

ARTISAN®
TECHNOLOGY GROUP

Your **definitive** source for quality pre-owned equipment.

**Artisan Technology Group**
(217) 352-9330 | sales@artisantg.com | artisantg.com

**Full-service, independent repair center**
with experienced engineers and technicians on staff.

**We buy your excess, underutilized, and idle equipment**
along with credit for buybacks and trade-ins.

**Custom engineering**
so your equipment works exactly as you specify.

- Critical and expedited services
- In stock / Ready-to-ship
- Leasing / Rentals / Demos
- ITAR-certified secure asset solutions

**Expert team | Trust guarantee | 100% satisfaction**

All trademarks, brand names, and brands appearing herein are the property of their respective owners.

**Visit our website - *Click HERE***

BRUKER




# D8 ADVANCE
# D8 DISCOVER

**User Manual**
**Original Instructions**

XRD

Innovation with Integrity

The reproduction, transmission or use of this document or its contents is not permitted without express written authority. Offenders will be liable for damages. All rights reserved.

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.

Order no. DOC-M88-EXX153 V3. Updated: Nov 18, 2010.

© 2010 Bruker AXS GmbH, Karlsruhe, Germany.

All trademarks and registered trademarks are the sole property of their respective owners.

Printed in the Federal Republic of Germany.

Bruker AXS GmbH
Östliche Rheinbrückenstr. 49
76187 Karlsruhe, Germany
Tel. +49 721 5 09 97-0
Fax +49 721 5 09 97-56 54
info@bruker-axs.de
www.bruker.com

# D8 ADVANCE / D8 DISCOVER User Manual Vol. 1



**Caution!**

Read the manuals ‘D8 DISCOVER Introductory User Manual’ and ‘General Safety Instructions" completely before switching on the D8 diffractometer.

## Table of Contents

**Getting Started ..... 1**
- Introduction ..... 1
- Starting the Instrument ..... 3
- Placing Sample in Sample Changer/Loading Sample by Hand ..... 7
- Starting the Software ..... 8
- Initializing the Drives ..... 9
- Performing a Scan ..... 12
- Saving the Results of Scans ..... 16
- Shutting Down the Instrument ..... 16

**General Physical Principles ..... 18**
- Production of X-Rays ..... 18

Continuous Spectrum ... 19
Characteristic Spectrum ... 20
**X-Ray Diffraction ... 23**
The Bragg Equation ... 23
**Measurement Geometries of X-Ray Diffraction Systems ... 25**
Bragg-Brentano Geometry ... 25
θ-θ and θ-2θ Systems ... 27
Parallel Beam Geometry ... 29

**The D8 ADVANCE / D8 DISCOVER: Locations and Functions of Basic Components ... 31**
**Common components of the D8 ADVANCE and D8 DISCOVER ... 31**
Power Switch ... 31
Enclosure Control Buttons ... 33
Generator Screen-key and System Status Screen-key ... 34
**The Experimental Area ... 35**
Goniometer ... 35
Counterweights ... 37
X-Ray Tube ... 37
Labyrinths ... 39
Accessories Shelf ... 39
**Overview of the locations of components in the D8 ADVANCE diffractometer ... 41**
**The External Housing of the D8 ADVANCE ... 41**
Door Mechanism D8 ADVANCE ... 42
**Underneath the Experimental Area D8 ADVANCE ... 43**
Removing the Panels ... 43
Lower Front D8 ADVANCE ... 43

Lower Back D8 ADVANCE ....44
Lower Right Side D8 ADVANCE ....45
Lower Left Side D8 ADVANCE ....46
**Overview of the locations of components in the D8 DISCOVER diffractometer ....48**
**The External Housing of the D8 DISCOVER ....49**
Door Mechanism D8 DISCOVER ....50
**Underneath the Experimental Area D8 DISCOVER ....52**
Removing the Panels ....52
Lower Front D8 DISCOVER ....52
Lower Back D8 DISCOVER ....54
Lower Left Side D8 DISCOVER ....55
Lower Right Side D8 DISCOVER ....56

**The D8 ADVANCE and D8 DISCOVER: Descriptive Listings of Common Components ....57**
**Primary Side: Alignment Controls and Optics Mounts ....57**
Alignment Concept ....58
Beam-Steering Plates ....59
Rotation Beam-Steering Plate ....59
Rotation + Translation Beam-Steering Plate ....61
Takeoff Angle Adaptor Plates ....63
**Secondary Side: Optics and the Universal Detector Mount ....66**
**Automatic Component Recognition ....66**
SNAP-LOCK ....67
**Tubes ....68**
**Primary Optics ....69**
Safety Slit ....69

Mirrors ... 69
Standard Primary D8 Mirrors ... 69
Monochromators ... 69
Installation of Johansson Monochromator ... 70
Details on Primary Monochromator in Transmission and Capillary Setup ... 71
Details on Primary Monochromator in Reflection Setup ... 74
**Secondary Optics ... 80**
Soller Slit ... 80
Diffracted Beam Monochromator ... 80
Application ... 81
Design and Mode of Operation for focussing geometry (Bragg-Brentano) ... 81
Technical Data ... 83
Installation ... 84
Application ... 85
Operation ... 86
Technical Data ... 86
**Slits, Filters, and Absorbers ... 87**
Axial Soller slits ... 88
UBC collimator ... 89
Air Scatter Screen ... 90
**Stages and Sample Holders ... 94**
Standard Sample Stage ... 94
Insertion of Samples ... 94
Accessories ... 97
Rotary Sample Stage ... 101
FLIP-STICK ... 106
General Features ... 107
D8 Standby ... 108

Change of Sample Rings ..... 108
Release of Magazine ..... 108
Insertion of Magazine ..... 109
Transmission Measurements ..... 110
Accessories ..... 111
Sample Rings ..... 112
Figures ..... 117
AUTO-CHANGER ..... 125
Compact XYZ Stage ..... 126
Compact Eulerian Cradle ..... 128
Capillary Stage ..... 131
Non Ambient ..... 133
**Detectors ..... 136**
Scintillation Detector ..... 136
LYNXEYE ..... 137
VÅNTEC-1 ..... 138
SOL-XE ..... 139

**Operating the D8 ADVANCE/ D8 DISCOVER ..... 143**
**Instrument Status ..... 143**
Checking and Optimizing the Primary Beam Intensity ..... 143
**Tube ..... 144**
Conditioning ..... 144
Changing Tubes ..... 145
Switching Between Point and Line Focus ..... 145
**Mounting Optics ..... 150**
Optics Mount (SNAP-LOCK) ..... 150

Optics mounting status displayed in DAVINCI plug-in ..... 153
**Mounting Sample Stages ..... 154**
Stage status displayed in DAVINCI plug-in ..... 156
**Mounting Detectors ..... 157**
Detector status display in DAVINCI plugin ..... 163
**Typical Scans ..... 164**
Axes vs. Motorized Drives ..... 164
Theta (θ) or Omega (ω) Axis ..... 164
TwoTheta and Detector Axis ..... 165
Offset ..... 165
Deflection Angle ..... 165
Reference Position ..... 165
General procedure for optimizing reference positions and deflection angles ..... 166
The beam path and deflection angle concept ..... 166
Basic setup ..... 167
Additional setups ..... 167
The focus position alignment ..... 168
Alignment of the focus position ..... 168
Glass slit alignment ..... 169
Zero Point Definition of the θ Scale ..... 169
Zero Point Definition of the 2θ Scale ..... 170
**Standard and TWIN/TWIN Configurations ..... 170**
1. Standard Configuration ..... 171
Dedicated Bragg-Brentano Setup (Standard) ..... 171
Transmission Setup (Standard) ..... 173
X-ray Reflectometry Setup (Standard) ..... 175
Non-Ambient Setup (Standard) ..... 176
Stress and Texture Setup (Standard) ..... 176

2. TWIN/TWIN Configuration ..... 179
X-ray Reflectometry Setup (TWIN/TWIN) ..... 181
Bragg-Brentano Setup (TWIN/TWIN) ..... 181
Grazing Incidence Setup (TWIN/TWIN) ..... 182
Microdiffraction Setup (TWIN/TWIN) ..... 182
**References ..... 183**

# D8 ADVANCE / D8 DISCOVER User Manual Vol. 1

# Getting Started

## Introduction

The Bruker AXS D8 ADVANCE/ D8 DISCOVER X-ray diffractometers are optimally designed for use in all X-ray diffraction applications in material research, powder diffraction and high resolution diffraction. All new D8 ADVANCE/ D8 DISCOVER goniometers are equipped with stepper motors with optical encoders to ensure extremely precise angular values. All X-ray components are mounted on high-precision dovetail tracks and can be quickly replaced owing to reproducible positioning. They can also be freely moved along the tracks. The DAVINCI module of the Bruker AXS DIFFRAC.SUITE, the software system that supports the Bruker AXS D8 ADVANCE/ D8 DISCOVER X-ray diffractometers, allows quick switching between the optics setups, sample stages and sample environments.

The Bruker AXS DIFFRAC.SUITE allows remote instrument control via intranet or internet for convenient operation. Alignment routines or measurements can be completely automated using a script language.

The D8 ADVANCE/ D8 DISCOVER diffractometers offer full security protection according to the safety regulations in force.

This “Getting Started” is designed as a quick and easy guide to starting the Bruker AXS D8 ADVANCE/ D8 DISCOVER X-ray diffractometer and performing an example X-ray scan using the Bruker AXS DIFFRAC.SUITE, a set of software applications that support Bruker AXS X-ray diffractometers. It is intended for persons unacquainted with these Bruker products and desirous of gaining a first experience of them.

The example X-ray scan for which instructions are provided involves the use of a Corundum sample. It is performed on a theta-theta diffractometer using a Copper anode.



**Note**

Before starting the instrument and performing a measurement, the proper instrument installation and alignment should have been carried out by a Bruker AXS service representative.

To learn more about working with the Bruker AXS D8 ADVANCE/ D8 DISCOVER X-ray diffractometer and the Bruker AXS DIFFRAC.SUITE, refer to the user manuals for these products.

# Starting the Instrument

Refer to Figure 1 for the instructions in this section.

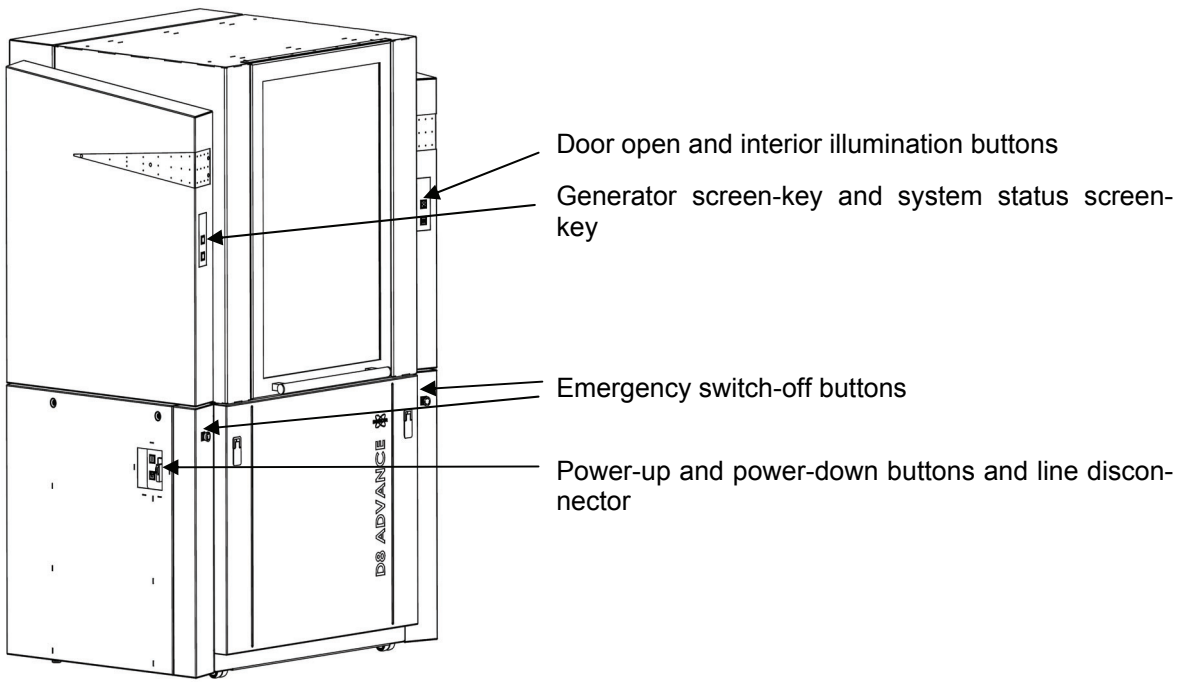



Figure 1: Controls of D8 ADVANCE Diffractometer.

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

Turn the line disconnector, which is located in the niche in the lower left side of the instrument, clockwise from “0” to “1”.

Result: The power supply to the instrument and its electrical components is switched on.

Press the power-up button in the niche in the lower left side of the instrument. This is the green push-button inscribed with “I”.

Result: 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 starts 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.

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

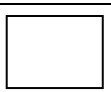
Result: The generator screen-key turns to yellow and starts blinking, indicating that the cathode is heating up. After a few seconds, the blinking stops and an X-ray symbol  appears, indicating that X-ray generation has started. In this phase, the generator voltage is ramped up to a value that has been 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.

**States of Generator and System Status Screen-keys**

In the tables below are described the different states of the generator and system status screen-keys as well as the symbols that appear on them and their meanings.

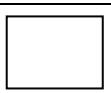
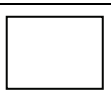
Tab. 1: Generator Screen-key

| | |
|---|---|
|  | **Constant illumination in white**<br>Indicates the instrument is in its start-up phase. |

| | |
|---|---|
|  | **Switch-on symbol**<br>Displayed when the generator is off and ready to be switched on. Press the screen-key once to start the generator and thus X-ray generation. |
|  | **Heating on**<br>blinking yellow light indicates the tube is being heated but no X-rays are being generated.<br>constant illumination in yellow indicates the tube is being heated, the switch-off circuit has been closed, but no X-rays are being generated.<br>Press once to switch off the generator. |
|  | **X-rays on, generator ramping up/down**<br>Displayed when X-rays are being generated and the generator is ramping the supply voltage up or down. The background is yellow.<br>Press once to switch off the generator. |
|  | **X-rays on, generator ready**<br>Displayed when the generator has reached a value set in the instrument configuration. This symbol is a negative image of the “ramping up/down” symbol.<br>Press once to switch off the generator. |
|  | **X-ray safety circuit error**<br>If a safety circuit error occurs, the generator will be automatically switched off. To diagnose the error, check the state of the safety board using the TOOLS plug-in of the Bruker AXS DIFFRAC.SUITE.<br>When the error is repaired and providing no other error has occurred, this symbol disappears and is replaced by the switch-on symbol. |
|  | **Generator error**<br>Either the generator or one of its connected components (tube, safety circuit or water cooling system) is affected by an error. To diagnose the error, check the state of the X-ray generator using the TOOLS software module of the Bruker AXS DIFFRAC.SUITE.<br>When the error is repaired and providing no other error has occurred, this symbol disappears and is replaced by the switch-on symbol. |

| | |
|---|---|
| ≈ | **Water cooling system error**<br>The water cooling system has switched off the generator because of an error affecting the cooling system. To diagnose the error, check the state of the water cooling unit using the TOOLS software module of the Bruker AXS DIFFRAC.SUITE.<br>When the error is repaired and providing no other error has occurred, this symbol disappears and is replaced by the switch-on symbol. |
| COND | **Conditioning enabled**<br>When this symbol is displayed on a blinking blue background, the tube conditioning is active. To abort this process, press the button once. The generator will switch off. After restarting the generator, you can proceed as normal. |

Tab. 2: System Status Screen-key

| | |
|---|---|
|  | **Blinking white light**<br>Indicates the instrument is booting. |
|  | **Illumination in white**<br>Indicates the instrument has booted and is ready for operation. |
|  | **Illumination in green**<br>Indicates the instrument is controlled by a Measurement client or server. |
|  | **Measurement in progress**<br>This symbol is displayed against a blue blinking background when a measurement is in progress. |
|  | **Door open**<br>The front door is open. A measurement can only be carried out when the door is closed. |

**Error Messages**

| | |
|---|---|
|  | **Sample changer error**<br>The built-in sample changer is affected by an error and user action is required. (You can diagnose the error and repair it using the TOOLS software module of the Bruker AXS DIFFRAC.SUITE.) |
|  | **Detector error**<br>At least one detector is affected by an error. |
|  | **Drive collision**<br>At least two drives have collided. You can carry out repairs using the TOOLS software module of the Bruker AXS DIFFRAC.SUITE. |
|  | **Drive error**<br>At least one drive is affected by an error. You can carry out repairs using the TOOLS software module of the Bruker AXS DIFFRAC.SUITE. |

# Placing Sample in Sample Changer/Loading Sample 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 Bruker AXS DIFFRAC.SUITE.

If the instrument is not equipped with a sample changer, load the sample by inserting it in the sample stage as described in the user manual for the Bruker D8 ADVANCE/ D8 DISCOVER X-ray 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.

# Starting the Software

Bruker AXS X-ray instruments are delivered with the Bruker AXS 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 Bruker AXS DIFFRAC.SUITE User Manual.

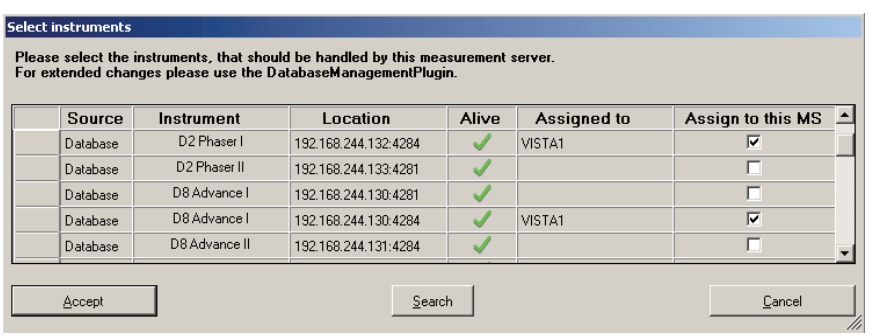



Figure 2: Dialog box **Select instruments** of Measurement Server.

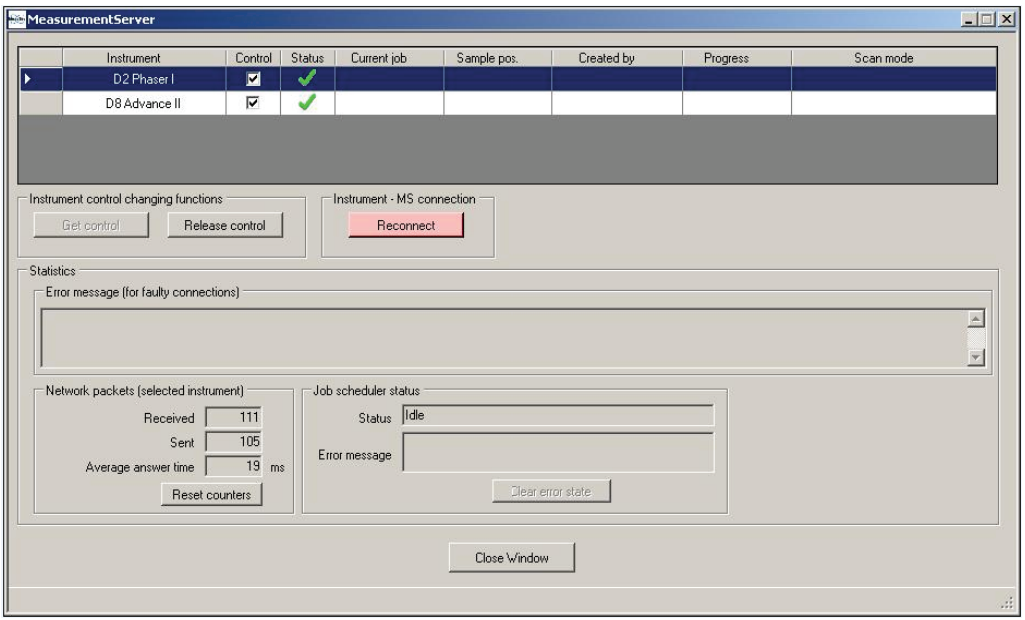



Figure 3: Dialog box **Status window** of Measurement Server.

# 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 Bruker AXS 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.

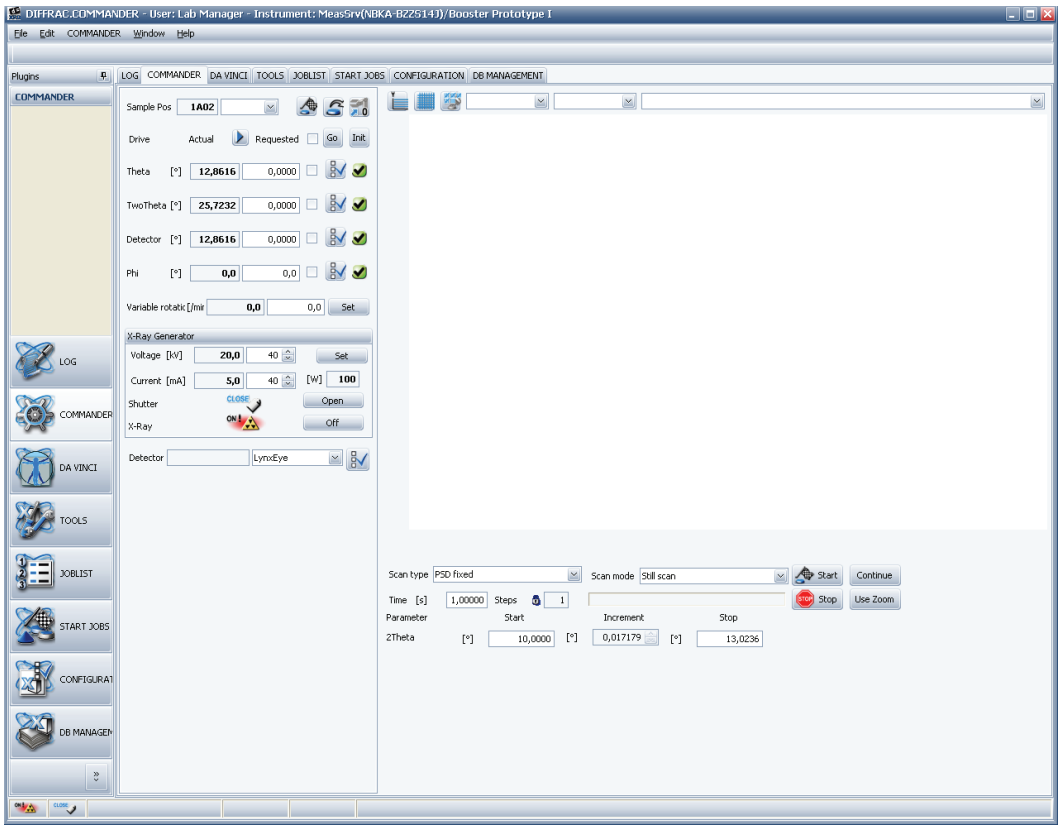



Figure 4: COMMANDER interface – appearance after connection established with instrument.

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 Bruker AXS 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, 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. 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 Bruker AXS DIFFRAC.SUITE User Manual.

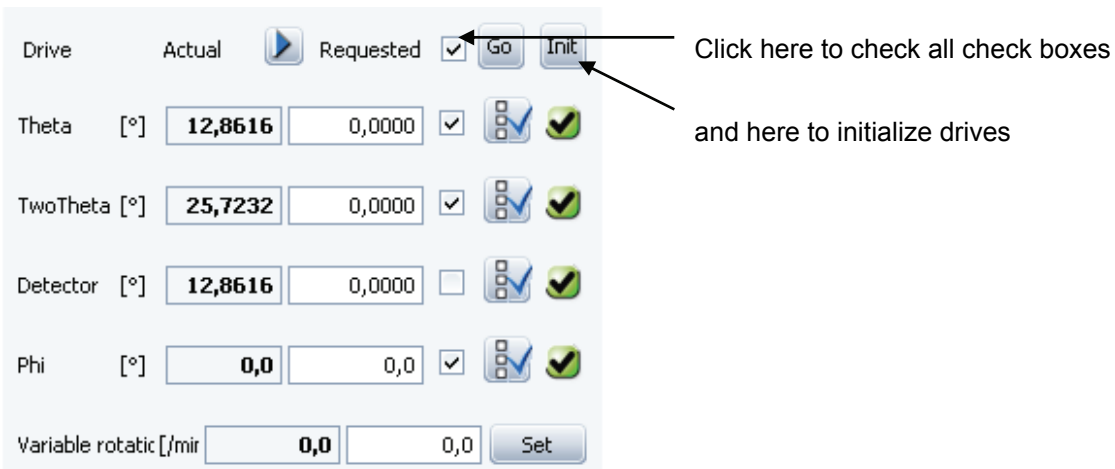



Figure 5: Drive Control area of COMMANDER page.

**Note**

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.

# Performing a Scan

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

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.

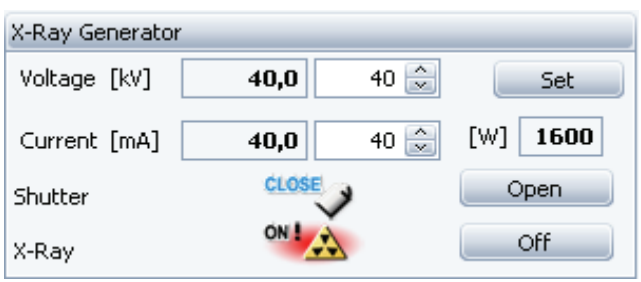



Figure 6: Generator Control area of COMMANDER page.

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.

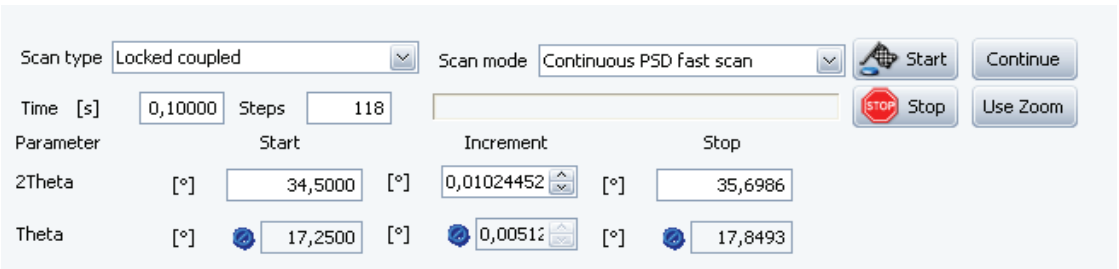



Figure 7: Scan Control area of COMMANDER page.

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, click on the button  in the Sample Changer Control area.

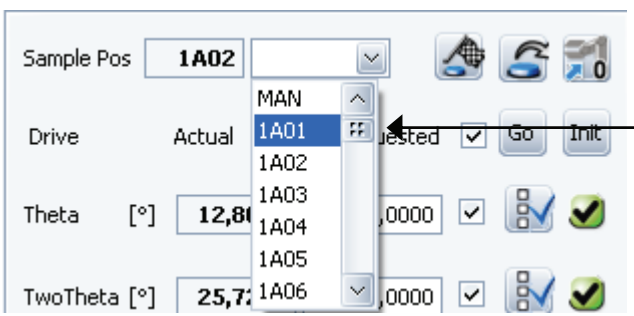



Figure 8: Sample Changer Control area.

