian Cradle with Motorized Z Drive User Manual	DOC-M88-EXX220
LYNXEYE User Manual	DOC-M88-EXX095
LYNXEYE XE User Manual	DOC-M88-EXX240
LYNXEYE XE-T User Manual	DOC-M88-EXX239
VÅNTEC-1 User Manual	DOC-M88-EXX072
VÅNTEC-500 User Manual	DOC-M88-EXX177
PILATUS3 R 100K A User Manual	DOC-M88-EXX280
EIGER2 R 500K User Manual	DOC-M88-EXX293
MTC- Modular Temperature Chambers for D8 ADVANCE / D8 DISCOVER	DOC-M88-EXX250

Documentation	Order number
TC - Temperature Chambers for D8 ADVANCE / D8 DISCOVER	DOC-M88-EXX269
Anton Paar Temperature Chambers for D8 ADVANCE / D8 DISCOVER	DOC-M88-EXX268
DIFFRAC.SUITE Installation Guide	DOC-M88-EXX190
DIFFRAC.SUITE User Manual	DOC-M88-EXX191
XRD Wizard Reference Manual	DOC-M88-EXX192
DIFFRAC.EVA User Manual	DOC-M88-EXX200
D8 Systems Instrument Verification Booklet	DOC-M88-EXX157

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