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 manuallyby hand as described in Section 0.

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. This is the position in which you will have placed the sample. 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.

Click on the Start button 

in the Scan Control area to start the measurement.

Result: 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.

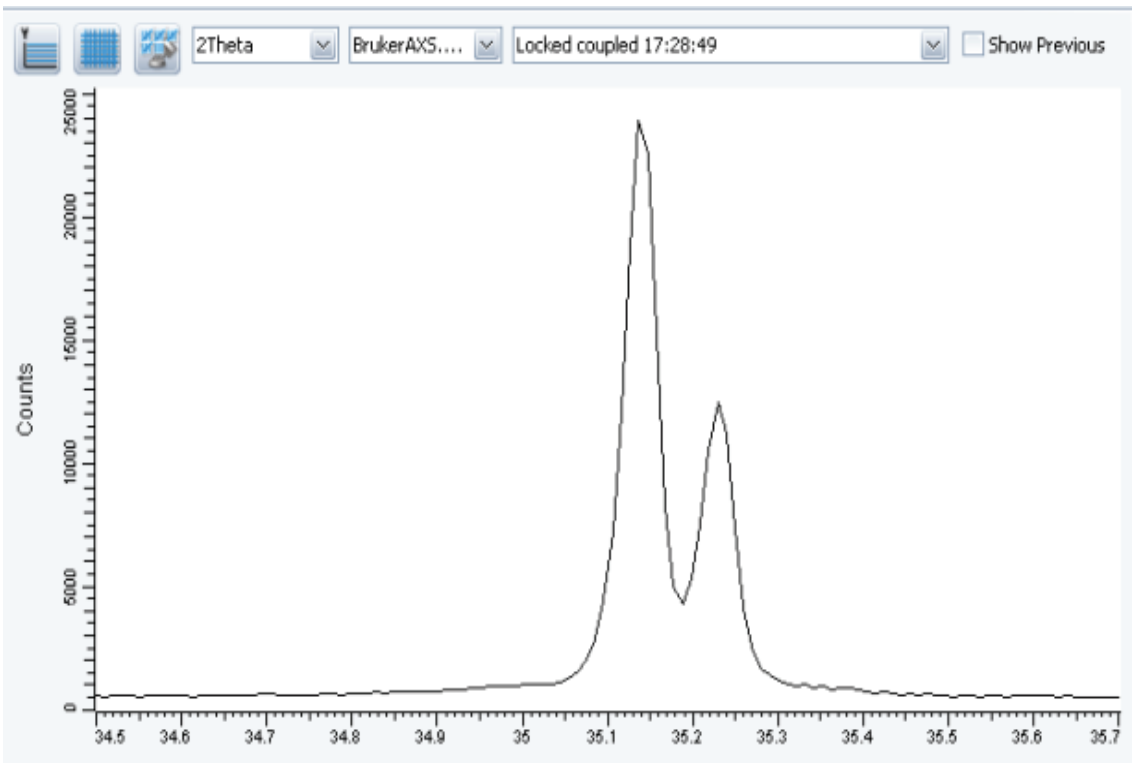



Figure 9: Results of a coupled Theta/TwoTheta scan using a Corundum sample.

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

**Evaluation of Scans**

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

# Shutting Down the Instrument

To shut down the instrument, follow these steps.

Press the generator screen-key to ramp down the generator.

Result: 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.

Press the power-down button, which is the white button inscribed with “Standby” in the niche on the lower left side of the instrument.



**Warning!**

Do not press the power-down button 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 life of the X-ray tube and the high voltage generator.

Result: The instrument is powered down, that is, switched into standby mode. The X-ray generator and all drives stop immediately.

**Shutting Down the Instrument Completely**

If you want to shut down the instrument completely for servicing or safety purposes, turn the line disconnector anti-clockwise from “I” to “0”.



**Warning!**

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.

Result: 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.

# General Physical Principles

## 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.

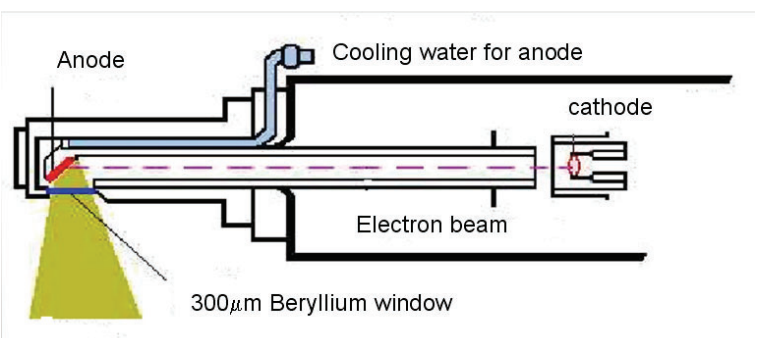



Figure 10: Schematic diagram of X-ray tube.

## Continuous Spectrum

When accelerated electrons collide with atoms of the metal target, they can be stopped on impact, in which case all their energy is converted into radiant energy, or they can be decelerated in steps, in which case a part of their total energy is released in the form of X-rays of greater wavelengths at each step until it is expended. The incident electron enters the electric field between the electrons and the nucleus of an atom and is deflected and decelerated by it at the same time. This process is repeated when the deflected electron enters the electric fields of other atoms.

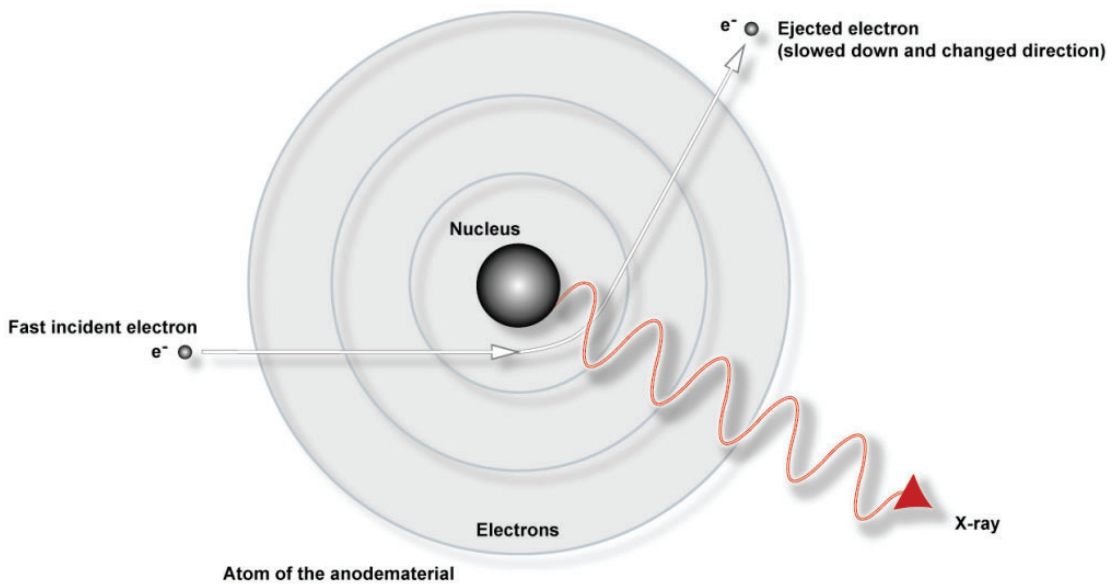



Figure 11: Production of continuous (white) radiation.

The waves of X-rays generated in this way are called “continuous” radiation because they form a continuous spectrum of wavelengths.

## Characteristic Spectrum

High-energy electrons can also interact with the atoms of the metal target to produce another type of radiation called “characteristic” radiation because the wavelengths of the photons emitted are characteristic of the anode metal.

In this case, the incident electron collides with an electron of the first, innermost electron shell of an atom and knocks it out of its orbit. To restore stability to the energy state of the atom, the ejected electron is replaced by an electron from an outer shell. Because the energy levels of shells increases with their distance from the nucleus of the atom, the transition of a replacement electron from an outer to an inner shell is accompanied by a loss in energy, which is released in the form of X-rays.

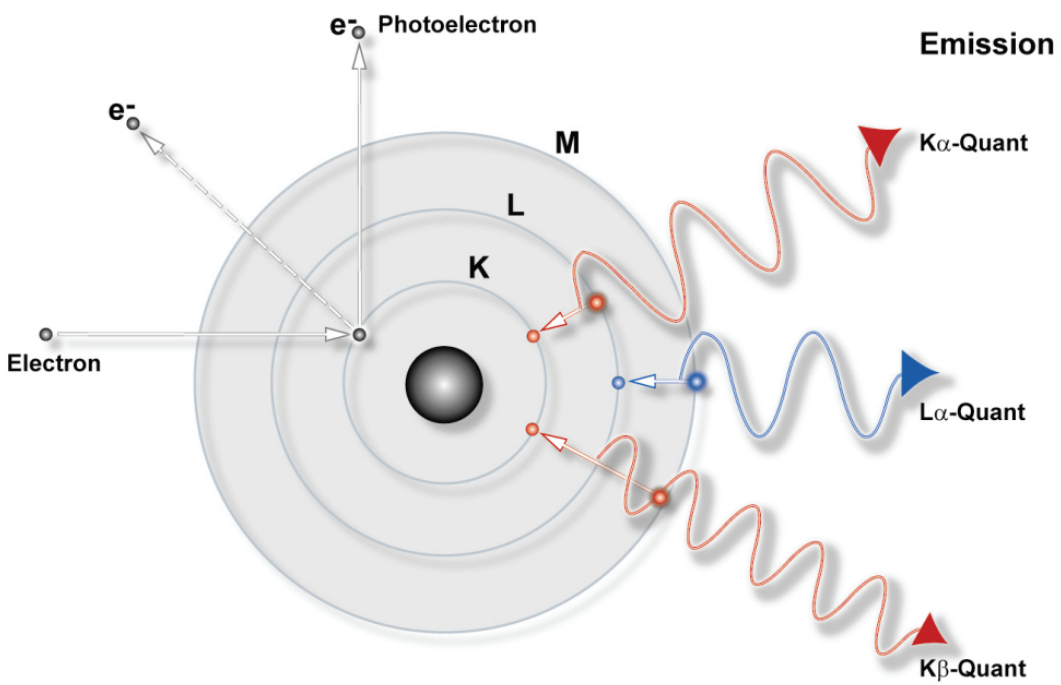



Figure 12: Production of characteristic radiation – simplified representation.

Figure 12 is a simplified representation of the process by which characteristic radiation is produced through the transition of electrons at the atomic level. 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 is produced by an electron transfer from shell L to shell K, Kβ by a transfer from shell M to shell K, and Lα by a transfer from M to L. Note that the photons have different characteristic wavelengths.

In fact, on closer examination of the atom and its electron shells, it can be seen that the shells are divided into subshells as shown in Figure 13 below. 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 and so on. The difference between the wavelengths of photons produced by the transfer of electrons of different subshells within the same shell is very small.

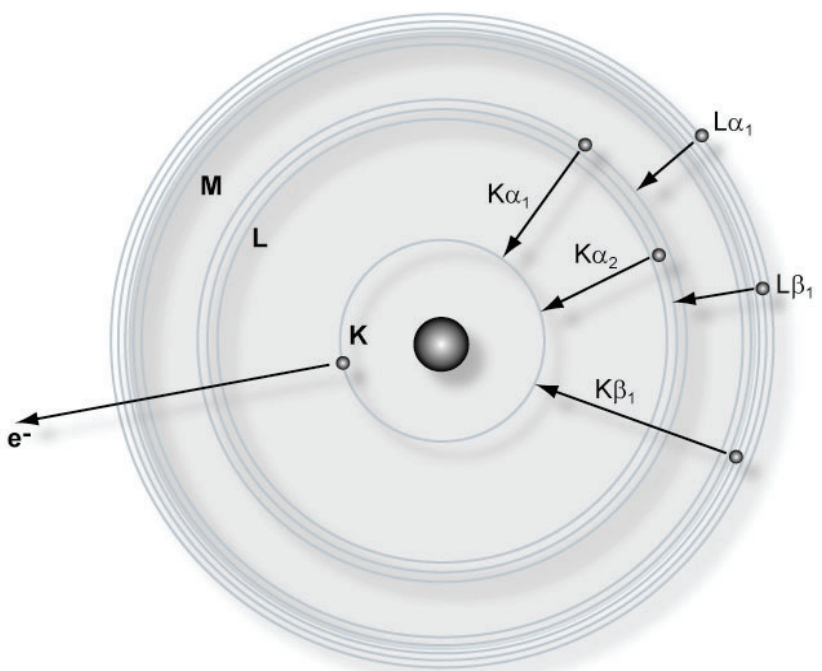



Figure 13: Subshells and their transferred electrons.

In Figure 14 below, the wavelength distribution of both continuous and characteristic radiation is shown as a function of intensity or the number of pulse counts per second. The characteristic peaks $K\alpha_1$, $Ka_2$ 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 (SWL) and corresponds to the radiation produced by the electrons that give up all their energy on the initial impact. Thus for a particular supply voltage, these electrons give rise to photons of maximum energy (minimum wavelength). With an increase in supply voltage, the SWL as well as the point of maximum intensity of the continuous spectrum moves down the wavelength scale. In other words, an increase in operating potential produces a shift of the continuous spectrum in the direction of lower wavelengths and higher energy. On the other hand, the characteristic spectrum, because it is characteristic of the anode metal, merely undergoes an increase in intensity (pulse count rate).

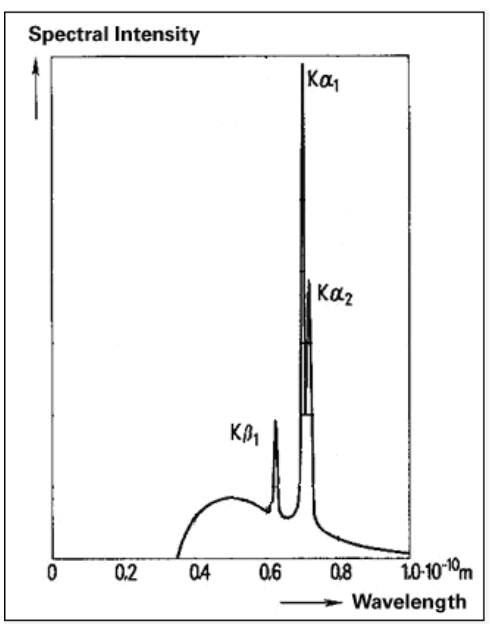



Figure 14: Spectrum of an X-ray tube.

# X-Ray Diffraction

X-ray diffraction is a process in which X-rays impinging on a crystalline material are reflected by the atoms, which are arranged in ordered arrays, when 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 of 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 (coherent 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 reinforce 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 thus 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 it chances of being in the correct position to satisfy the Bragg equation.

## 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.

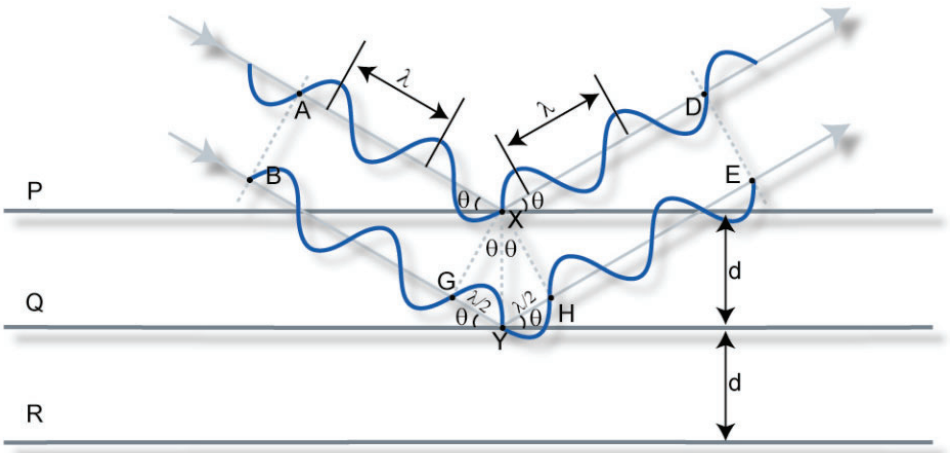



Figure 15: Fulfillment of the Bragg equation.

Figure 15 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.

# Measurement Geometries of X-Ray Diffraction Systems

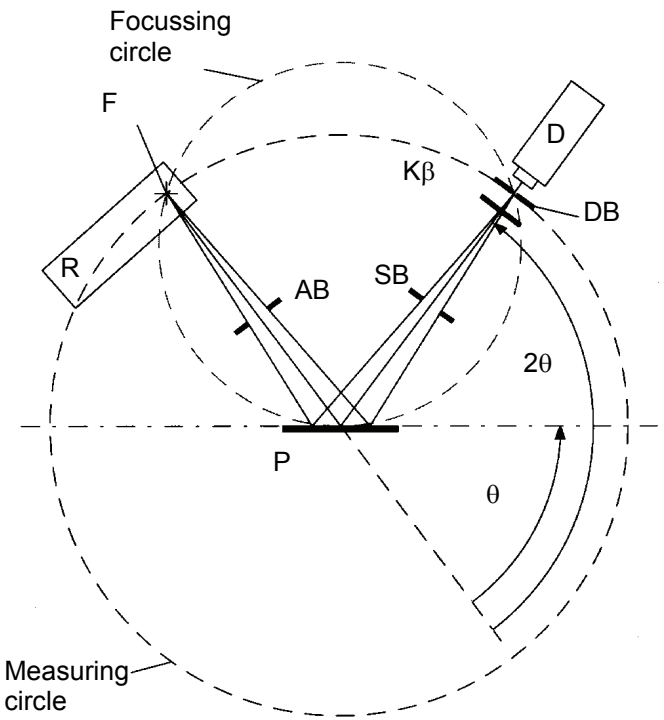
## Bragg-Brentano Geometry

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 in Figure 16. 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.




| | |
|---|---|
| $\theta$ | *Incident angle* |
| $2\theta$ | *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 16: The Bragg-Brentano diffraction geometry.

## θ-θ and θ-2θ Systems

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

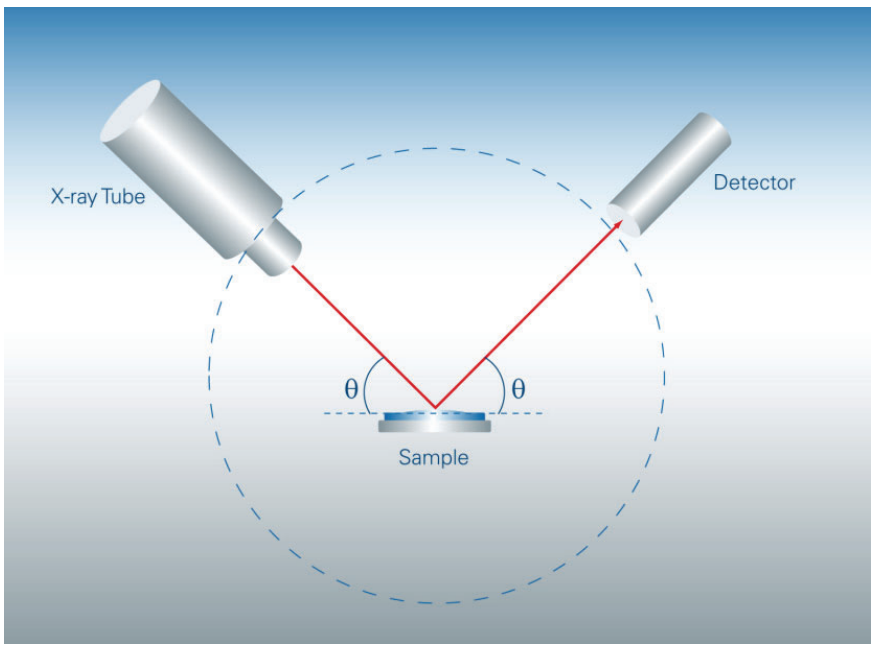



Figure 17: θ:θ configuration.

In θ:θ goniometers (see Figure 17), 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 for capture of 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.

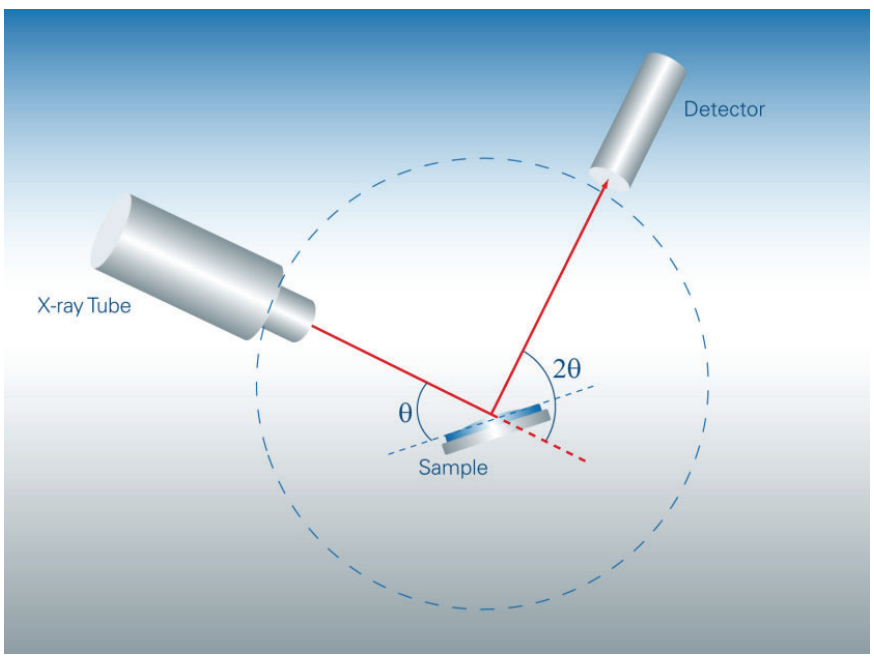



Figure 18: θ-2θ configuration.

In θ-2θ diffactometers (see Figure 18), 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.

## 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.

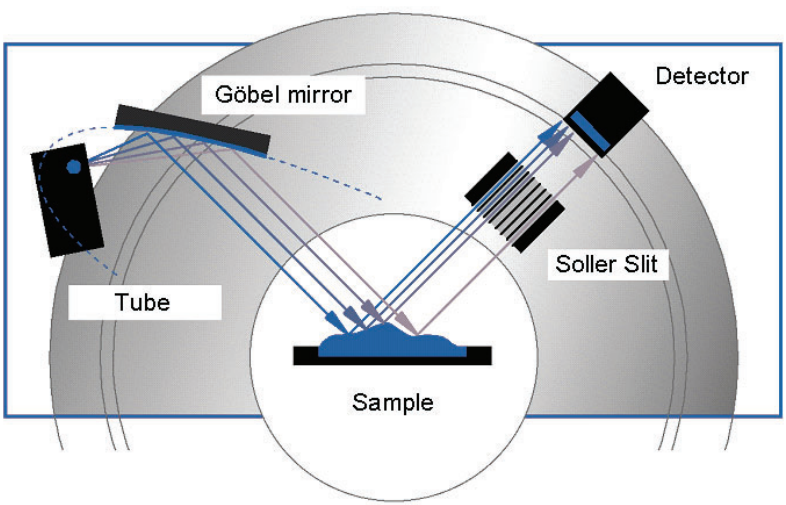



Figure 19: Parallel beam path of diffractometer.

### 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

# The D8 ADVANCE / D8 DISCOVER: Locations and Functions of Basic Components

## Common components of the D8 ADVANCE and D8 DISCOVER

### 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.

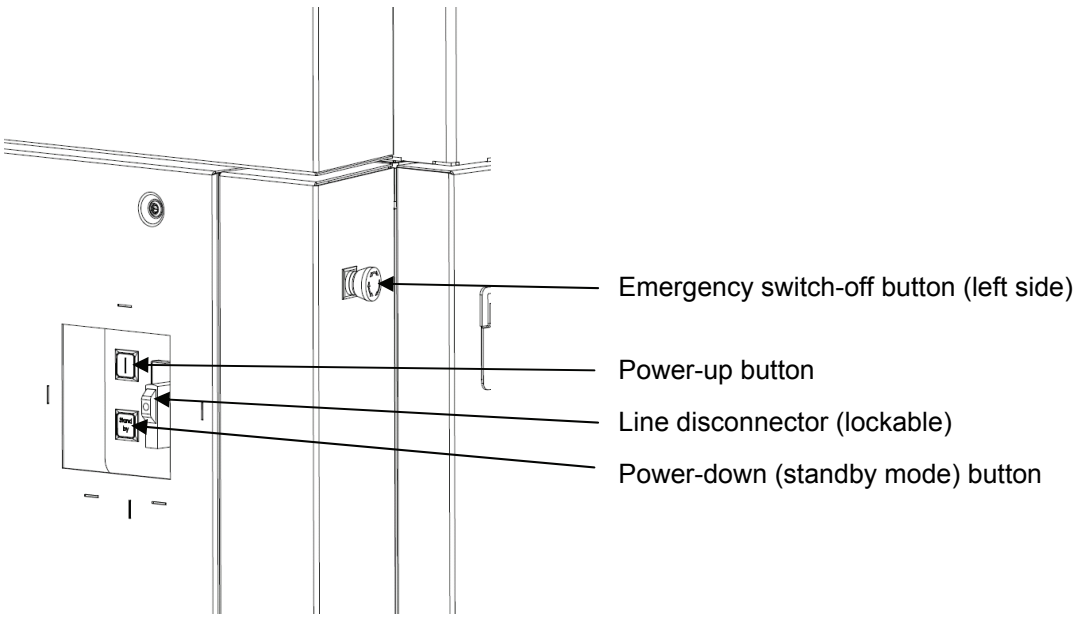
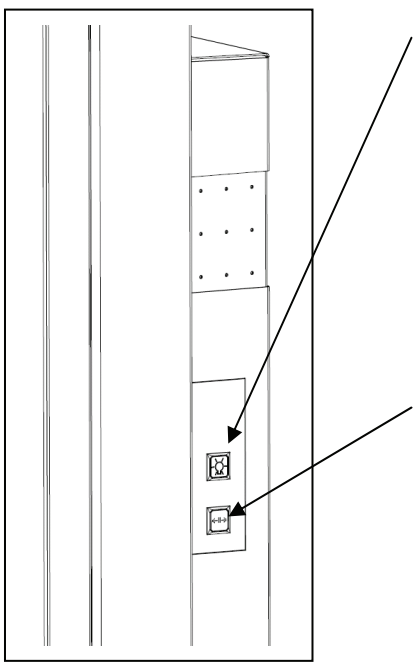



Figure 20: Power switch

# 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.

Figure 21 The enclosure control buttons.

## Generator Screen-key and System Status Screen-key

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

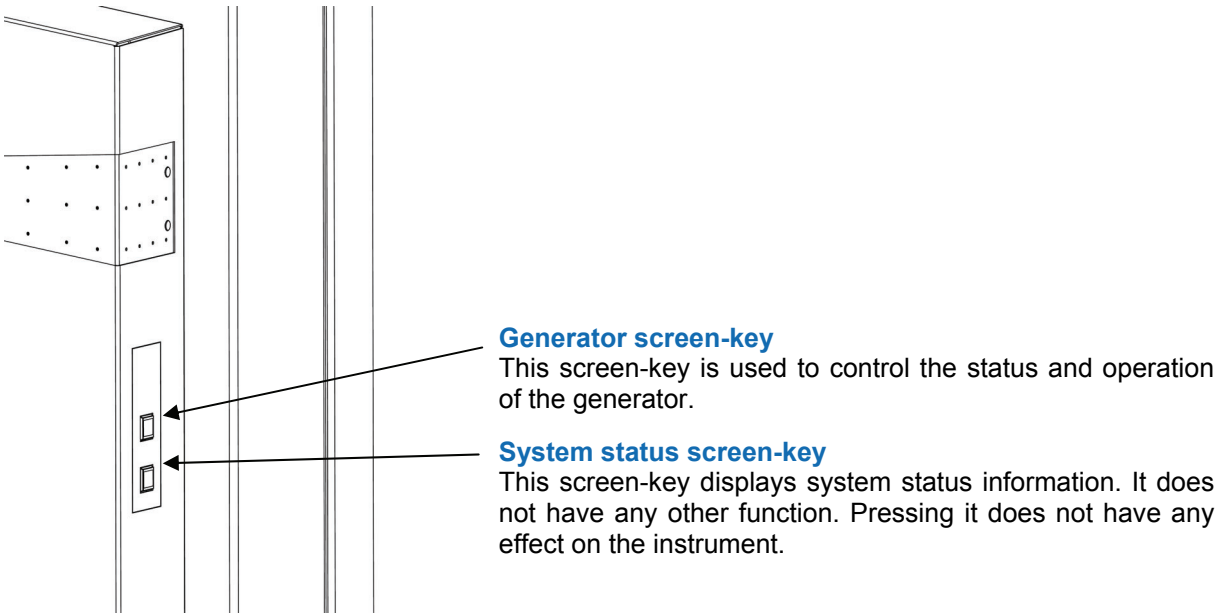



Figure 22: The Generator screen-key and system status screen-key.

The generator screen-key and the system status screen-key have the following states:

For more details on the generator screen-key states please refer to Tab. 1.

For more details on the system status screen-key states please refer to Tab. 2.



**Note**

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 be also be used to diagnose all existing errors and warnings and carry out repairs.

# The Experimental Area

## 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 23).

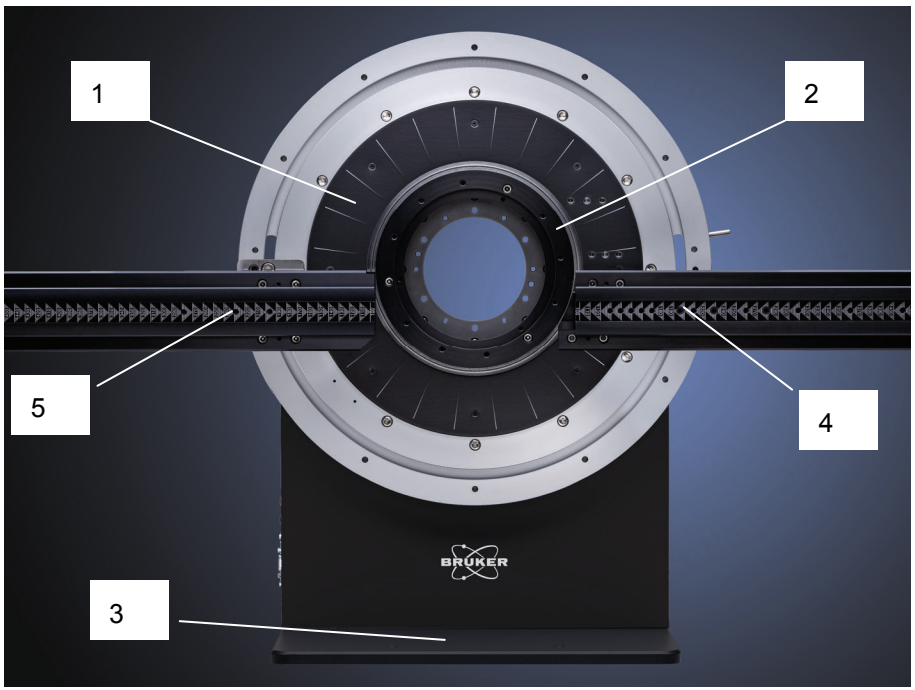



Figure 23: θ-2θ goniometer of the D8 ADVANCE / D8 DISCOVER.

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).

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.

### 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.

## X-Ray Tube

The basic principles of X-ray generation have already been described in the “General Physical Principles” chapter of this manual. Used as standard for X-ray generation in D8 ADVANCE/D8 DISCOVER diffractometers are Siemens X-ray tubes. One of these tubes is shown in Figure 24 below.




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

Figure 24: An example of the type of Siemens X-ray tube used in the D8 ADVANCE/ D8 DISCOVER.

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 104.

Refer to the chapter “Operating the D8 ADVANCE/ D8 DISCOVER” to learn how to switch between point focus and line focus operation.

## 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.

## 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 in Figure 25.

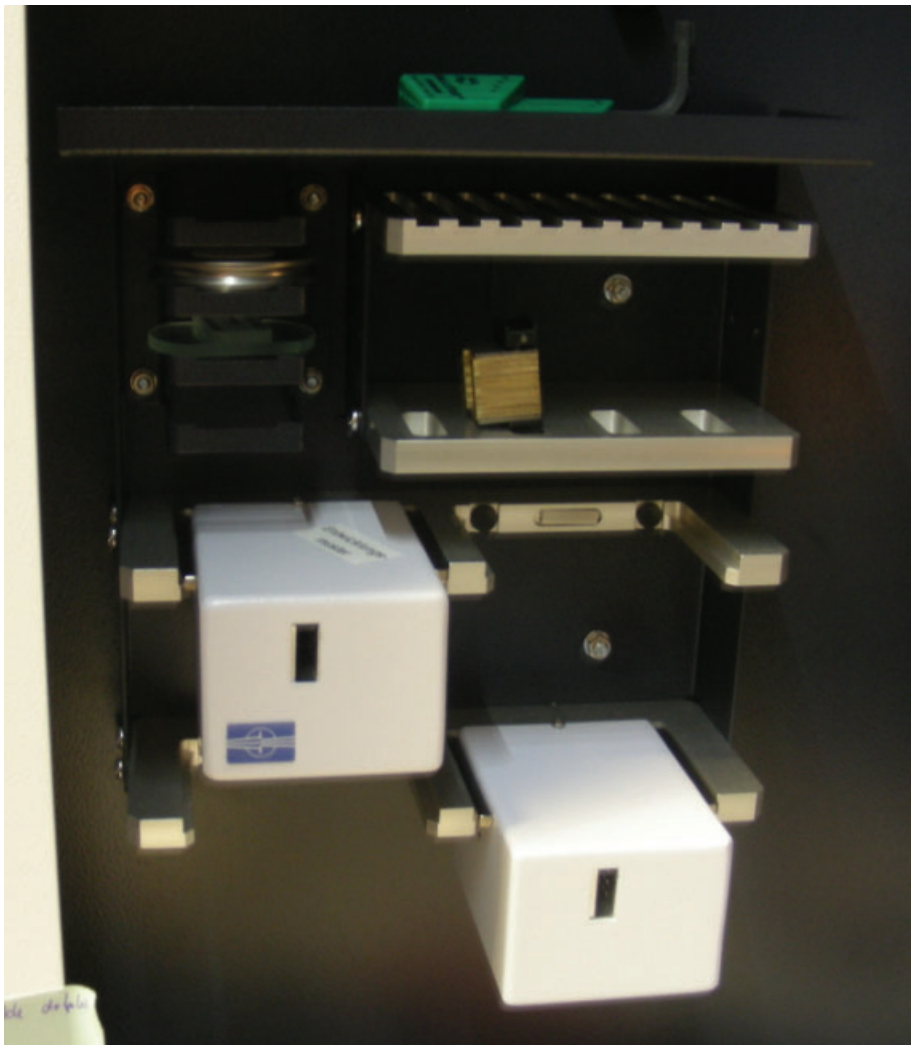


Figure 25: One of the two accessories shelves inside the radiation enclosure.

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

**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 |
| 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 |

# 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.

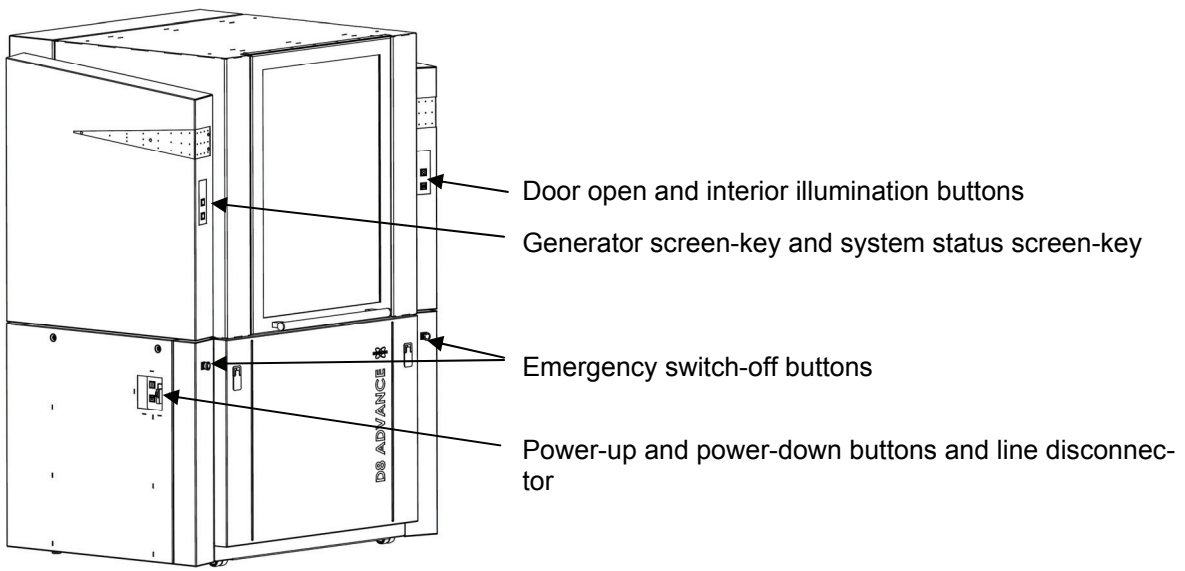



Figure 26: The external housing of the D8 ADVANCE.

# 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.

# Underneath the Experimental Area D8 ADVANCE

## Removing the Panels

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.

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.

The rear panel can be removed only after the left side panel has been removed.

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.

## Lower Front D8 ADVANCE

At the lower front side of the diffractometer are located the following components:

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.

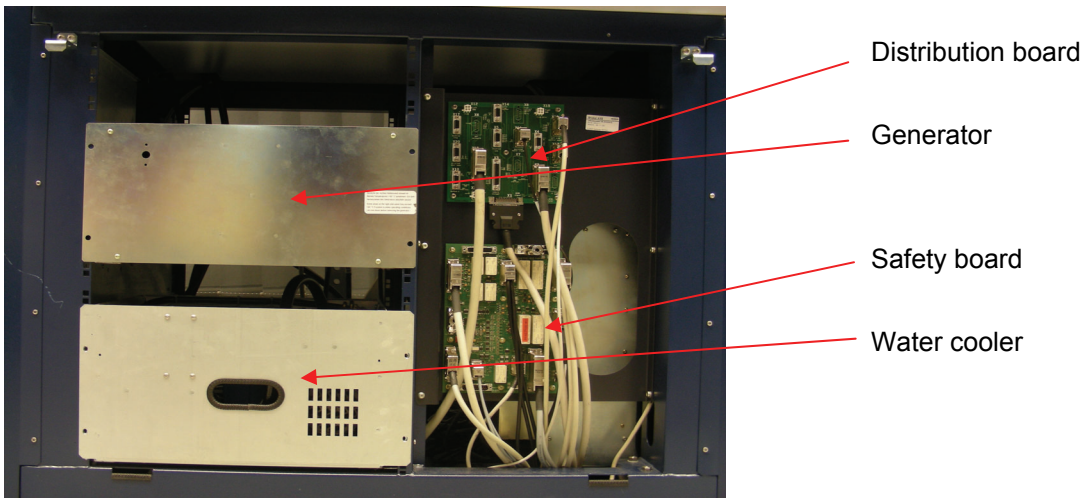



Figure 27: The lower front region, underneath the experimental area.

## Lower Back D8 ADVANCE

At the lower rear side of the instrument are located the following components:

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.

## Lower Right Side D8 ADVANCE

At the lower right side of the instrument are located the following components:

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 Figure 28, two of these sets are occupied by a controller.

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

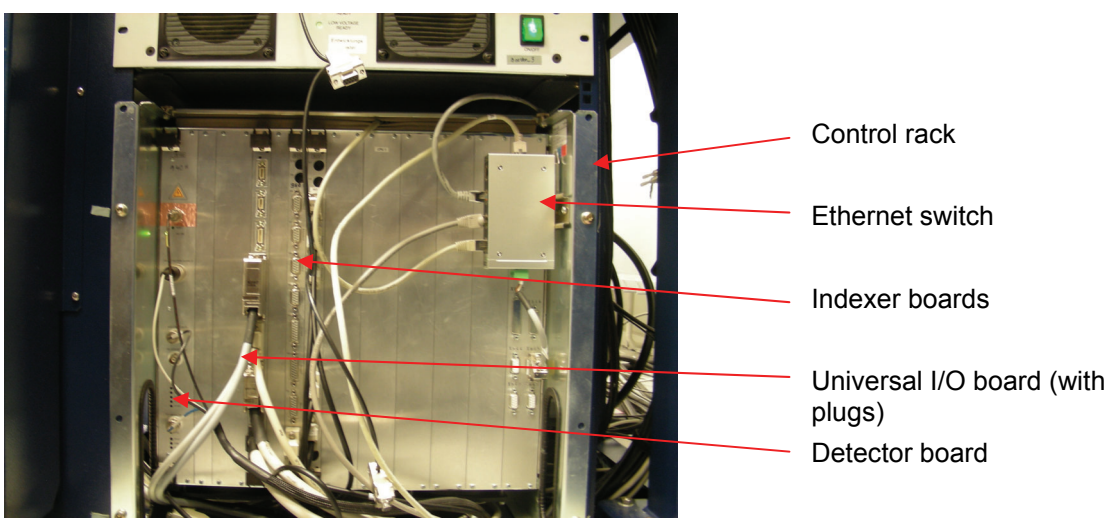



Figure 28: The lower right side of the instrument.

## Lower Left Side D8 ADVANCE

At the lower left side of the instrument are located the following components:

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

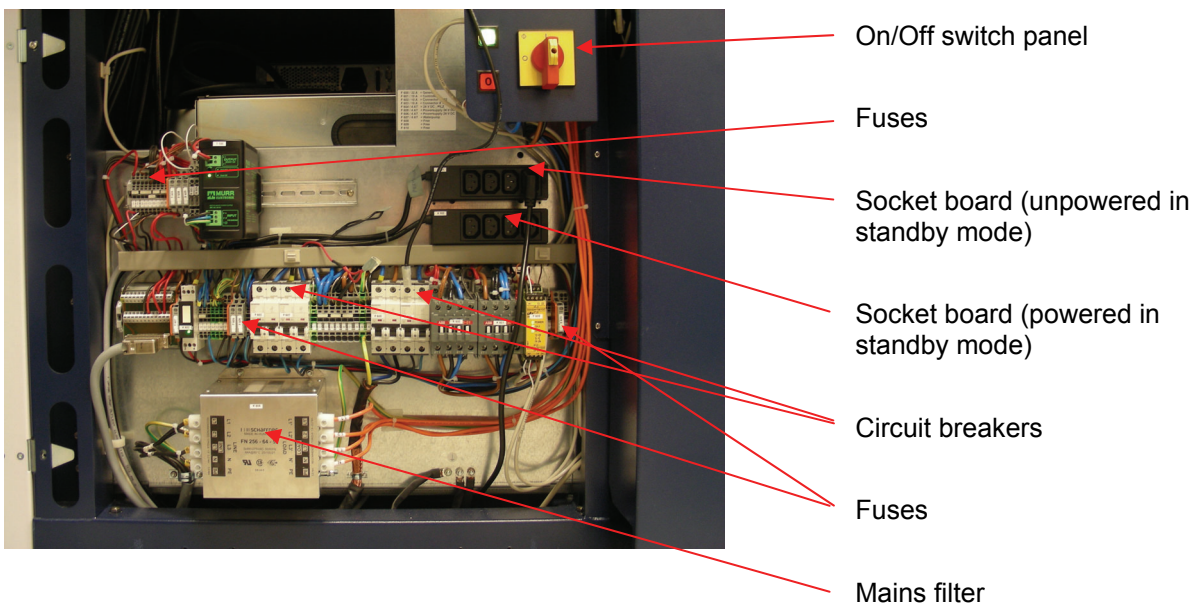



Figure 29: The lower left side of the instrument.

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.

To change a fuse, flip the fuse holder upwards, open its cover, and replace the fuse by the spare fuse stored to the left of it in the holder. 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.

To the right and above the row of terminals, circuit breakers and fuses are located two black socket boards that regulate the flow of current to the various components. 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 switches and the line connector, which are visible at the top right and can be accessed from the outside of the instrument.

The instrument has three electrical states:

**Electrical states**

| State | Power-up Button | Power-down Button | Line Connector | Upper Socket Board | Lower Socket Board |
|---|---|---|---|---|---|
| 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 |

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

**Location of components in diffractomer**

| 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 |

# 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.

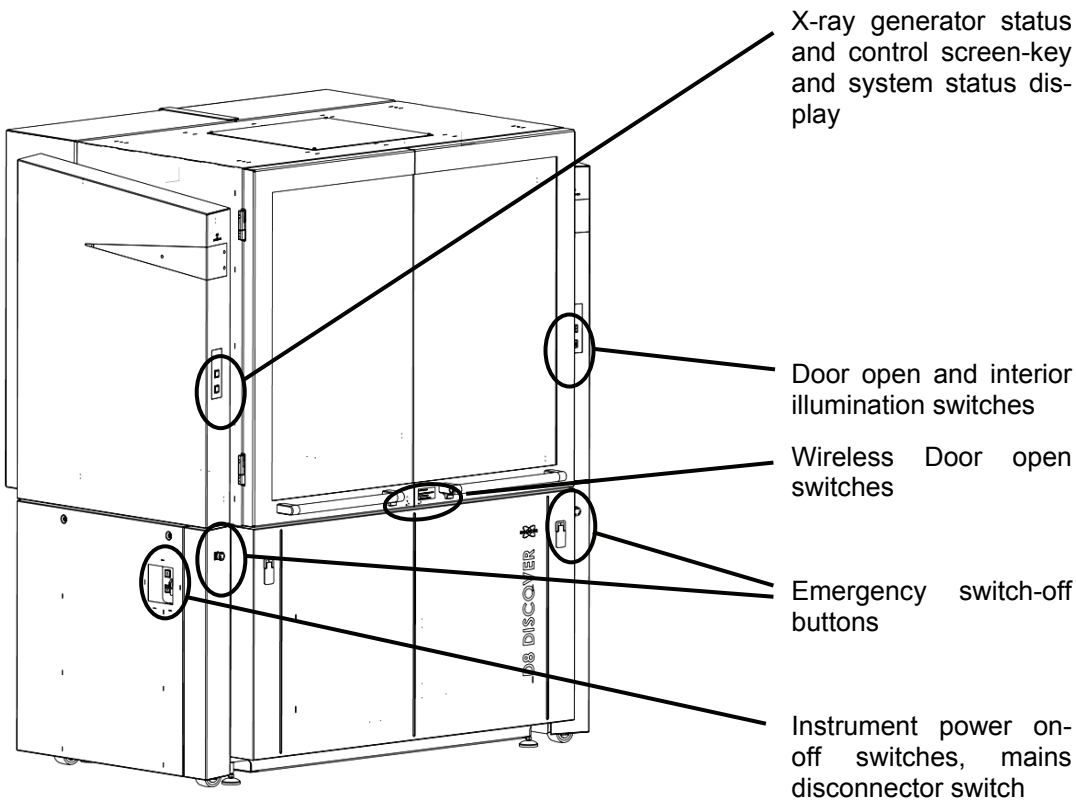



Figure 30: The external housing of the D8 DISCOVER

# 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.

### 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.

### 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.

### 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.

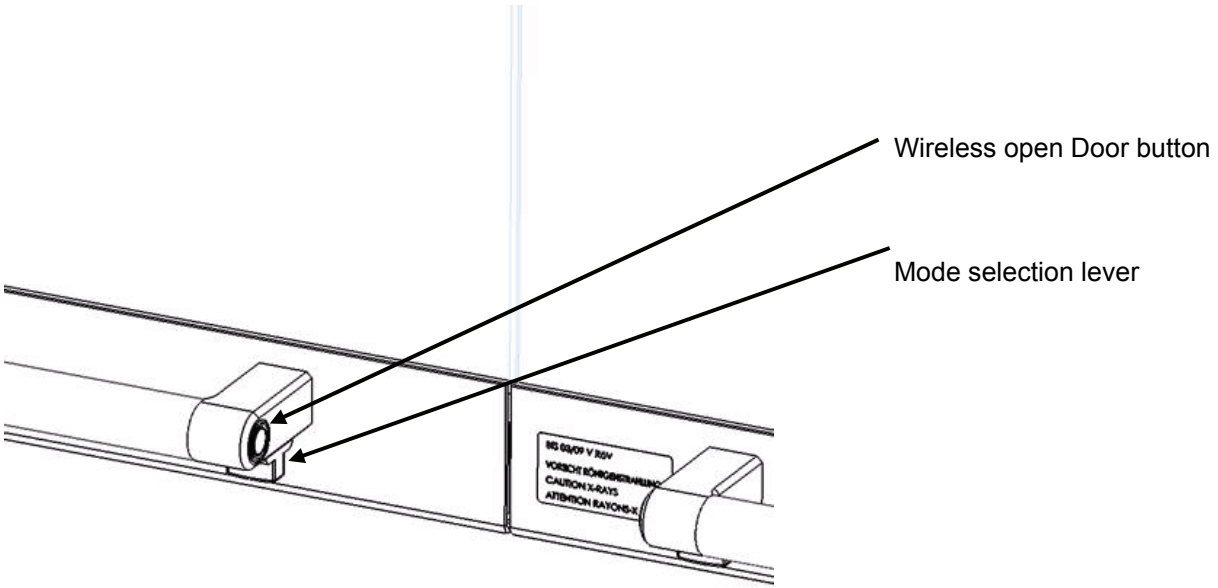



Figure 31: The mode selection lever is used to switch between swing and slide mode of the doors.

# Underneath the Experimental Area D8 DISCOVER

## Removing the Panels

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.

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.

The rear panel can be removed only after the side panels have been removed.

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.

## Lower Front D8 DISCOVER

At the lower front side of the diffractometer the following components are located:

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.

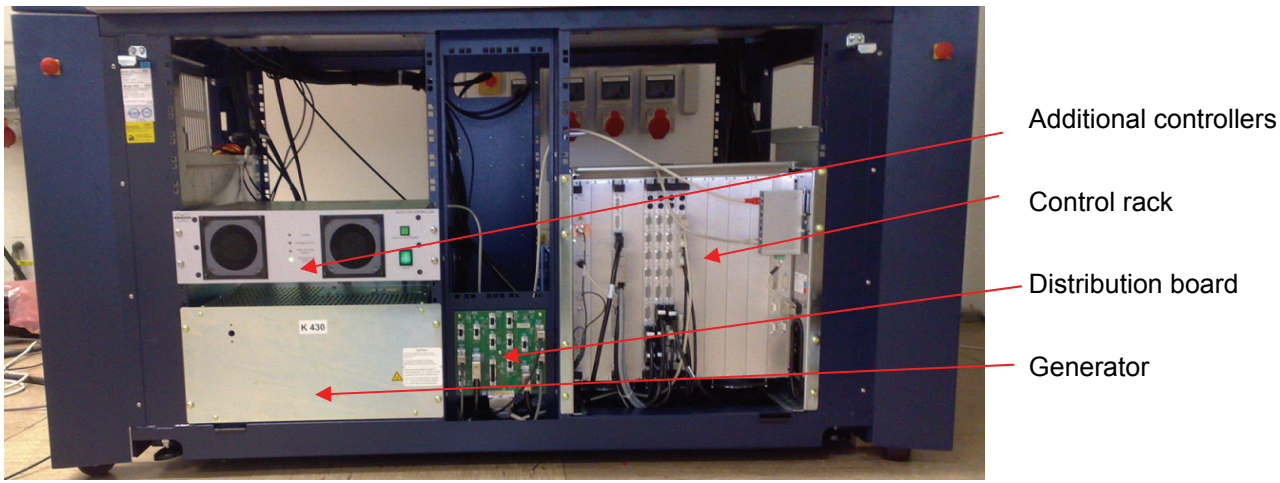



Figure 32: The lower front region of the D8 DISCOVER, underneath the experimental area.

### 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.

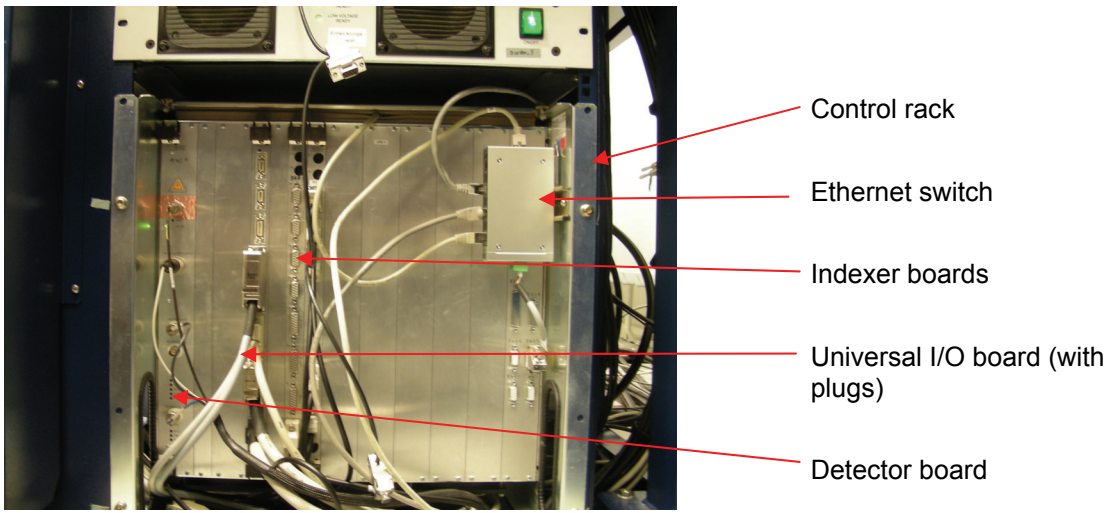



Figure 33: Control Rack.

## Lower Back D8 DISCOVER

At the lower rear side of the instrument are located the following components:

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.

## Lower Left Side D8 DISCOVER

At the lower left side of the instrument are located the following components:

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

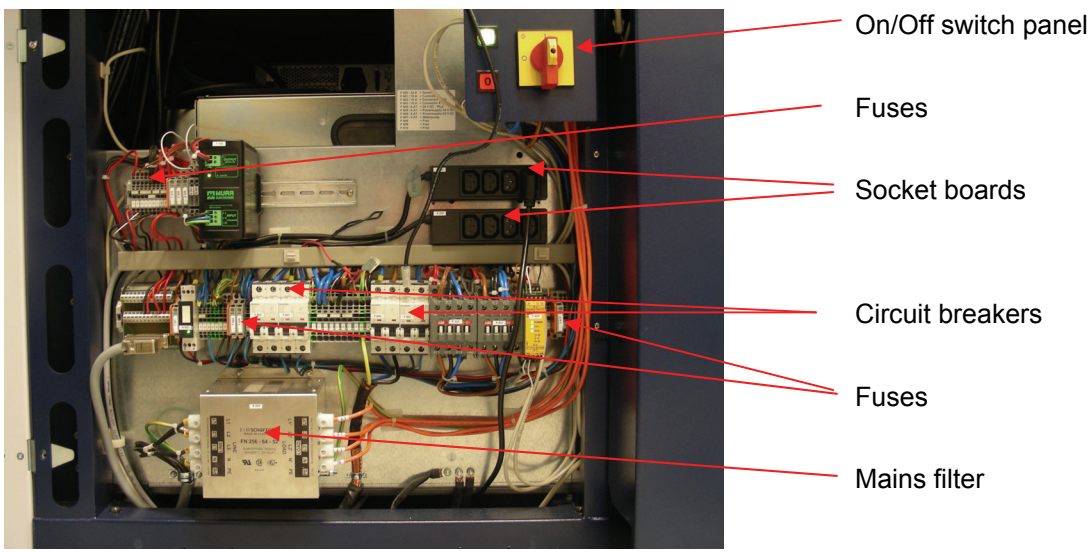



Figure 34: The lower left side of the instrument.

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.

To change a fuse, flip the fuse holder upwards, open its cover, and replace the fuse by the spare fuse stored to the left of it in the holder. 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.

To the right and above the row of terminals, circuit breakers and fuses are located two black socket boards that regulate the flow of current to the various components. 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 switches and the line connector, which are visible at the top right and can be accessed from the outside of the instrument.

The instrument has three electrical states:

**Electrical states**

| State | Power-up Button | Power-down Button | Line Connector | Upper Socket Board | Lower Socket Board |
|---|---|---|---|---|---|
| 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 |

## 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.

# The D8 ADVANCE and D8 DISCOVER: Descriptive Listings of Common Components

## 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 in Figure 35 for the primary side. The functions of the elements shown in Figure 35 are summarized in Tab. 3.

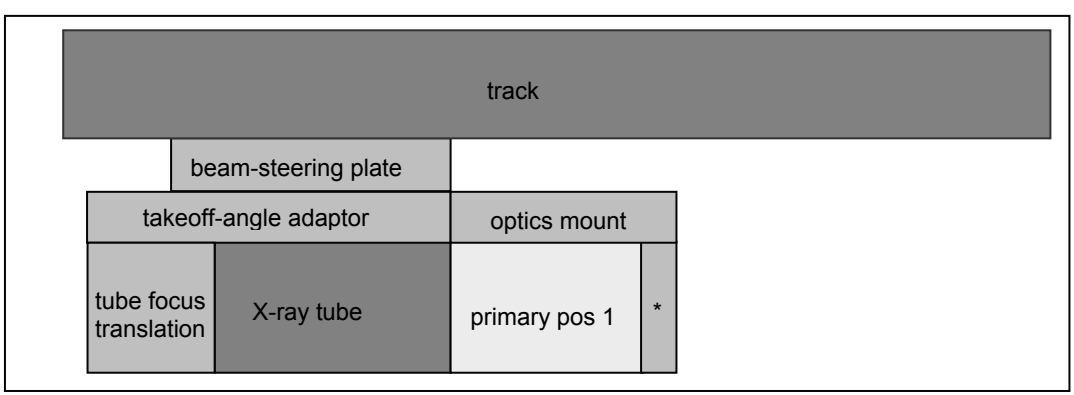



Figure 35: A schematic diagram for mounting components to the primary track. The component labelled with an asterisk * is the safety slit. The optics mount is also called “optical bench”.

**Note**

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.

Tab. 3 summarizes the functions of the primary side adjustments:

Tab. 3: 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. |

## 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.

# 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. Figure 36) 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

## Rotation Beam-Steering Plate

The rotation beam-steering plate is shown in Figure 36 and in Figure 37.

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…540 mm (diameter 280…1080 mm) depending on configuration |

| | |
|---|---|
| D8 ADVANCE | 140…360 mm (diameter 280…720mm) depending on configuration |
| Measurement height: | |
| D8 DISCOVER | 150mm, 214mm, 258 mm depending on configuration |
| D8 ADVANCE | 150mm |

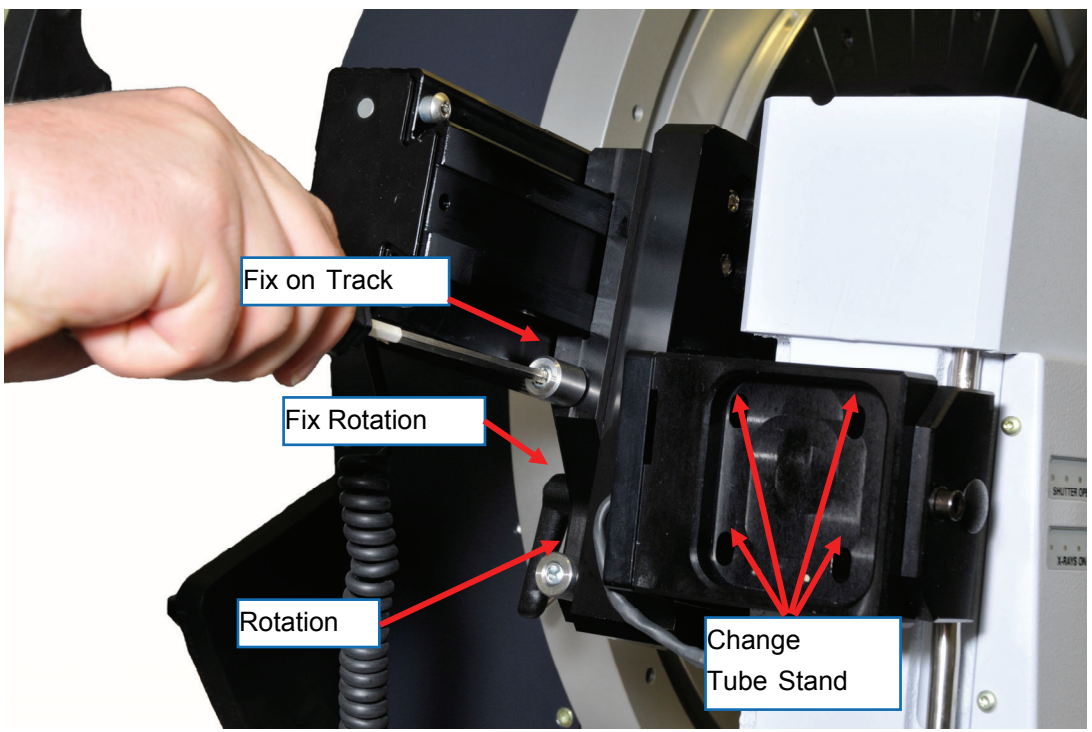



Figure 36: The rotation beam-steering plate: front side.

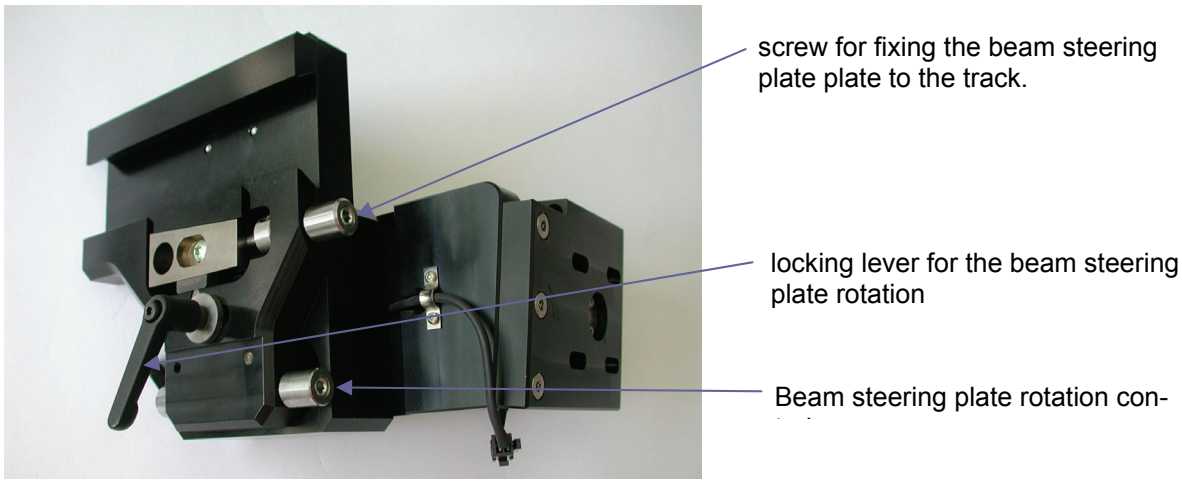



Figure 37: The rotation beam-steering plate: front side.

## Rotation + Translation Beam-Steering Plate

The rotation and translation beam-steering plate is shown in Figure 38. Figure 38 shows the manual version. A motorized translation is available.

**Specifications:**

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

| | |
|---|---|
| D8 ADVANCE | 140…360 mm (diameter 280 … 720 mm) depending on configuration |
| Measurement height: | |
| D8 DISCOVER | 150 mm, 214 mm, 258 mm depending on configuration |
| D8 ADVANCE | 150 mm |

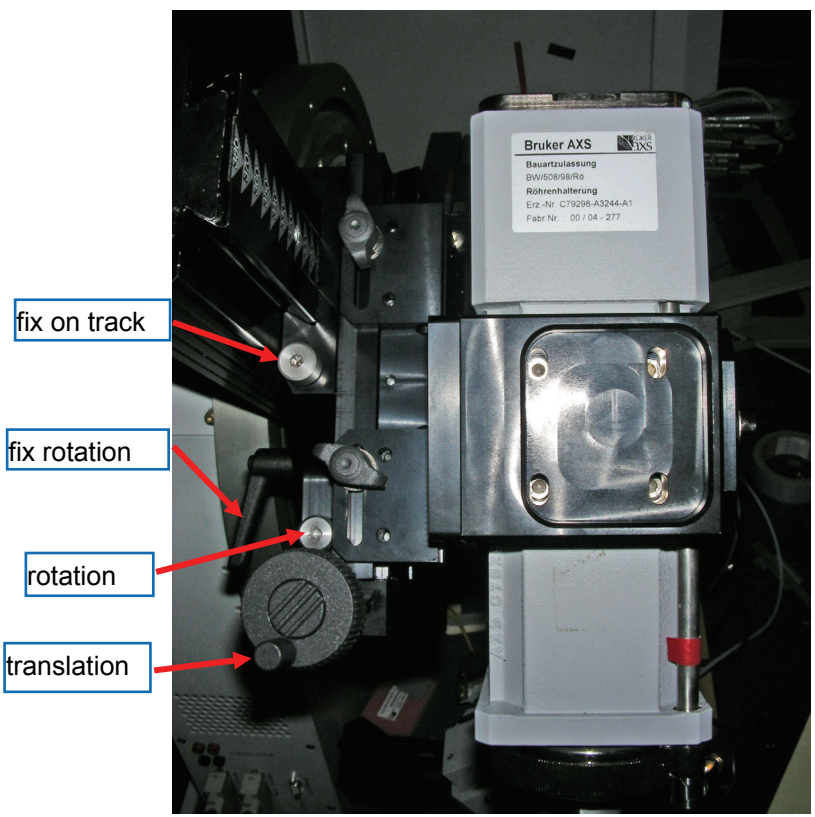



Figure 38: The rotation and translation beem-steering plate (non-motorized version).

## 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° (Figure 39). 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.
- Remove tube stand. Figure 36 shows the four screws which have to be loosened.
- Remove adaptor plate, four screws in Figure 39.
- Install new adaptor plate. Pins take care of good position. Interlocks check correct installation.
- Install tube stand. Pins take care of good position. Interlocks check correct installation.
- 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.

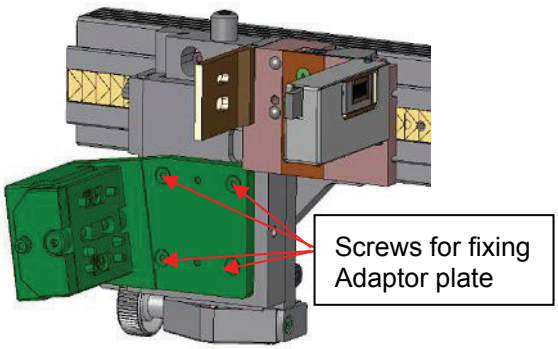



Figure 39: Standard takeoff angle adaptor plate (-7.5°). Used for Bragg-Brentano, Göbel mirror, TWIN optics, POLYCAP: all optics with primary beam deflection close to zero.

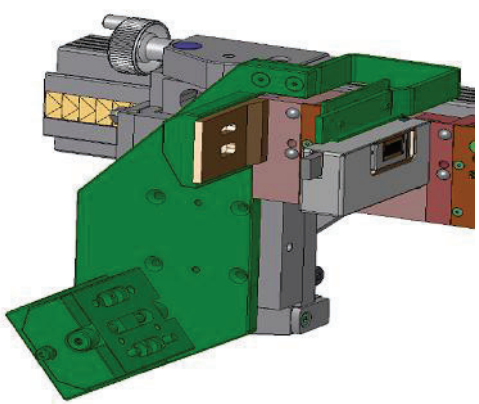


Figure 40: Takeoff angle adaptor plate with large negative primary beam deflection angle (-33°). Used for Johansson monochromator. The adapter plate includes a primary beam stop for radiation safety.

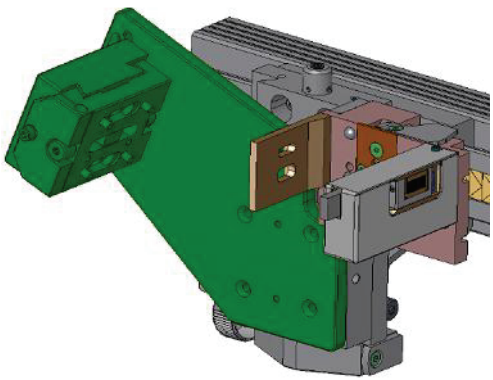


Figure 41: Takeoff angle adaptor plate with large positive primary beam deflection angle (+20°).

# Secondary Side: Optics and the Universal Detector Mount

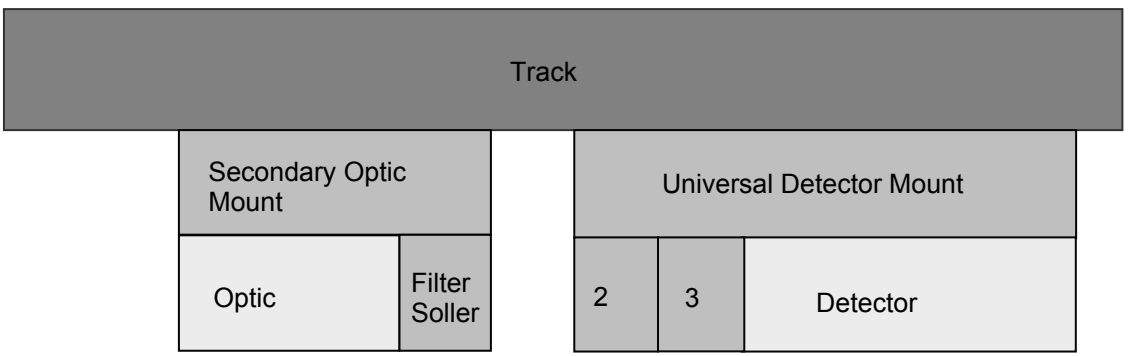



Figure 42 A schematic diagram for mounting components to the secondary track. Typically, position 2 is used for a detector slit and position 3 is used for the rotary absorber. The Universal Detector Mount exists in different versions: Position 2 and 3 are optional. Detectors compatible with the Universal Detector Mount are: LYNX-EYE, SOL-XE, Scintillation counter.

# 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 in Figure 43, or an embedded chip, as shown in Figure 44. The holes array is used for filters and slits, and the chips for all other components.

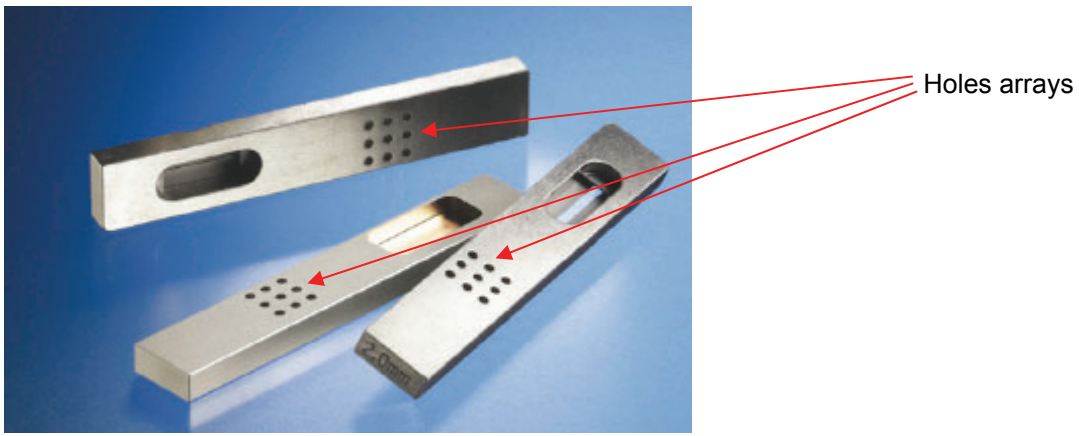



Figure 43: Slits with holes arrays, used for automatic component recognition.

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.

## 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 in Figure 44.

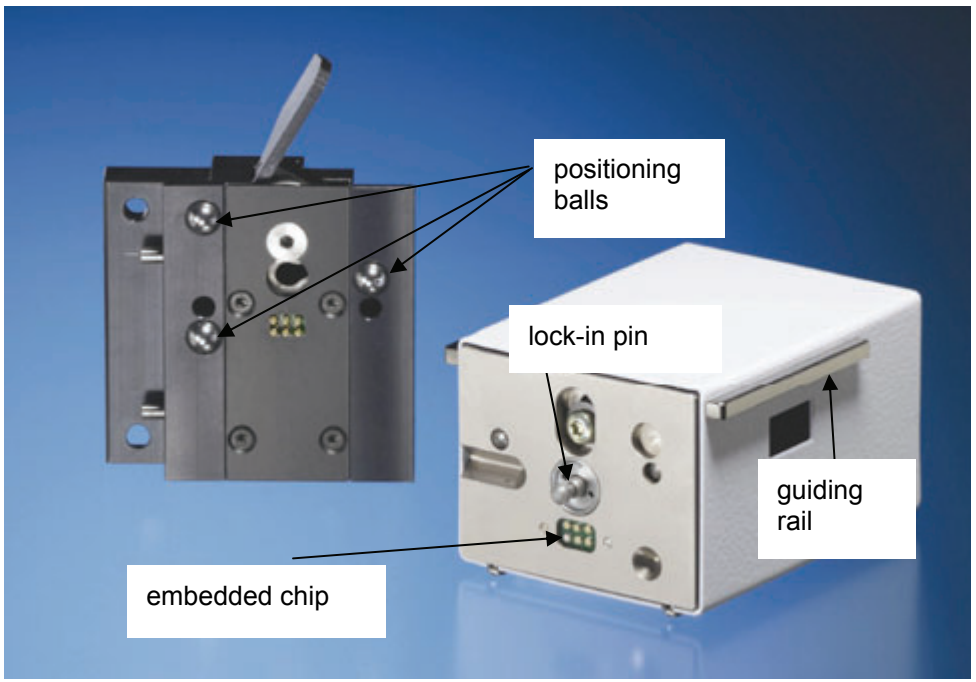



Figure 44: The SNAP-LOCK and automatic component recognition elements, as shown on an optics mount and optics housing.

# 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.

# Primary Optics

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

## 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.

## 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.

### 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.

## Monochromators

The focusing primary monochromator of Johansson type generates $K\alpha_1$-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 K$\alpha_1$ 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 K$\alpha_1$ 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.

## 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:

- Check if takeoff angle adaptor plate is correct (-33° plate). Install correct plate if necessary.
- Move beam steering plate to correct distance. Standard measurement circle diameter is 435mm. 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.

### Reflection setup

See Figure 47, Figure 48, Figure 50: Distance between tube focus and primary focus slit must be

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

**Practically:** Measurement circle diameter for most sample stages is 435mm. Therefore, put detector slit, respectively 1-D detector to 435 mm diameter. Place primary focus assembly to 435 mm diameter. Install flight tube in front of primary focus assembly and shift tube against it. Move tube against flight tube. 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

### Transmission setup

See Figure 45 and Figure 46: Distance between tube focus and detector must be primary focal length (a) + secondary focal length (b) of the monochromator. Default value is

$\sqrt{a^2 + b^2 - 2ab\cos(180° - 2\theta_{Johansson})} \approx 470mm$ ± 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 1-D detector.

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

- detector at 435 mm measurement circle diameter
- tube focus 470 mm away from detector at theta=2theta=0°

### Check monochromator intensity

- 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.
- Steer beam through goniometer centre and redefine optics offset theta. Use standard procedure with glass slit.
- Redefine optics offset twotheta
- 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.

## Details on Primary Monochromator in Transmission and Capillary Setup

Figure 45 and Figure 46 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.

Tab. 4: Specification data of Johanssan monochromator for D8 ADVANCE/D8 DISCOVER - transmission and capillary mode.

| **Radiation** | **CuK$_{\alpha 1}$** |
|---|---|
| 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 $\alpha$ | 6.722° |
| Exit angle $\beta$ | 20.559° |
| $2\theta_{Johansson}$ | 27.281° |

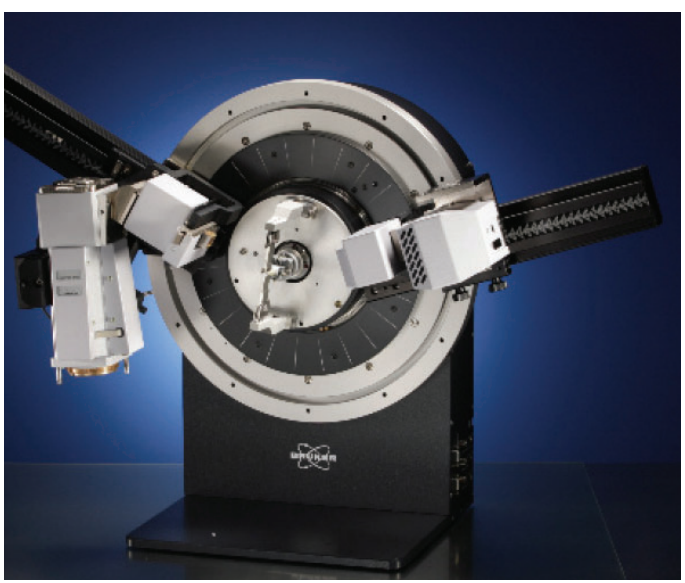
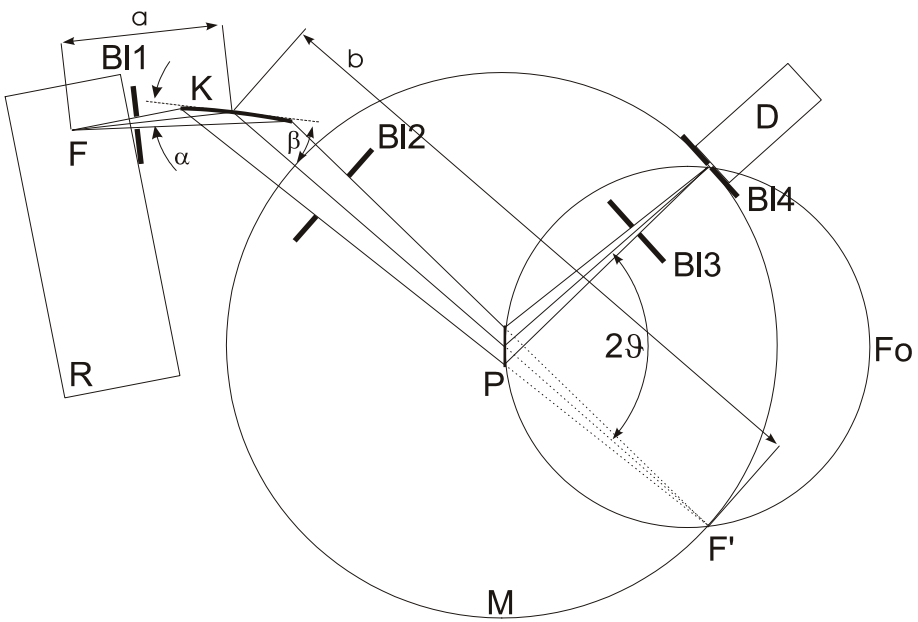


Figure 45: Johansson monochromator for transmission/capillary mode.




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

Figure 46: Beam path for Johansson monochromator in transmission geometry.

## Details on Primary Monochromator in Reflection Setup

Figure 50 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 48).

Tab. 5: Specifications for Johansson monochromator - reflection setup.

| **Radiation** | **$Cu_{K\alpha 1}$** |
|---|---|
| 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 $\alpha$ | 9.337° |
| Exit angle $\beta$ | 17.304° |
| $2\theta_{Johansson}$ | 26.641° |




Figure 47: Johansson monochromator for reflection mode.

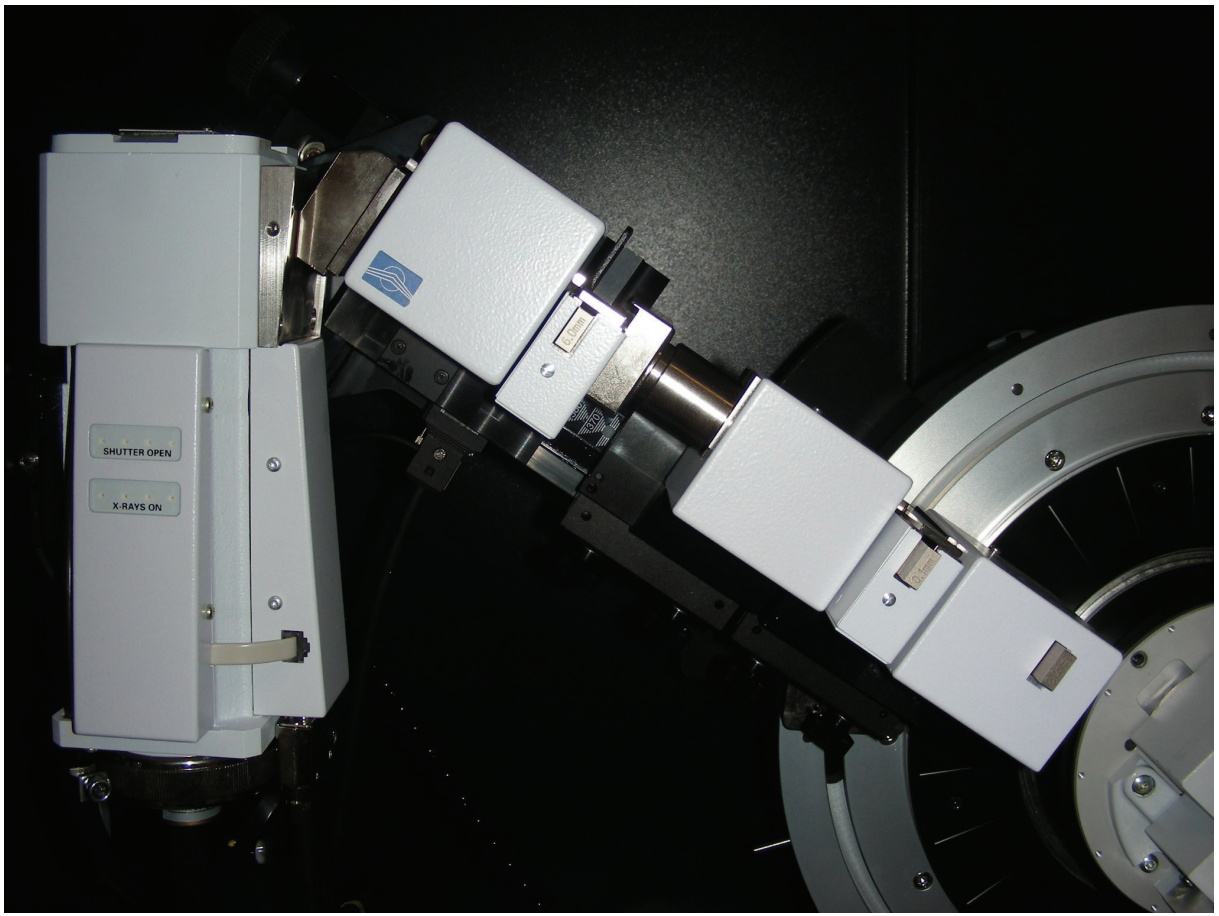



Figure 48: Johansson monochromator for reflection mode.

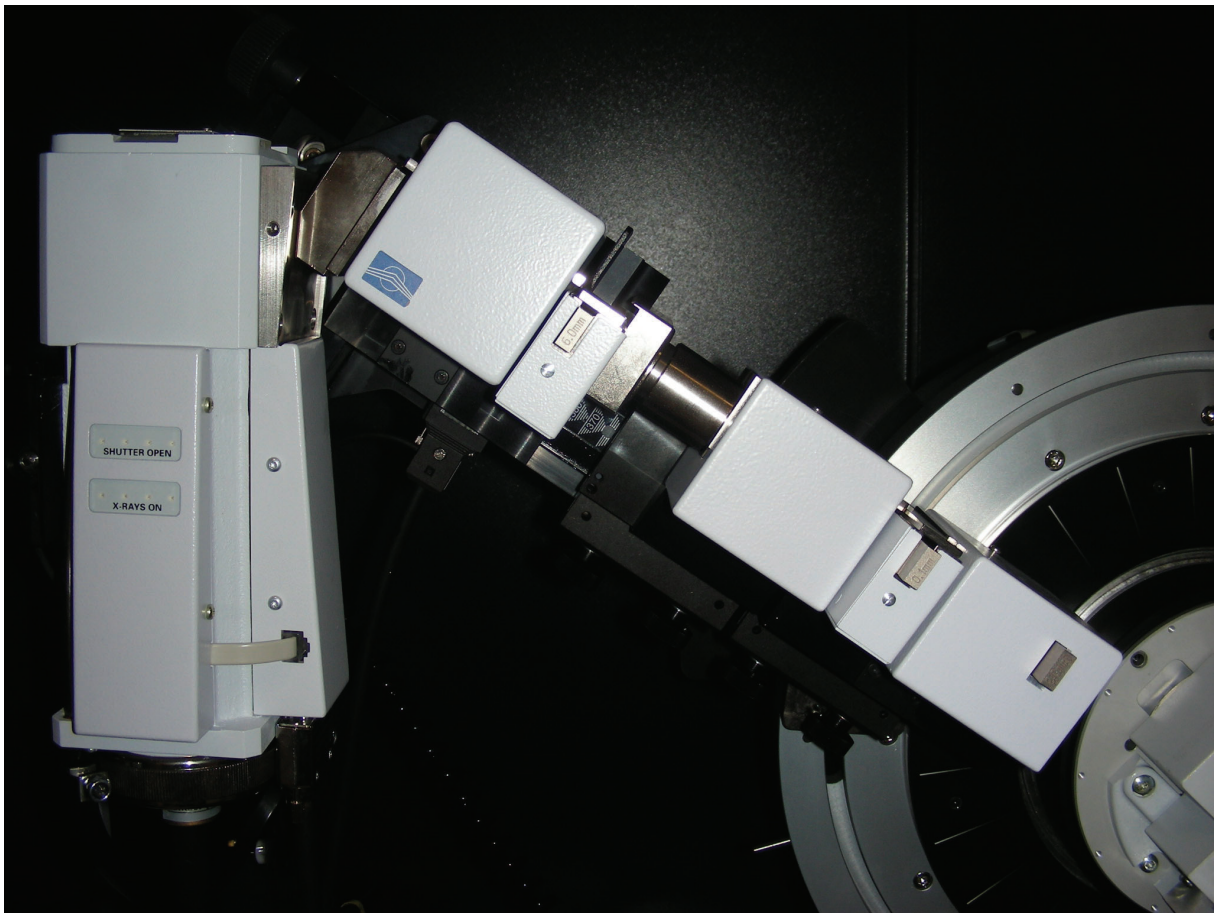
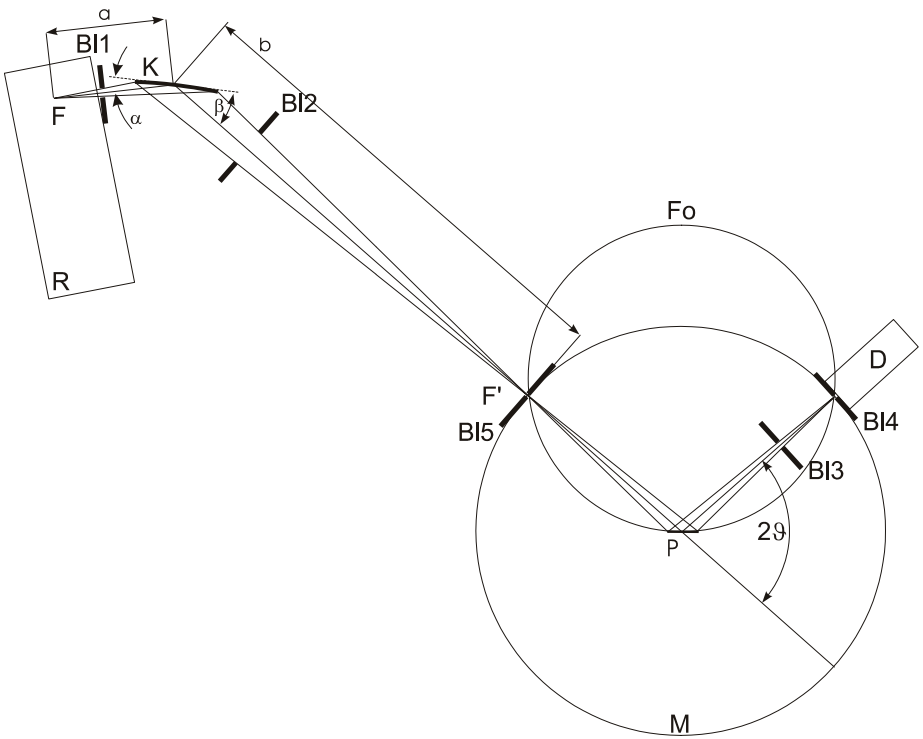



Figure 49: Johansson monochromator with reflection setup including guiding tubes, focussing slit, and Bragg-Brentano divergence slit assembly.




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

Figure 50: Beam path for reflection measurements

### Adjustment of the Focus Slit

- Move beam steering plate and optical bench away from the goniometer center, so that you can mount the focus slit holder and antiscatter tube.
- Mount Focus slit holder at a measurement circle radius of 217.5 mm
- Mount antiscatter tube. The sample side ending of the antiscatter tube should be flush with the focus slit holder.
- Move beam steering plate so that the ration safety slit is flush with the tube side ending of the antiscatter tube.
- Mount fluorescent screen as sample.
- Set generator to 40kV and 40 mA.
- Do not mount focus slit or divergence slit.
- 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.
- 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.
- Use 6 mm detector slit and if necessary to prevent saturation 0.1 mm or 0.2 mm Cu absorber.
- 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.
- As a rule of thumb, through a 100 µm focus slit intensity lose of less than 10% should occurr compared to 1 mm focus slit.

### Adjustment of the Antiscatter Tube

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

# Secondary Optics

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

## 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 parallely aligned tungsten blades. They are used for parallel beam measurements.

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

## Diffracted Beam Monochromator

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

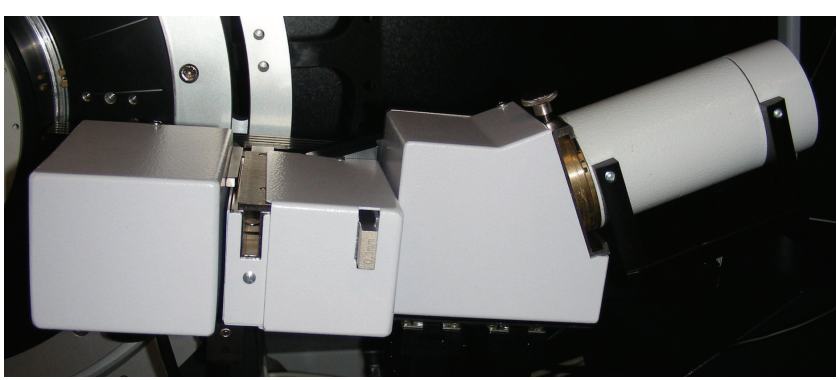


Figure 51: Diffracted-Beam Monochromator. Diffracted beam path consists of variable slit, fixed detector slit, graphite monochromator for CuK$\alpha$ radiation, and scintillation detector.

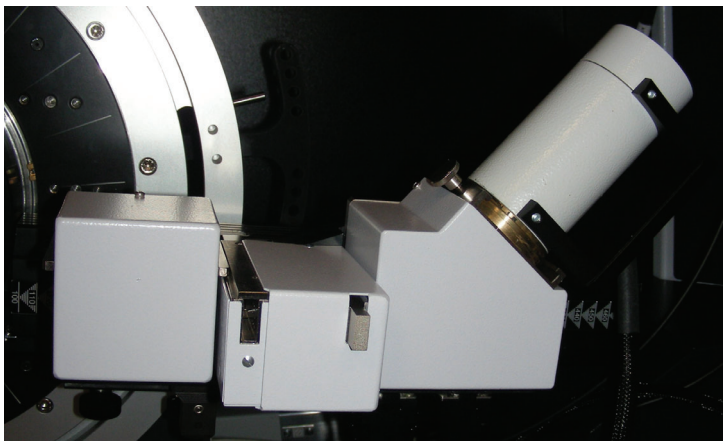


Figure 52: Diffracted-Beam Monochromator for Parallel beam applications. Diffracted beam path consists of Soller slit, variable slit, slit for limiting diffracted beam width, flat LiF monochromator for CuK$\alpha$ radiation, and scintillation detector.

## 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 K$\beta$ radiation.

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

## 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.

Figure 53 shows the beam path with sample and diffracted-beam monochromator in detail.

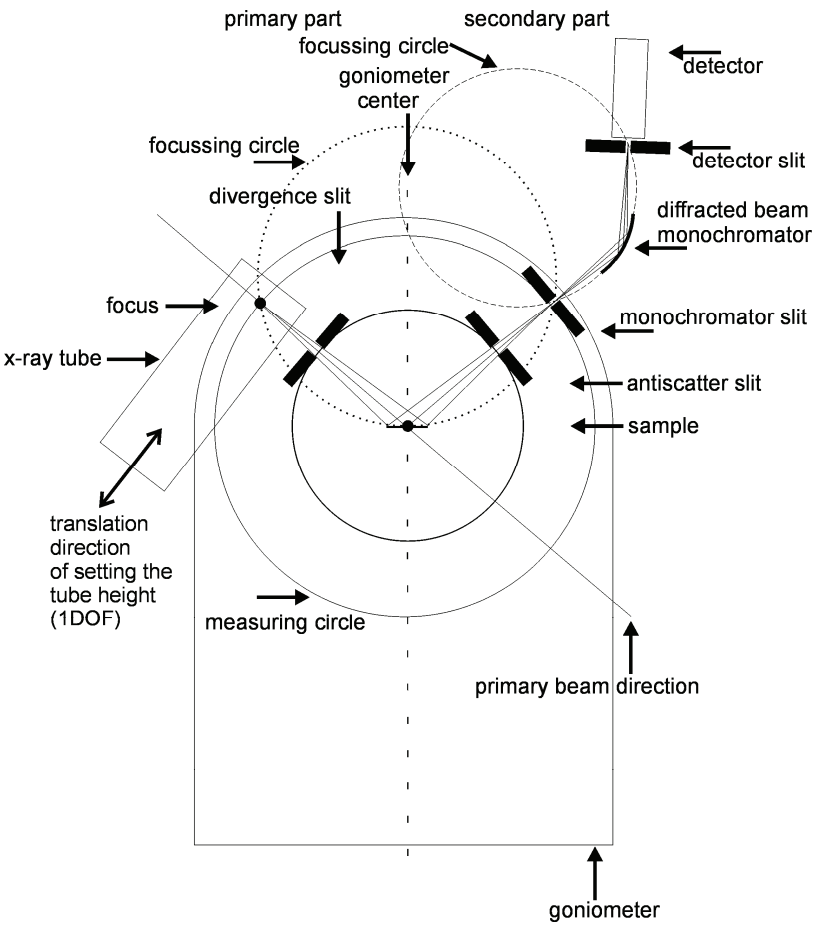



Figure 53: Schematic beam path of the D8 with diffracted-beam monochromator.

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.

Tab. 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 |

## 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°

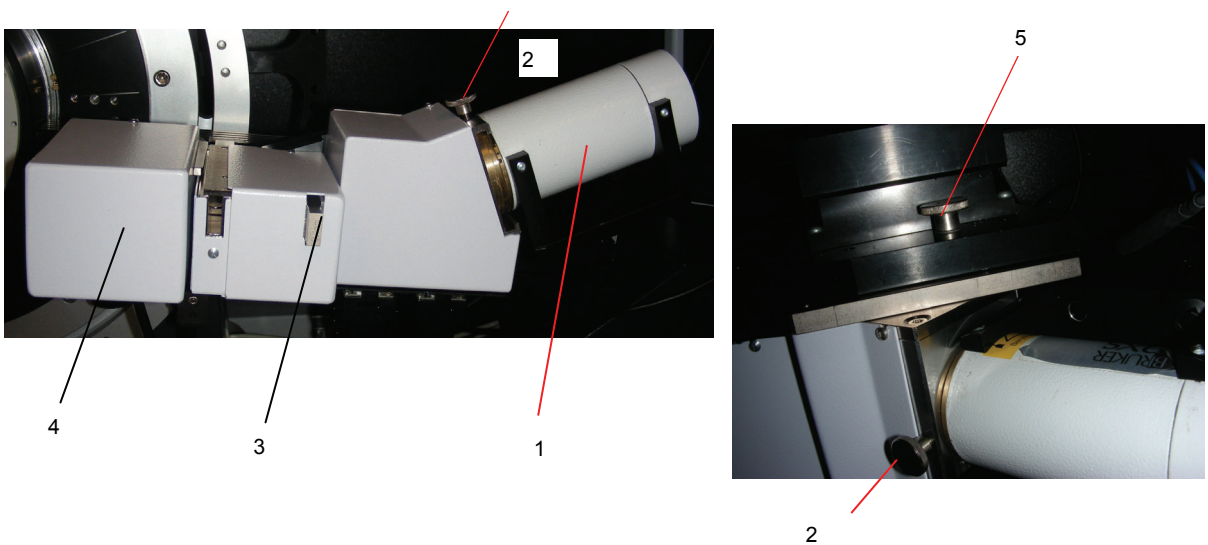
Tab. 7: Wavelenghts as used in the DIFFRAC.SUITE

| 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 |

## Installation

The assembly is shown in Figure 54. Loosen locking screw (5) on the backside of universal detector mount.

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




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

Figure 54: Secondary monochromator.

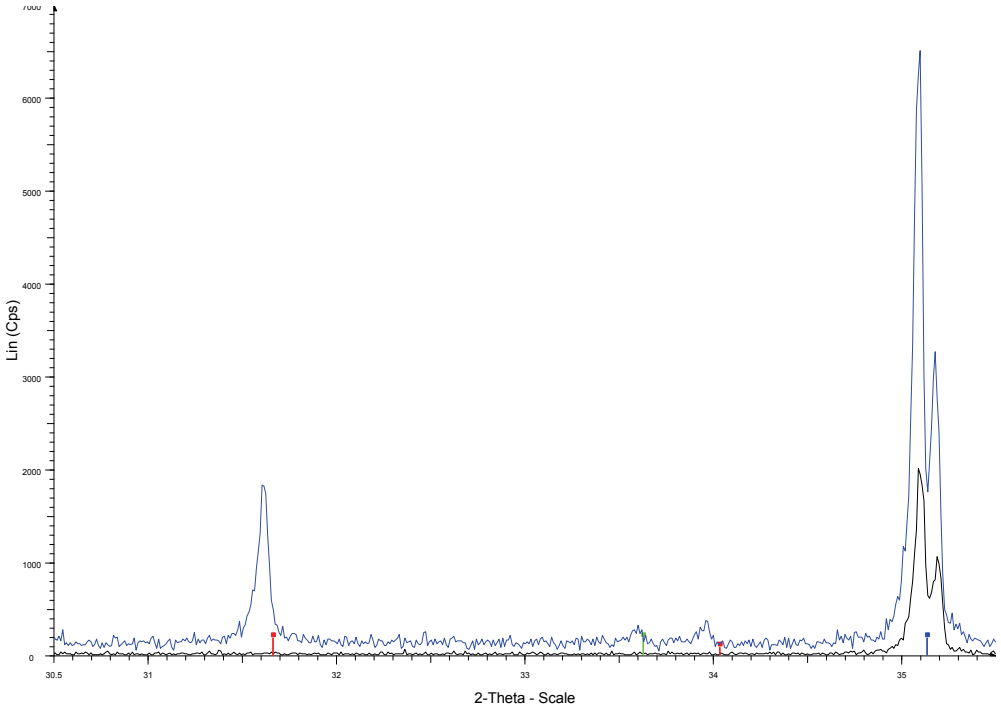



Figure 55: SRM 1976A measured with (black) and without (blue) graphite secondary monochromator.

## 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.

## Operation

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

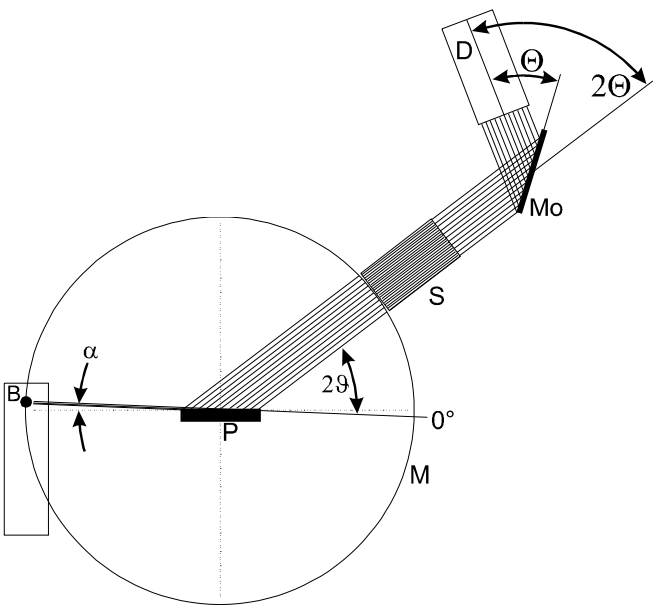



Figure 56: Monochromator in parallel beam setup with grazing incidence.

## Technical Data

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

# 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

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 57: Plugin slits, filters, absorbers.

## 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

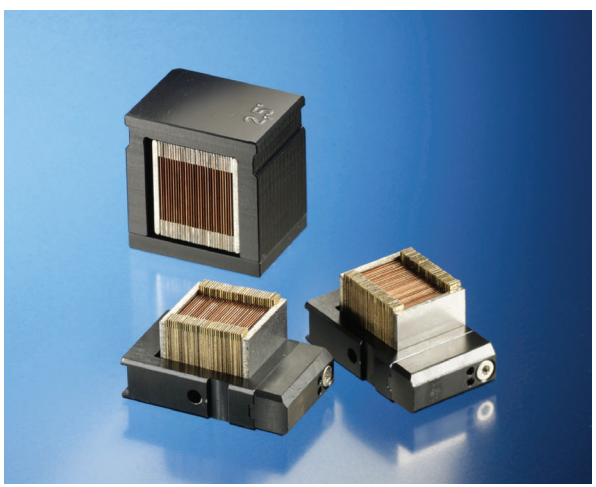


Figure 58: Axial Soller slits.

## UBC collimator

UBC collimators can be mounted at end of 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 with the data on the chip placed in the UBC collimator.

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

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



Figure 59: UBC collimator.

## Air Scatter Screen

Especially at small incidence angles and when using 1-D detectors like LYNXEYE and VÅNTEC-1 X-rays scattered by air create high background in the data. The air scatter screen takes care for reducing this 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.

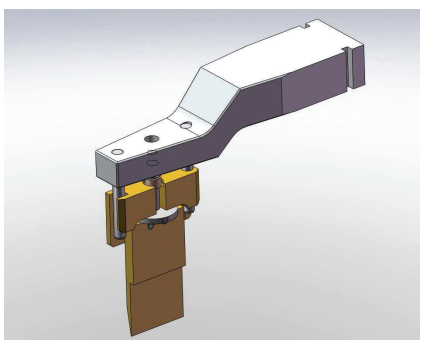


Figure 60: Air scatter screen.

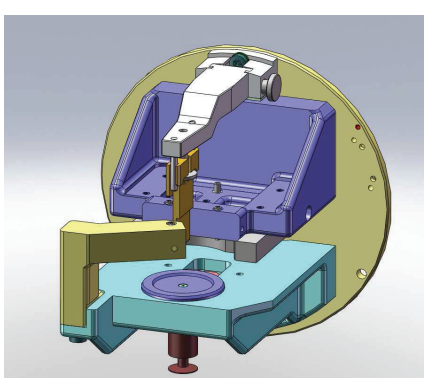


Figure 61: Air scatter screen mounted on standard sample stage.

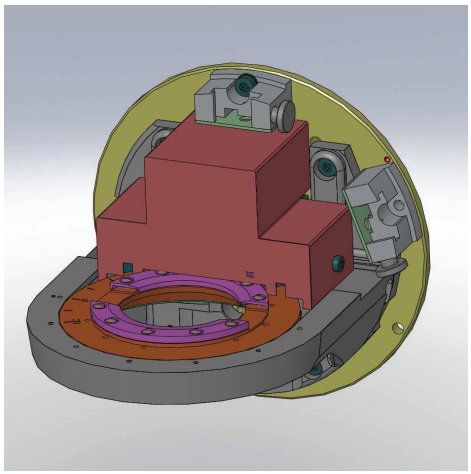


Figure 62: The Rotary sample stage provides two insertion positions for the air screen. One is used for reflection measurements and the second in case of transmission.

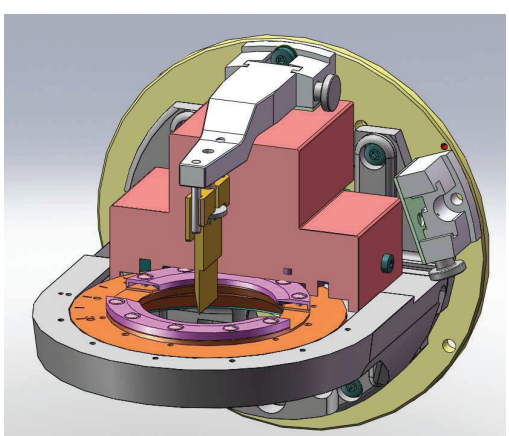


Figure 63: Rotary sample stage setup for reflection.

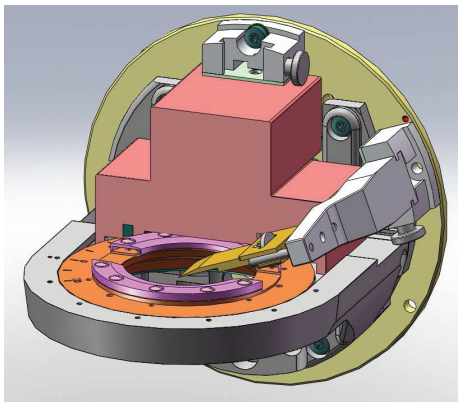


Figure 64: Rotary sample stage setup for transmission.

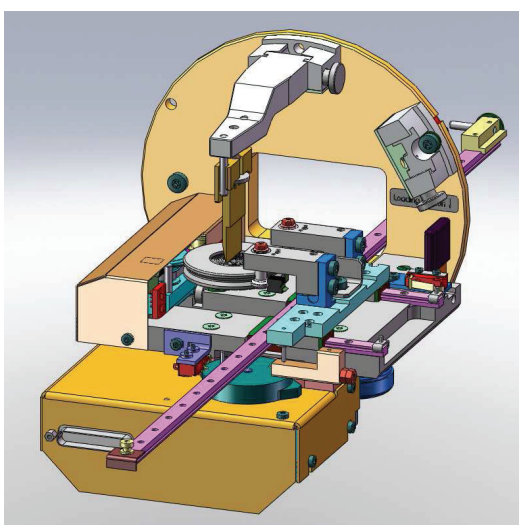


Figure 65: FLIP-STICK setup for reflection.

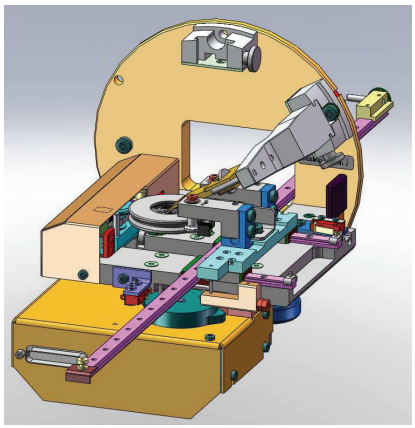


Figure 66: FLIP-STICK setup for transmission.

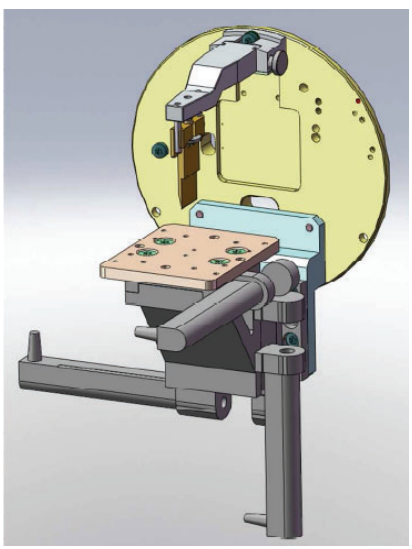


Figure 67: Air scatter screen mounted on compact XYZ stage.

# Stages and Sample Holders

## Standard Sample Stage

The standard sample stage (Figure 68) 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.



**Caution!**

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

## Insertion of Samples

With help of a quick-release lock the sample is pressed in the standard sample stage (Figure 69). 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.

Samples up to a thickness of 20 mm may be inserted. An insertion aid (Figure 70) is available for samples of 50 x 50 mm and with 50 or 60 mm diameter.

The pressure disk (Figure 70) 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 (Figure 69). The insertion aid (Figure 70) must be removed in this case.

The front stop bracket (Figure 69) 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 72 shows these two possible installations for the standard sample stage.

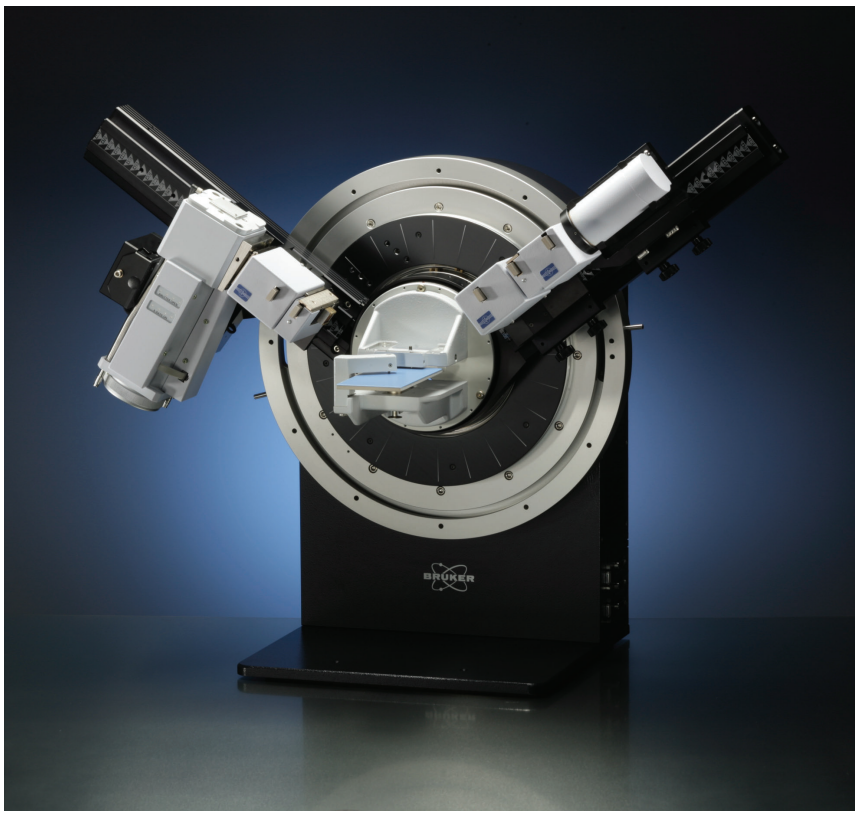
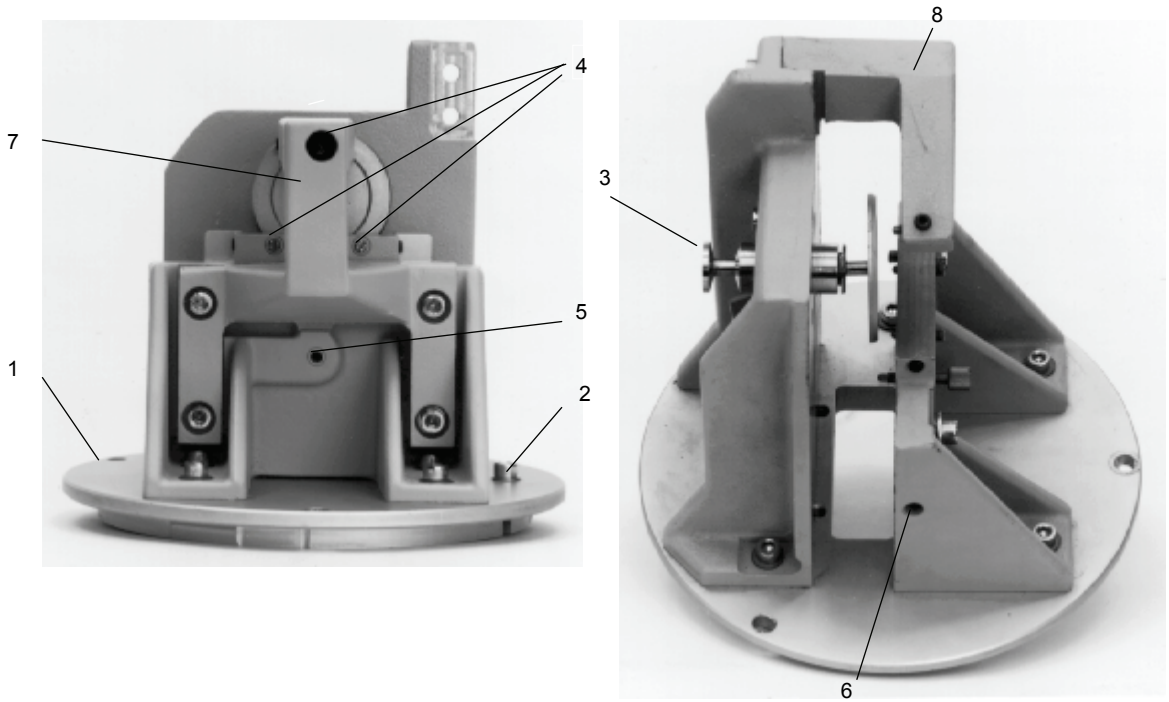



Figure 68: Standard sample stage mounted on goniometer.

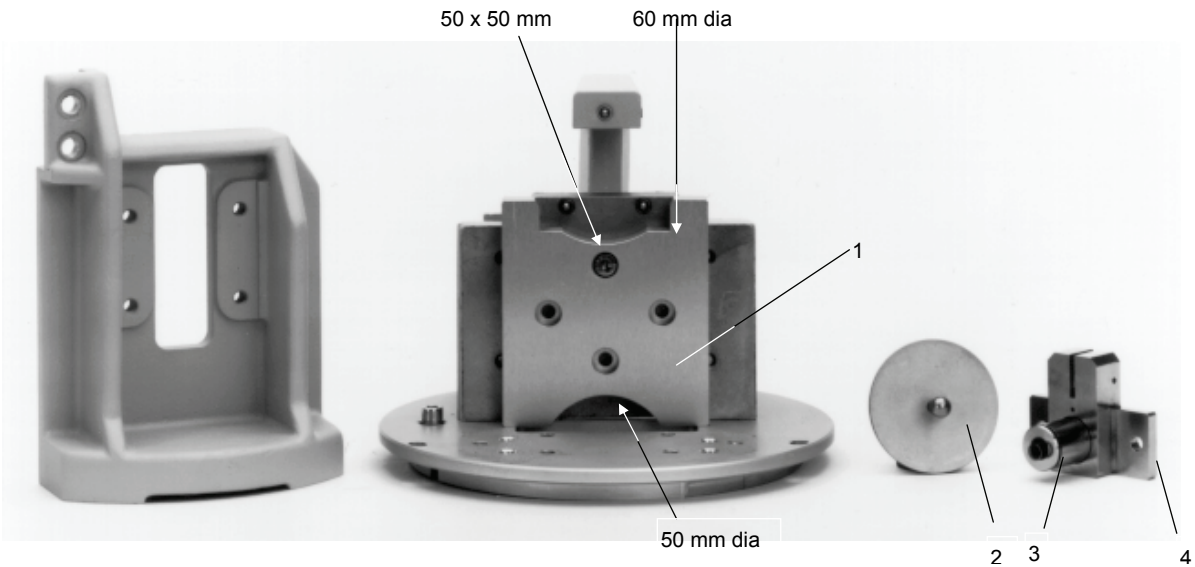



*1 Flange*
*2 Centering screw*
*3 Pressure unit*
*4 Set screws, factory-adjusted*
*5 Set screw*
*6 Lock Screw for [5]*
*7 Standard bracket*
*8 Front stop bracket*

Figure 69: Standard sample stage.

## 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 (Tab. 8) are available for use with the standard sample stage. These accessories are also used with the standard cup for the rotary sample stage.



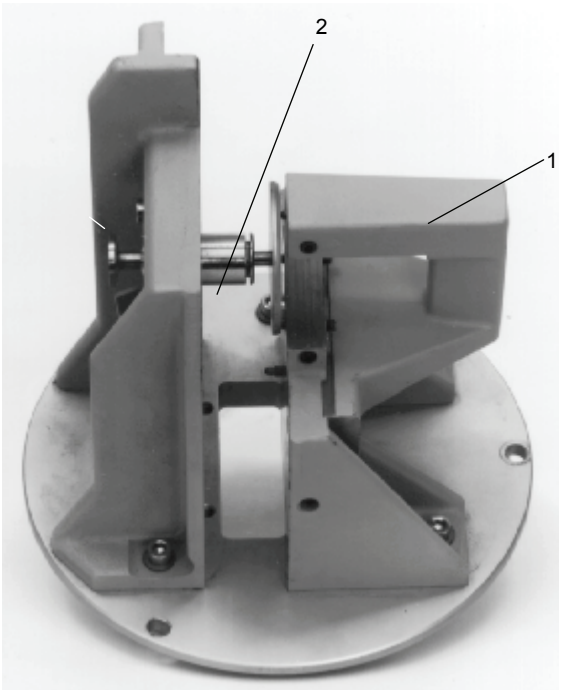

*1 Insertion aid*

*2 Pressure disk*

*3 Pressure pin*

*4 Pressure unit*

Figure 70: Insertion aid and pressure unit.



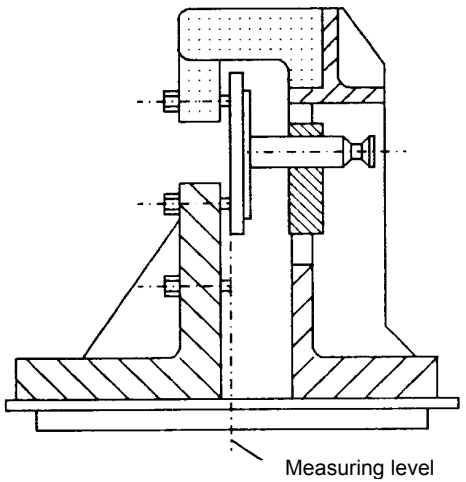

*1 Front stop bracket*

*2 Pressure unit*

Figure 71: Standard sample stage with front stop bracket.

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

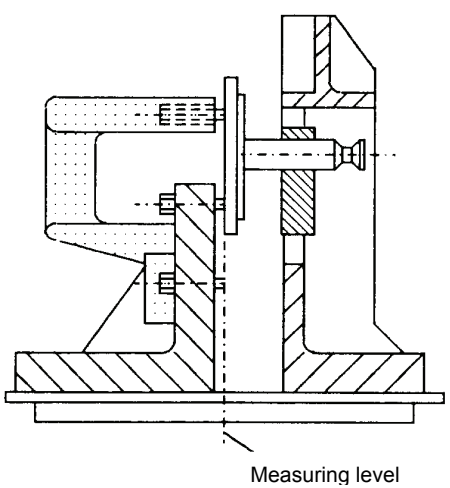
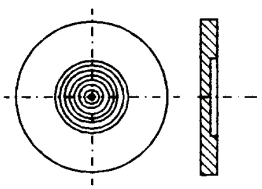
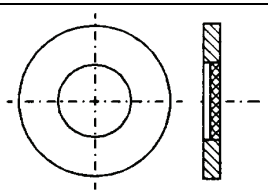
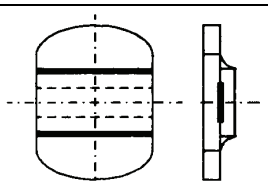
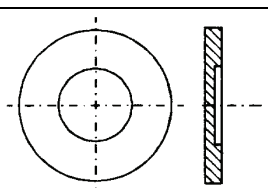



Figure 72: Standard sample stage.

Tab. 8: Accessories for the standard sample stage.

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

## Rotary Sample Stage

Instead of the standard sample stage, the sample stage with stepper motor drive (Figure 73, Figure 74) may be used for rotating measurements in reflection and transmission geometry.

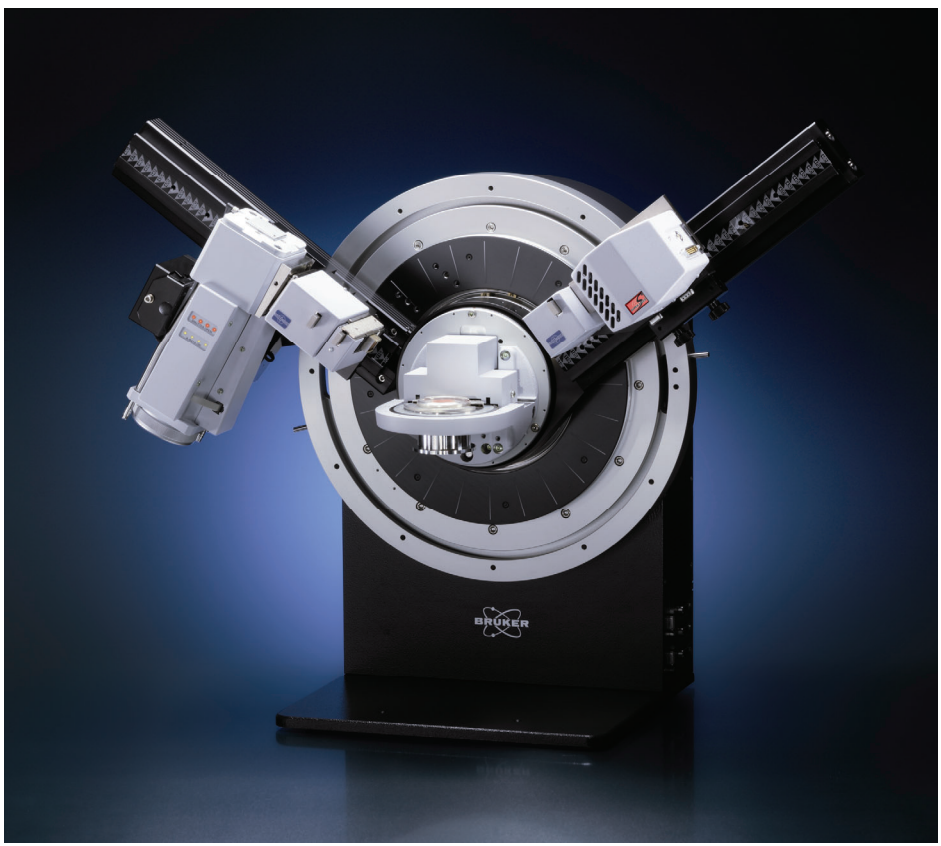
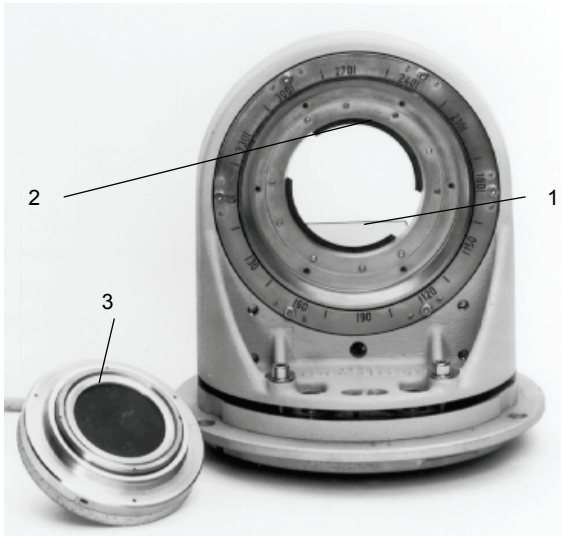



Figure 73: D8 ADVANCE with rotary sample stage.




*1 Drive motor*

*2 Seating ring for sample cup*

*3 Transmission sample cup*

Figure 74: Rotary sample stage.

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.

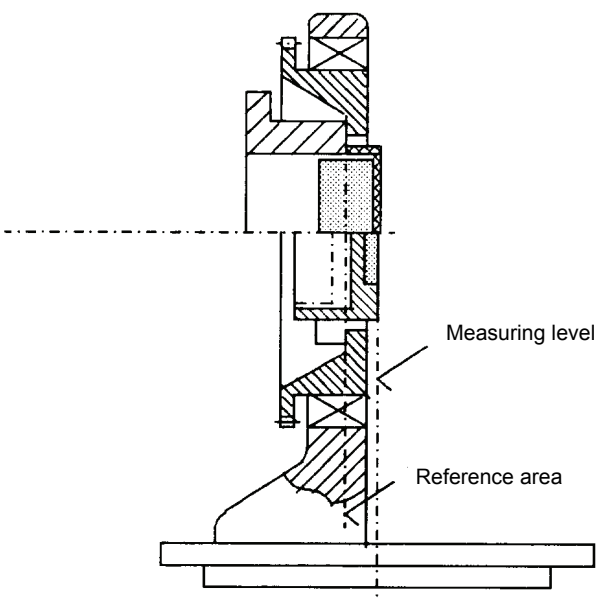


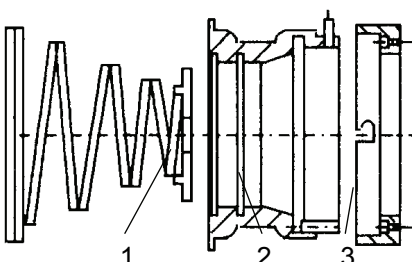

Figure 75: Design of the rotary sample stage.

## Accessories

A universal cup permits measurement of samples up to 50 mm diameter and 40 mm thickness (Figure 76).

Smaller samples may be supported using an intermediate ring (Figure 77) in the universal cup.

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.




*1 Pressure unit*

*2 Upper part*

*3 Lower part*

Figure 76: Universal cup

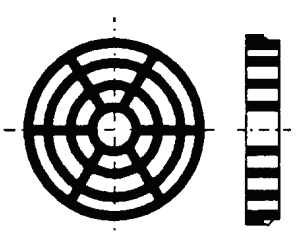


Figure 77: Intermediate ring, 50 mm diameter.

A transmission cup (Figure 78) for samples of up to 50 mm diameter and 4 mm thickness is provided for transmission measurements.

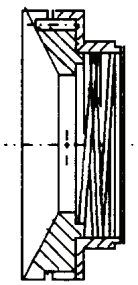


Figure 78: Transmission cup.

A sample holder (Figure 79) 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.

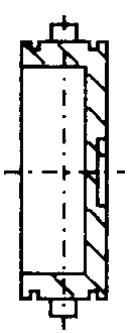


Figure 79: Sample holder.

Further accessories are sample holders (Figure 80) for measuring fibres and threads.

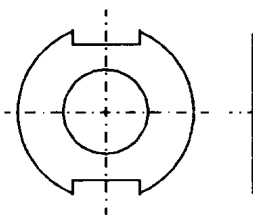
| | | Used with | Application |
|---|---|---|---|
| Sample holder, 50 mm diameter |  | Universal cup for rotary sample stage<br>Transmission cup for rotary sample stage | Examination of fibres and threads |

Figure 80: Sample holder for fibres and threads.

## FLIP-STICK

The FLIP-STICK is meant for routine reflection and/or transmission measurement of up to 9 samples of different types (Figure 81).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). 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 $\theta$–$\theta$ and $\theta$–$2\theta$ goniometers.

## General 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 |

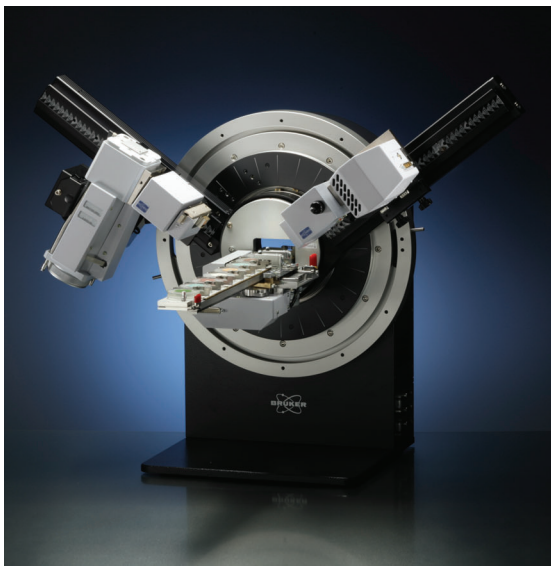


Figure 81: FLIP-STICK in reflection mode

## D8 Standby

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

## Change of Sample Rings

Push sample release, push sample ring against support rings, and release actuator (Figure 85).

## Release of Magazine

Move magazine to release position, if necessary: Sample magazine horizontal and magazine at position of sample 7 (Figure 86), use magazine release button in DIFFRAC.TOOLS).

Push red “translation release” actuator to the right on 9 Position Sample Stage to give free translation of magazine (Figure 87).

Move out magazine as far as possible. Use red “Magazine release” actuator on magazine to take out magazine. (Figure 88).



**Caution!**

Do not bend linear guide of 9 Position Sample Stage!



**Caution!**

Do not touch light barrier (fig. Figure 89)!

## Insertion of Magazine

Red “translation release” actuator of sample translation must be in out position (right, Figure 87).

Move out linear guide of 9 Position Sample Stage as far as possible – **do not bend!**

Shift magazine against bolt (Figure 88, Figure 89).

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 86).

Translate magazine roughly to position of sample 7

Push red “translation release” actuator of sample stage back to working position (left, Figure 90).

Initialise 9 Position Sample Stage.



**Caution!**

Do not touch light barrier! (fig. Figure 89)!

## Transmission Measurements

Figure 82 shows the D8 with FLIP-STICK in case of a transmission measurement. Take 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°.

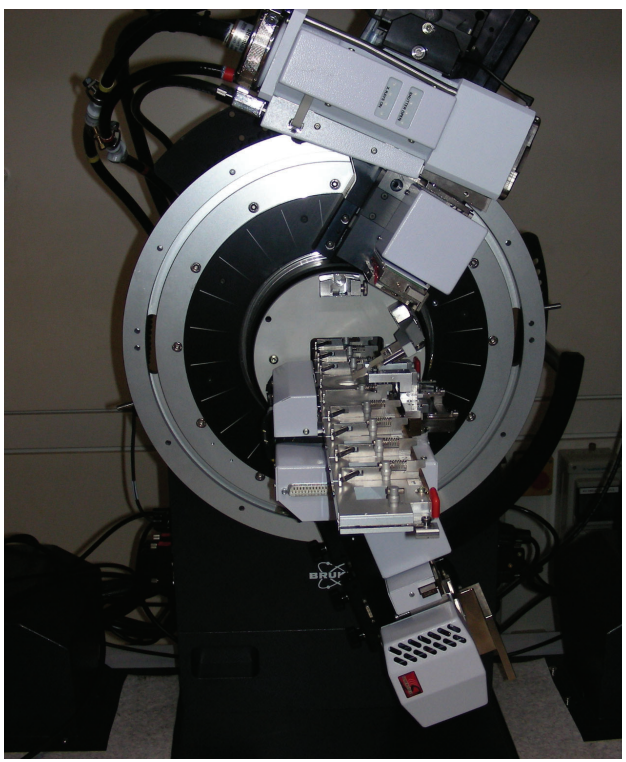


Figure 82: FLIP-STICK in transmission mode.

# 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 93

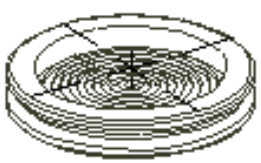
**Thick samples** (reflection only), sample thickness 20 mm:

C79298-A3244-B252, Figure 94

# Sample Rings

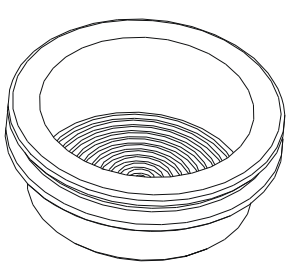
## Standard Sample Ring

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



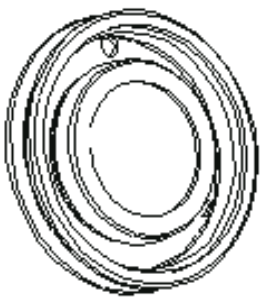
## Thick Samples

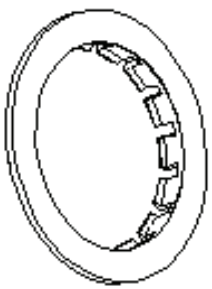
| Part Number 9 Sample Rings | Ø Sample | Sample Height | Ring Height | Material |
|---|---|---|---|---|
| C79298-A3244-C254 | 40 | 17 | 20 | Steel |



### Air Sealed

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

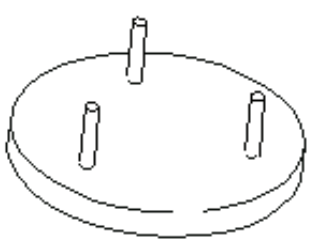




### Preparation Tool

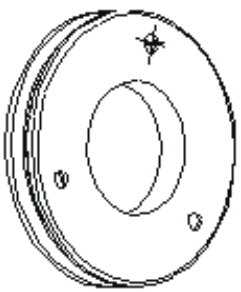
Preparation of back loading, air sealed, transmission, side loading sample rings)

C79298-A3244-B259



### Back Loading Sample Ring

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



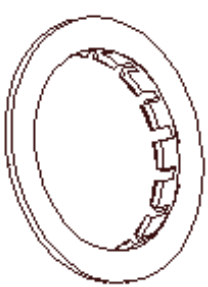


### Transmission Sample Ring

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

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

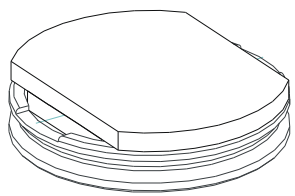




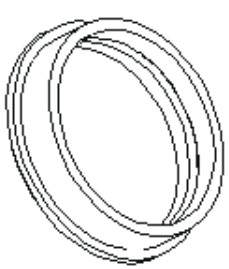
### Glas Slit

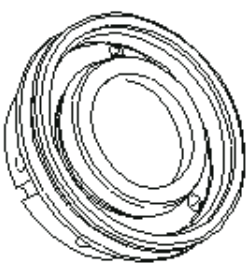
Alignment

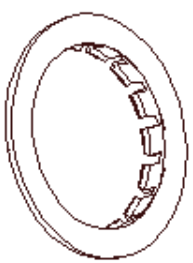
C79298-A3244-B255



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







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

C79298-A3244-B249



## Figures

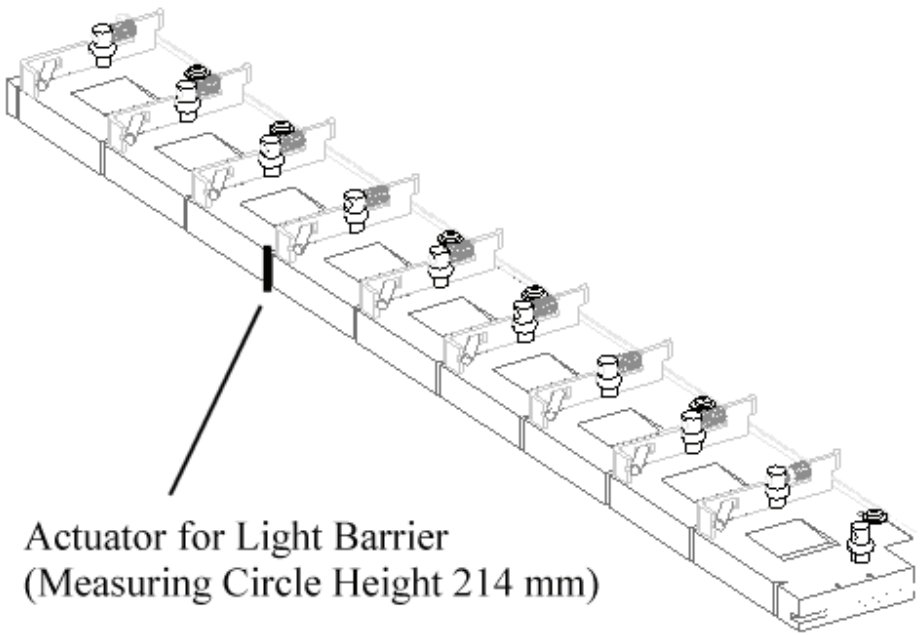



Figure 83: Standard magazine for reflection and transmission.

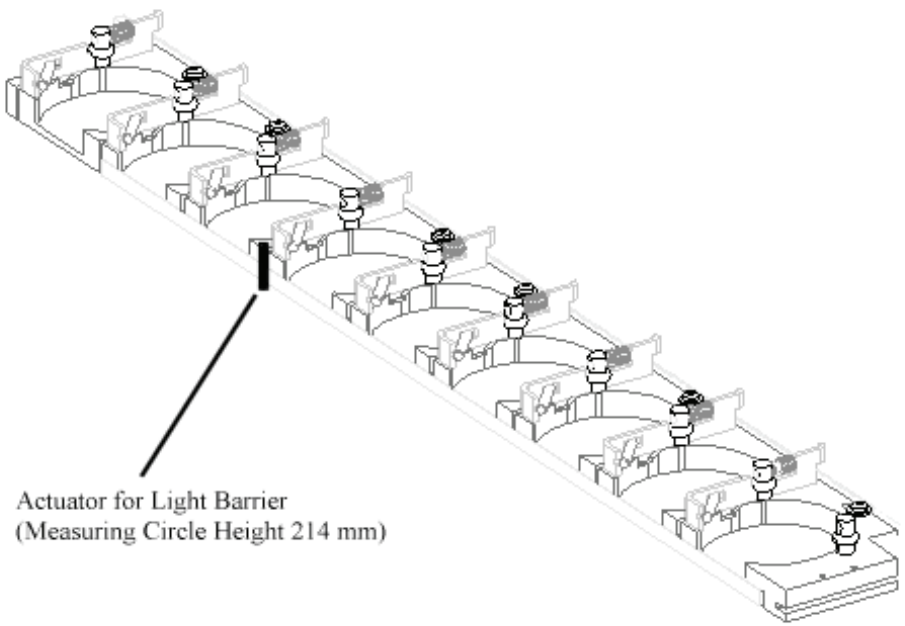



Figure 84: Magazine for reflection measurements on thick samples.

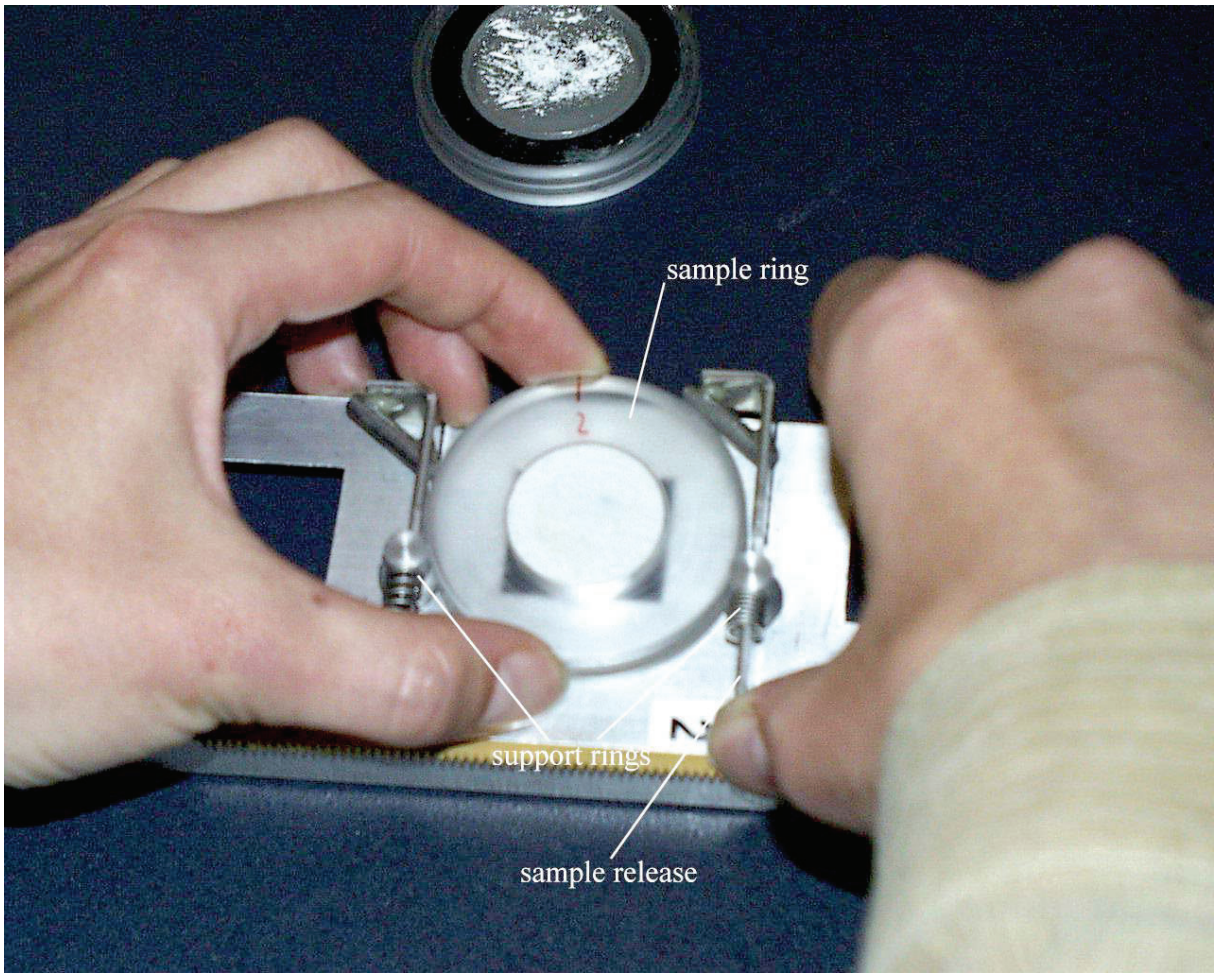



Figure 85: Changing of sample rings.

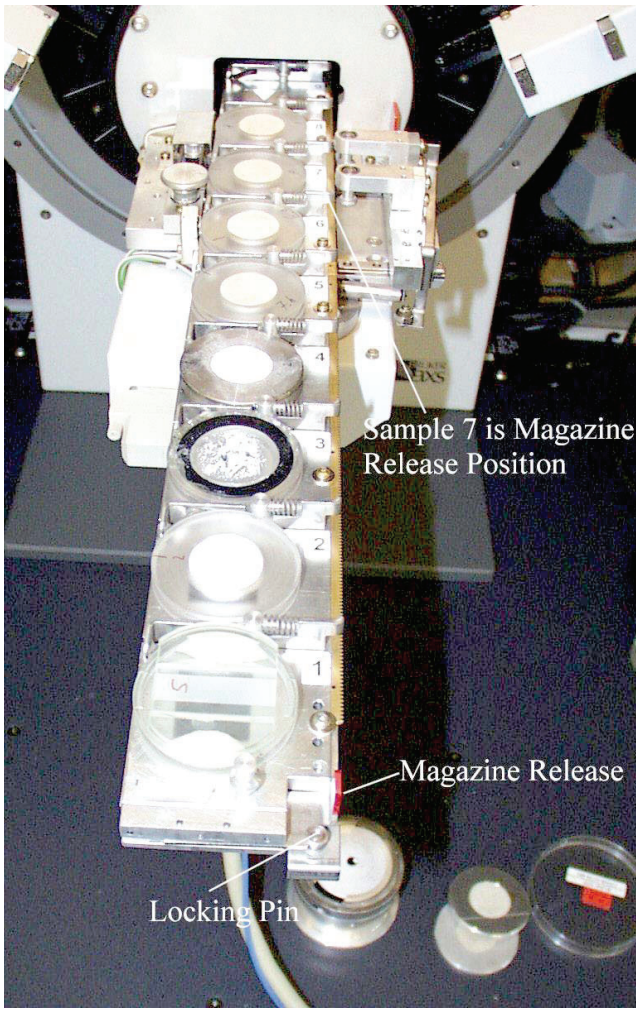



Figure 86: FLIP-STICK in release position.

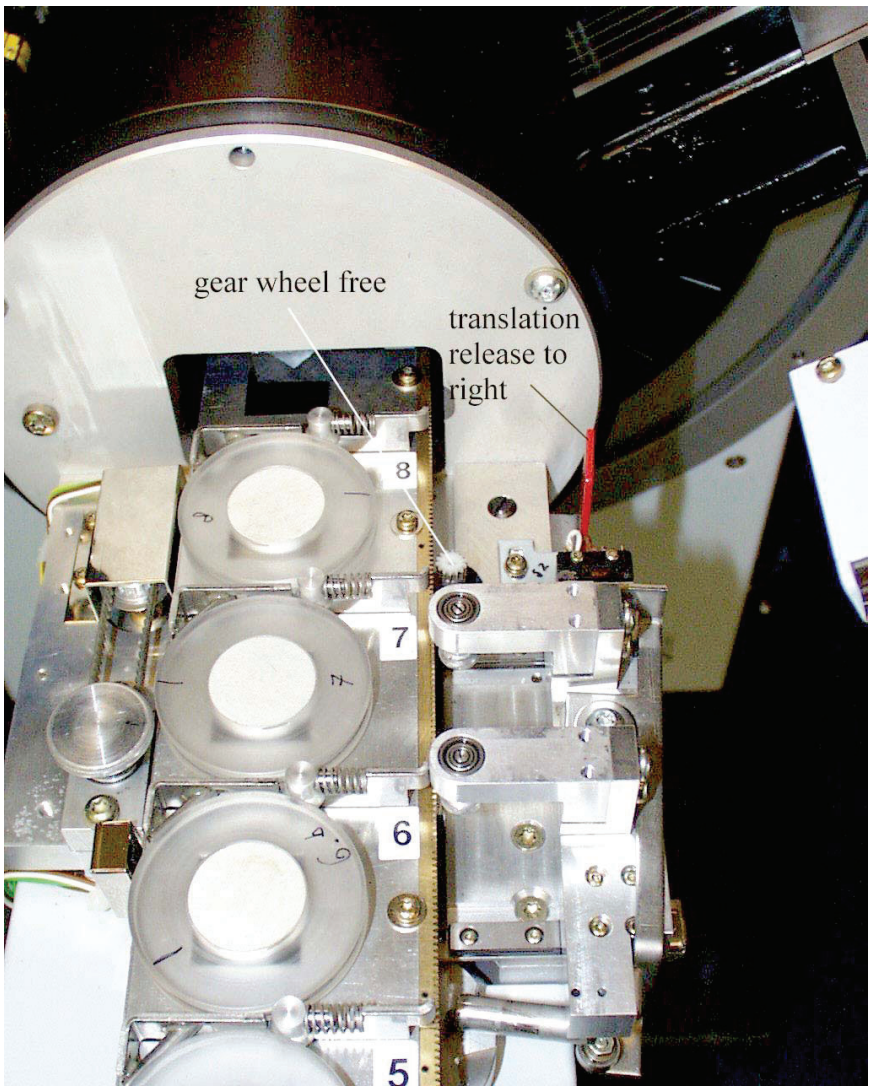



Figure 87: Magazine translation released.

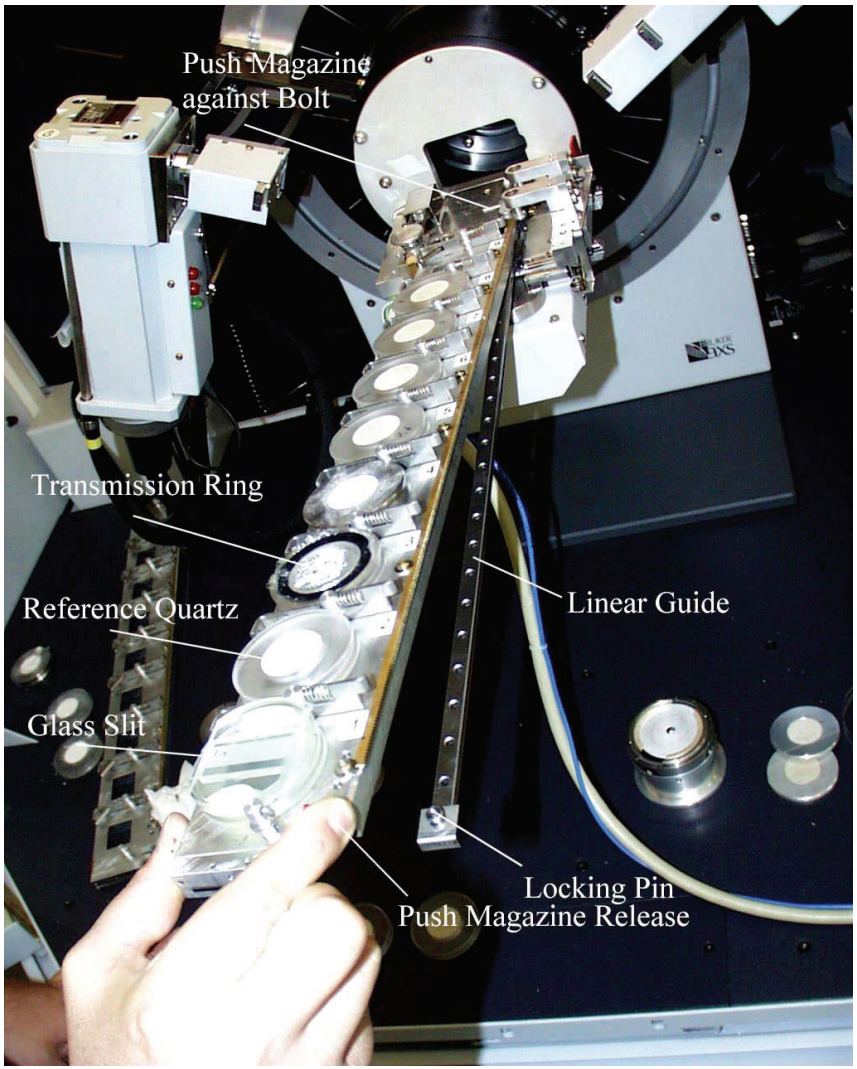



Figure 88: Changing of sample magazine.

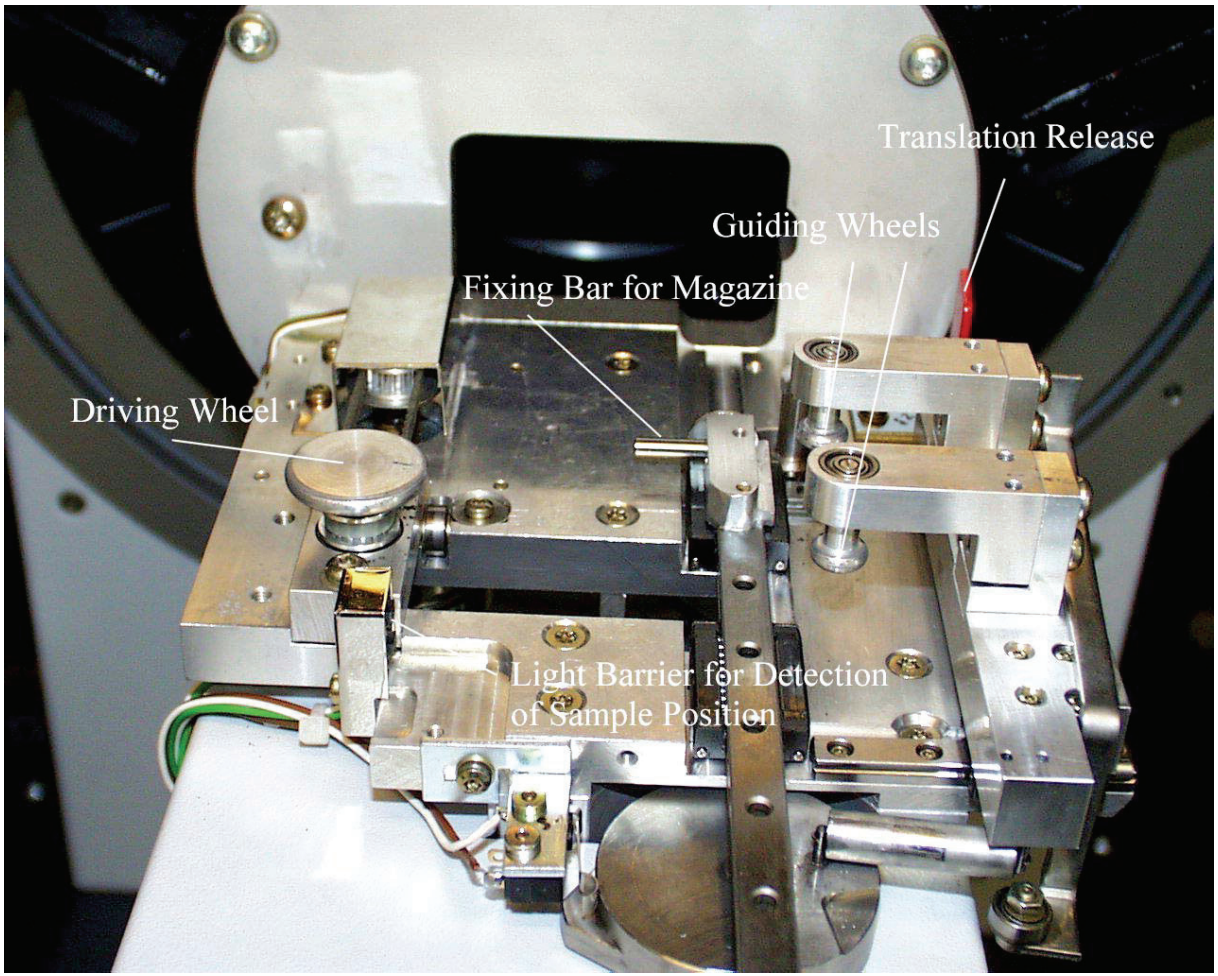



Figure 89: FLIP-STICK without magazine.

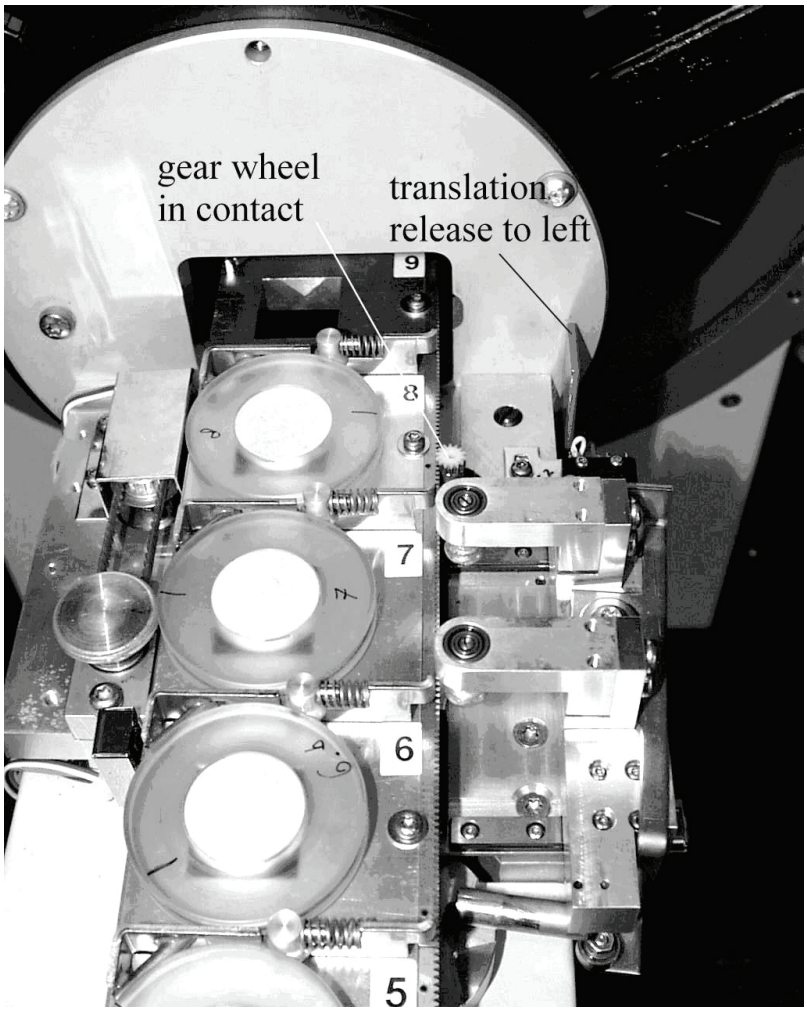



Figure 90: FLIP-STICK with magazine translation in working condition.

# 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. 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 manual DOC-M88-EXX101. Possible measurements:

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

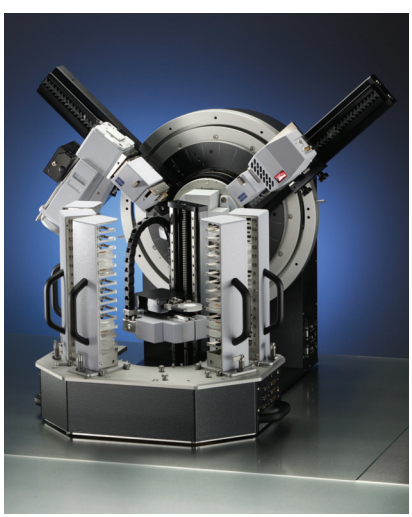


Figure 91: AUTO-CHANGER - sample stage for automatic exchange of many samples.

## Compact XYZ Stage

The motorized XYZ Sample Stage (Part-Nr.: A24B91) was designed for accurate alignment and/or mapping of a sample in the X-ray beam in a vertical D8 diffractometer. Completely implemented in the measurement software, the stage provides 25 mm travel in three directions which can be used for scanning or for alignment purposes.

The size of the support plane is 63 x 63 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.

**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

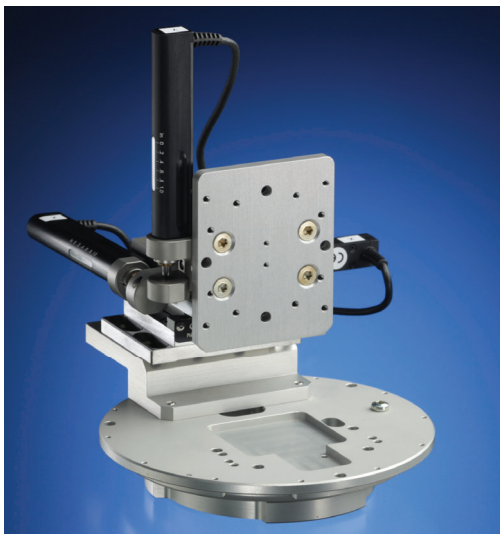


Figure 92: Compact XYZ stage.

**Warning!**

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!

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 XYZ stage user manual DOC-M88-EXX124) 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.



**Warning!**

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

Install the XYZ Stage and connect X, Y and Z cables to the 4-axis Indexer Board B232 (series 2 version). Configure the drives according to the example of the X-Drive (see XYZ stage user manual or by using the appropriate configuration section).

## Compact Eulerian Cradle

The Compact Eulerian Cradle (A25D285) 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 are motorized and can be used for positioning as well as scanning.

**Technical specifications:**

- for D8 ADVANCE/ D8 DISCOVER type instruments Theta/Theta or Theta/2Theta, 150 mm measurement height, 560 mm measurement circle diameter is recommended, two free ports on a 4-axis Indexer Board are required.
- bayonet mounting base for a alignment-free exchange of sample stages including recognition chip
- Chi circle: -5° to 95°, step size 0.02°, max. speed 20°/s
- Phi circle: unlimited, step size 0.001°, max. speed 3 rpm
- Manual Z-translation: max. 2 mm (one turn of the knurled thumb screw corresponds to 200 µm)
- 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** (see also Spare Parts List):

- Transport Box
- Compact Eulerian Cradle with User Manual
- Two 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, (order number A19B48). 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.

- Dial indicator for D8 (Part-Nr.: A100B99), for aligning the sample surface to the correct height in the center of the goniometer. If for some reason the sample surface can not be used for determination of the sample height (goniometer center), you may order an optional dial indicator arm, Part-Nr.: A100C714 and use a surface outside of the sample as reference.



**Caution: Crush Hazard!**

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!

- 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.

**Mounting the Compact Eulerian Cradle**

- Set the Compact Eulerian Cradle onto the goniometer.
- Partially screw in the three fastening screws into the mounting plate so that the cradle can still be shifted to the theta bearing.
- 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.
- Tighten the three fastening screws.
- 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.
- Remove two screws of the transportation lock, which fixes the Phi-stage to the C-bow. 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.

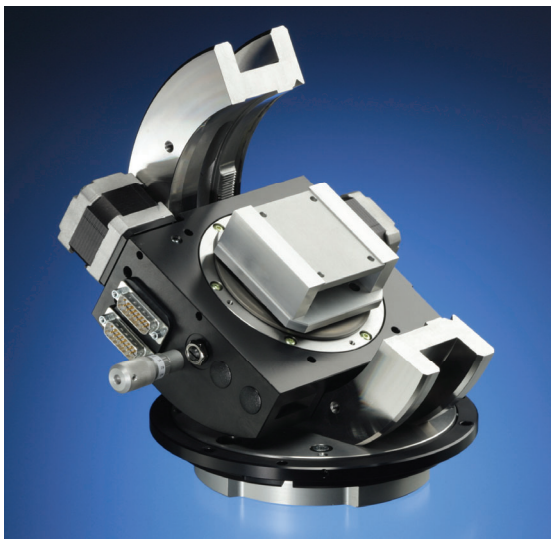


Figure 93: Compact Eulerian Cradle.

## 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 in Figure 96. Two knife edges can be mounted on the capillary stage (Figure 94, Figure 95). 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.

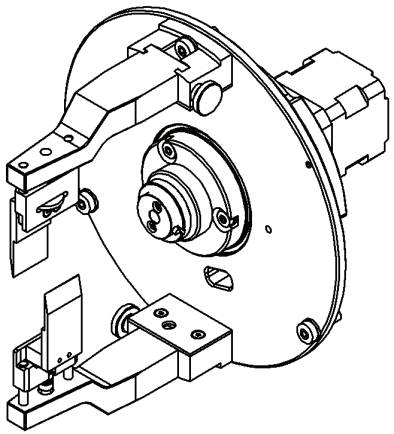


Figure 94: Capillary stage without goniometer head.

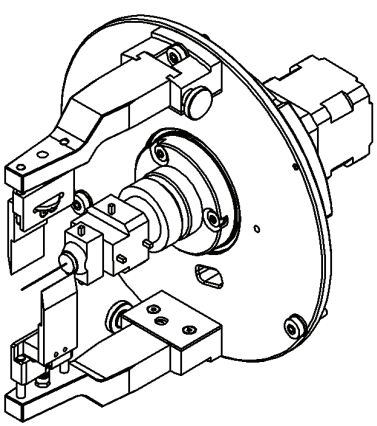


Figure 95: Capillary sample stage with goniometer head.

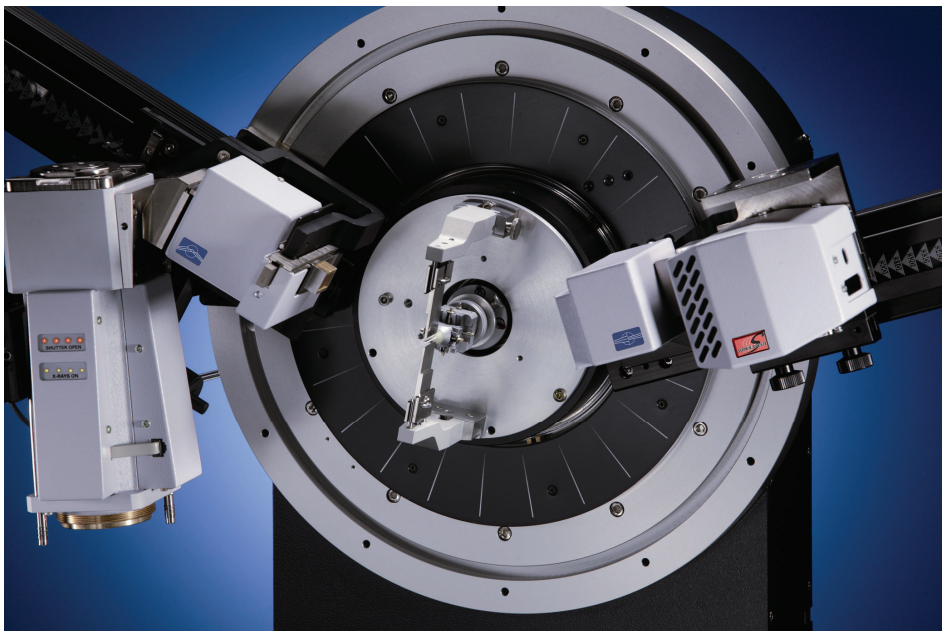


Figure 96: Transmission measurement with powder sample in capillary. A Johansson monochromator for transmission geometry is used on primary side in connection with a LYNXEYE detector on secondary side.

## 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 manual DOC-M88-EXX047 and in the user manuals of the suppliers.




Figure 97: Non ambient setup with Hot Humidity System and VÅNTEC-1.

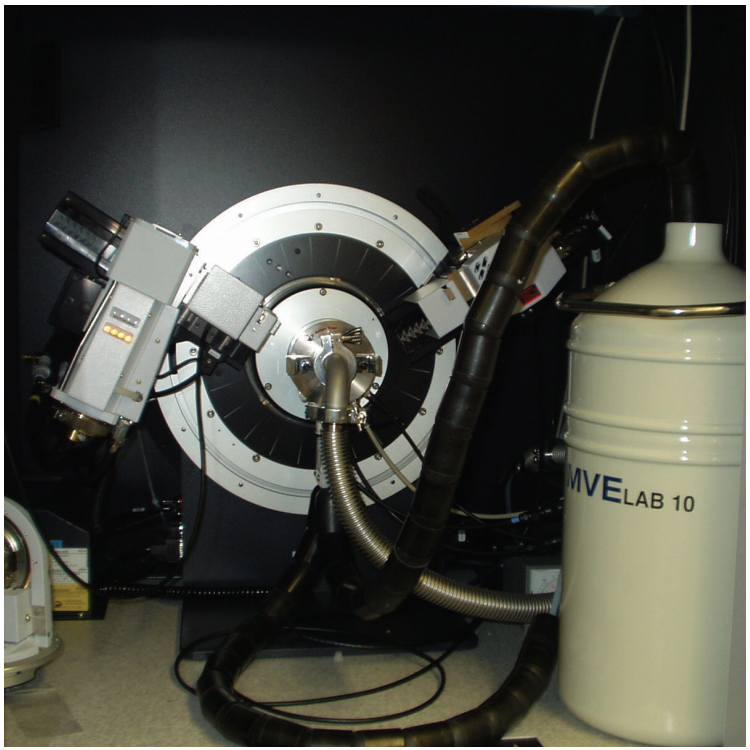



Figure 98: Non ambient setup with Hot Humidity System and LYNXEYE.

# Detectors

## Scintillation Detector

A scintillation counter is used as the most basic X-ray detector (Figure 99). It enables X-ray measurement in the wavelength range between 0.05 and 0.3 nm (4…25keV)

**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.

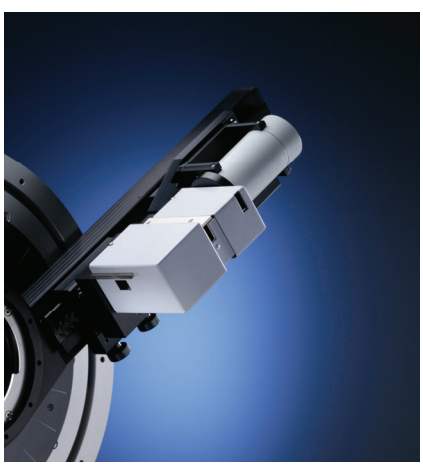


Figure 99: Scintillation counter.



**Warning!**

Be careful, the detector has a beryllium window. **Do not touch this window!**

# LYNXEYE

The LYNXEYE is a 1-D 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 1-D mode or in the integrating 0-D 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 1-Dimensional Bragg-Brentano measurements and rotated by 90° for out of plane measurements or for increasing the dynamical range in the integrated 0-D mode.

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



Figure 100: LYNXEYE detector.

# VÅNTEC-1

VÅNTEC-1 is a 1-D 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 result are quite compareable 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.

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 16mm. 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 user manual for VÅNTEC-1 DOC-M88-EXX072.



Figure 101: VÅNTEC-1 detector.

## SOL-XE

The SOL-XE detector is based on an active Si(Li) solid state element with a thermoelectric cooling unit (Peltier cooling element). Figure 102 shows how the SOL-XE detector is mounted together with a fixed slit assembly to the 2theta circle of a D8.

**Warning!**

Be careful, the detector has a beryllium window. **Do not touch this window!**

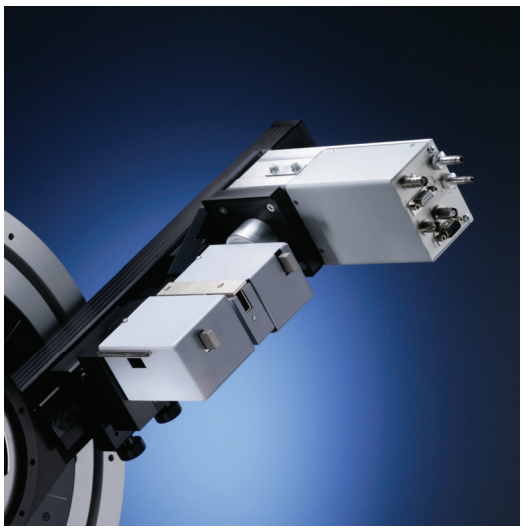


Figure 102: SOL-XE mounted on D8 ADVANCE.

Like for the scintillation detector all cables, wires and hoses have to lie in a correct way within the cable channel otherwise damages are possible.

The standard detector interface board can process the output signals of the SOL-XE.

The Si(Li) detector is mounted in a vacuum chamber which is evacuated by an ion pump. The solid state unit is cooled by a Peltier element down to –90°C. Weak charge pulse signals from the detector are amplified by a charge sensitive preamplifier. The electronic thermal noise is suppressed by cooling the Si(Li) chip.

**The detector has connectors for:**

- Electrical supply voltage for preamp and Peltier element.
- Detector high voltage.
- Signal output from preamp.
- HV for the ion pump.

- Water cooling. Water absorbs the heat from the warm side of the Peltier element and transports it to a heat exchanger.

The 19'' controller Multispectrum contains not only the electronic circuit boards for signal processing but also a closed cooling water circle for the Peltier element.

Multispectrum is processing the analogue data which come from the detector. This unit consists of:

- Analogue processor.
- AD-converter.
- Microprocessor with memory module.
- RS-232 interface.
- Power supply for the Peltier element, ion pump, detector preamp and HV for the Si(Li) chip.
- Safety circuit for checking the operational parameters of SOL-XE.

Important features of the SOL-XE solid state detector for D8 systems are:

- Energy range from 1,7keV to 30keV.
- Active area is 4 x 15 $mm^2$ and optimised for all useful D8 diffraction geometries.
- Linear counting rate up to 80 kcps with an energy resolution to separate Cu K$\beta$ from K$\alpha$ line.
- Energy calibration is supported by software.
- Seamless integration into the DIFFRAC.SUITE environment.
- Automatic setting of operating parameters.

The most important applications for this energy dispersive solid state detector are X-ray diffraction measurements from samples generating fluorescent radiation. The useful K$\alpha$ line can be separated from any fluorescent line by setting an energy window to the K$\alpha$ line. Simple qualitative analysis of the fluorescent radiation can also be performed.

A large active area guarantees that any restriction on the use of the detector for grazing incidence diffraction is not relevant.

**Warning!**



Keep the ion pump switched on continuously. The detector is connected to a battery for powering the ion pump in case of mains power failure or for transport. Besides for transportation the multispectrum power plug should never be disconnected from mains connection. **For further important notes and information read the detailed SOL-XE manual!**

# Operating the D8 ADVANCE/ D8 DISCOVER

## Instrument Status

### 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 Figure 103 and perform the following steps:

- Loosen the focus translation locking screw.
- Translate the tube focus using the screw shown in Figure 103.
- Monitor the intensity using the ratemeter of DIFFRAC.SUITE.
- When the optimal intensity is reached, tighten the translation locking screw.




Figure 103: The locations of the screws for performing a tube focus translation.

# Tube

## 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.

# Changing Tubes

## Switching Between Point and Line Focus

Figure 104 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 105 shows the plate in the point and line focus positions.

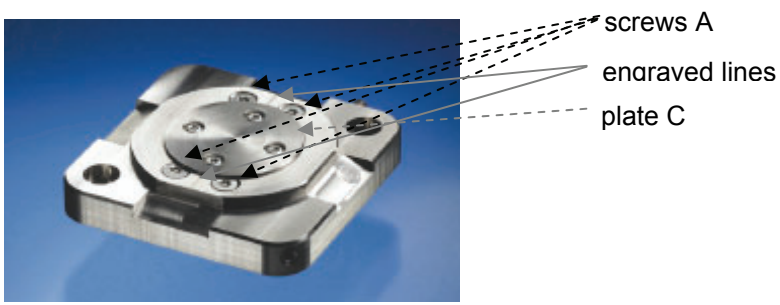



Figure 104: The TWIST-TUBE supporting plate. Screws A should be loosened (approximately one turn, to prevent jamming the screws under plate B) before performing the filament rotation. The engraved lines show the direction of the length of the filament.

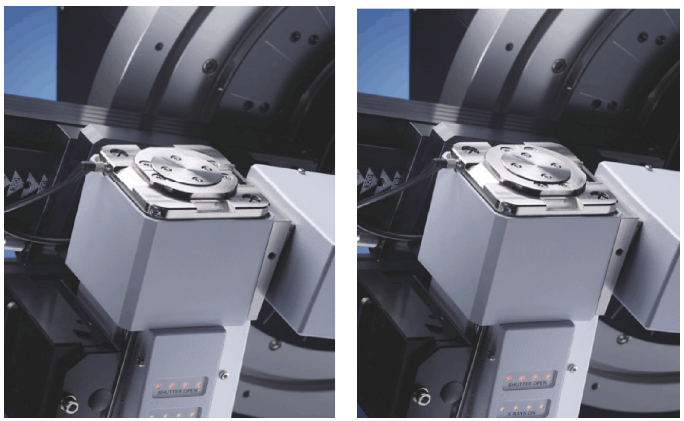


Figure 105: The supporting plates as shown on the tube housing, in the point focus (left) and line focus (right) configurations.



### Danger: Electrical Hazards

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

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” (figure 106). This must be done before touching and loosening any of the screws labelled as “A” within this section!

The high voltage applied to the X-ray tube is turned off if the display of the Screen-key indicates the “Switch-on” symbol according to table 9.

If the Screen-key displays any of the symbols “Heating on“, “X-rays on, generator ramping up/down” or “X-rays on, generator ready” (see table 10) you must press once the Screen-key switch and wait until it is showing the “Switch-on” symbol.

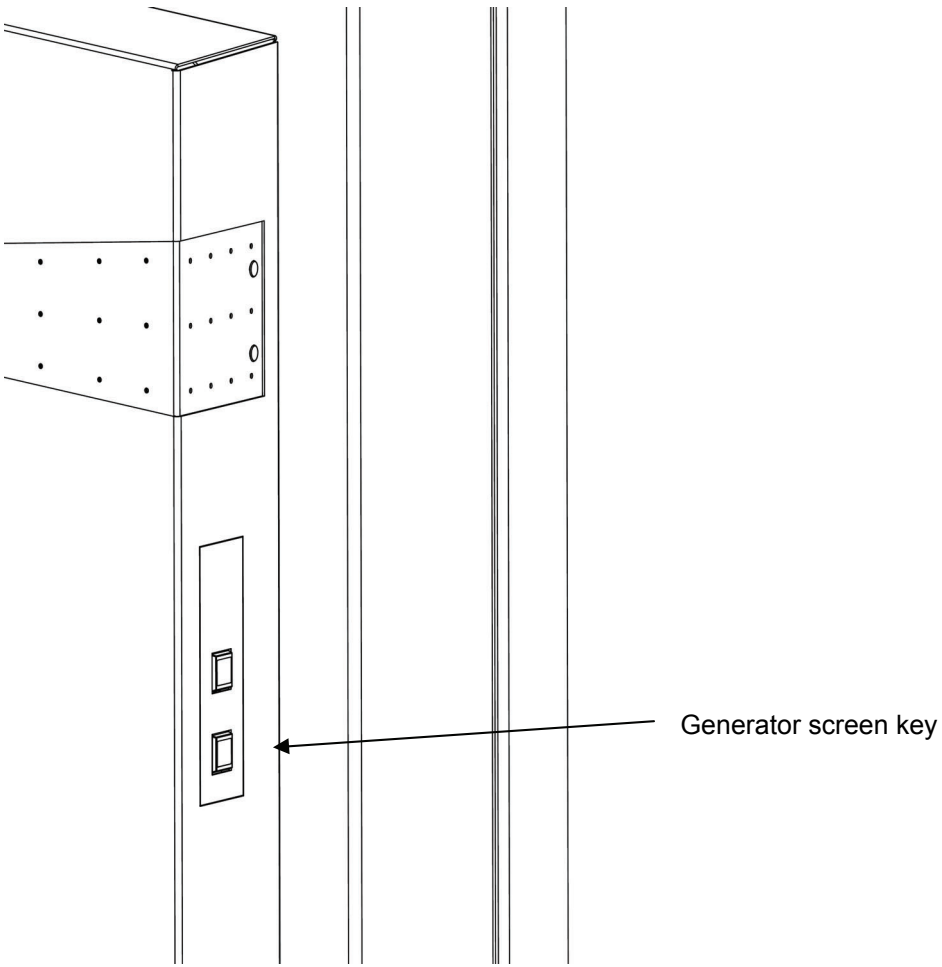



Figure 106: Location of the Generator Screen-key (Turn on/off X-rays)

Tab. 9: Generator Screen-key when high voltage turned off

| | |
|---|---|
|  | **Switch-on symbol**<br>Displayed when the generator is off. |

Tab. 10: Generator Screen-key when high voltage turned on or heating voltage is activated

| | |
|---|---|
|  | **Heating on**<br>Blinking or permanent yellow light indicates the tube is being heated. |
|  | **X-rays on, generator ramping up/down**<br>Displayed when X-rays are being generated and the generator is ramping the supply voltage up or down. The background is yellow. |
|  | **X-rays on, generator ready**<br>Displayed when the generator has reached a value set in the instrument configuration. This symbol is a negative image of the “ramping up/down” symbol. |

To perform a switch between point and line focus:

Step 1 - Turn off the high voltage (X-ray generator HV).

Step 2 - Loosen the four screws A shown in Figure 104.



**Note**

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.

Step 3 - Using the special tool provided, perform the rotation as shown in Figure 107.

Step 4 - Tighten screws A, to move the filament back into the proper focus position.

Step 5 - Turn on the generator high voltage.



Figure 107: Performing the filament rotation.

# Mounting Optics

## Optics Mount (SNAP-LOCK)

The optics mount is shown in Figure 108. 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.

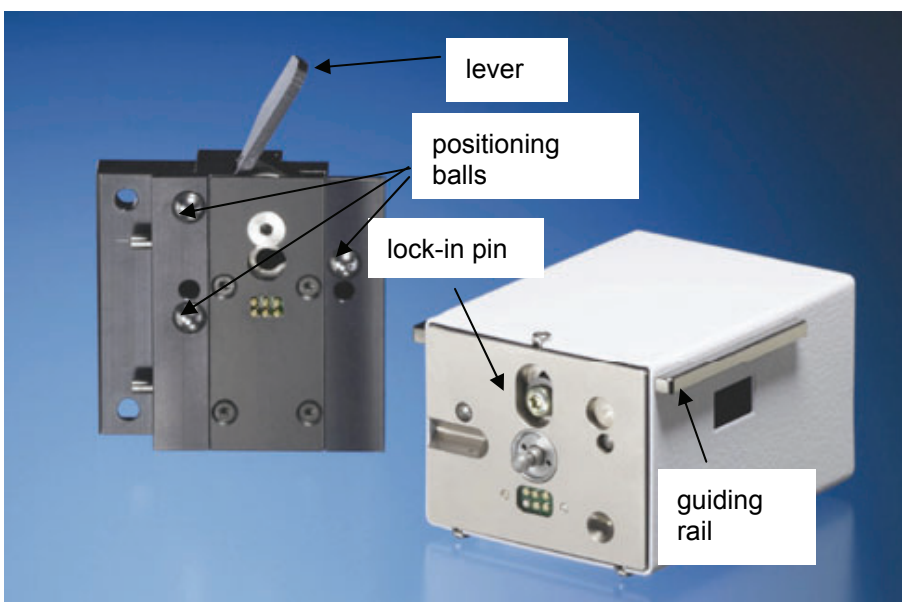



Figure 108: A detailed view of an optics mount and optic.

**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.

- Open lever (Figure 109).
- Put in the optics module. The guiding rails (Figure 110, Figure 111) 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.
- 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.

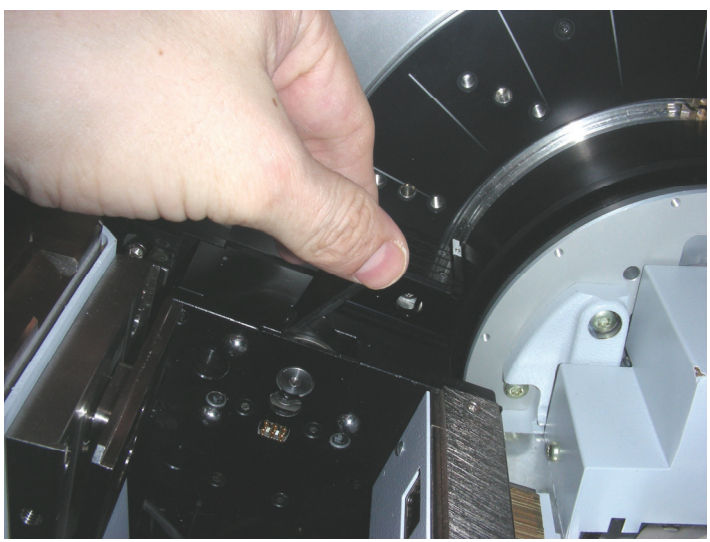


Figure 109: Lever.

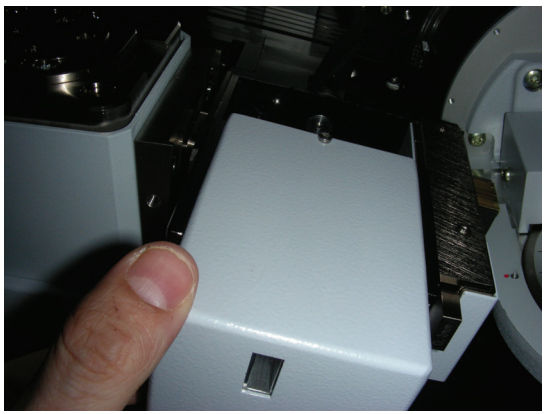


Figure 110: Guiding rail (a).

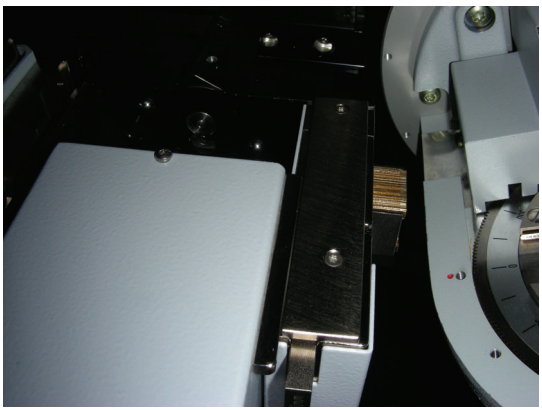


Figure 111: Guiding rails (b).

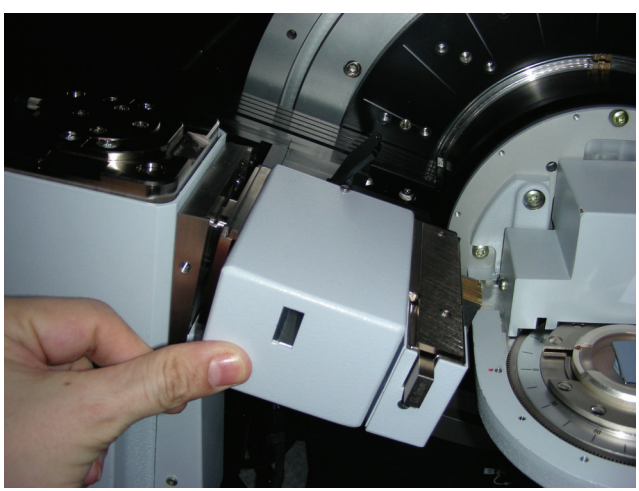


Figure 112: Press at this spot if you mount any optics.

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.

**Caution!**

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.

## 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. User Manual DOC-M88-EXX191). The change in DAVINCI is displayed in Figure 113 .

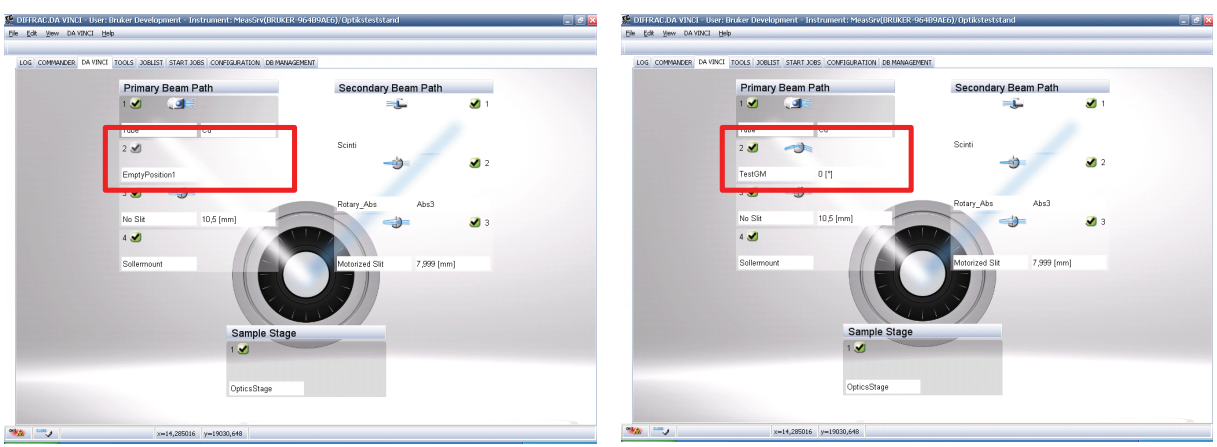



Figure 113: DAVINCI plug-in (Left: No optics is mounted on the primary beam path; Right: An optics called TestGM is mounted) on the primary side beam path).

# Mounting Sample Stages

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

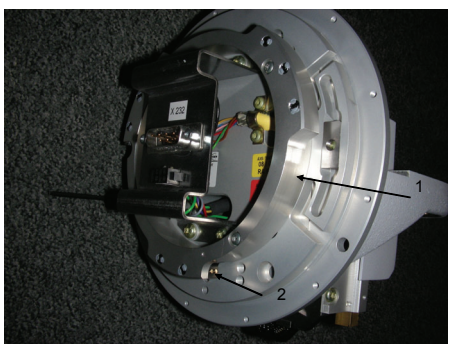



Figure 114: Stage with bayonet (1) and recognition chip (2).

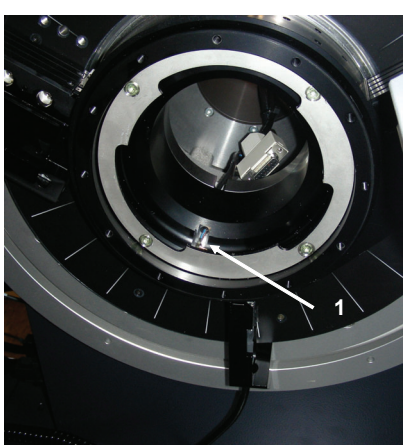



Figure 115: Goniometer with bayonet and recognition readout (1).

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. Then turn the stage clockwise to fix it with the bayonet mechanism. Use the centering screw (Figure 117) 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.

**Note**

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

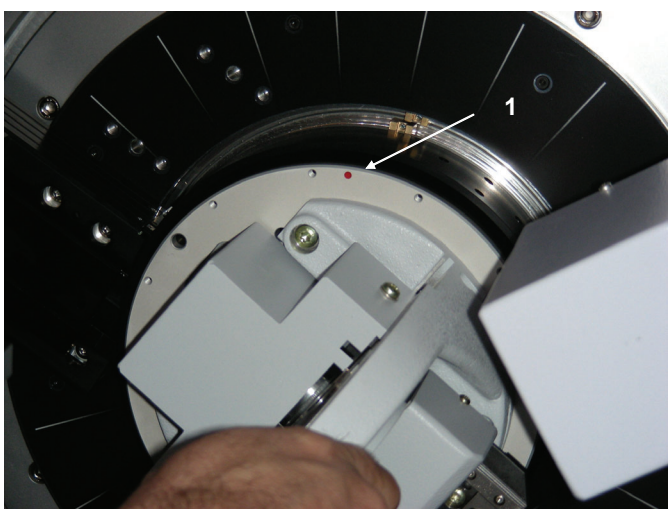



Figure 116: Stage with red mounting marker (1). When mounting the marker on the stage must match the marker position on the goniometer. Turn clockwise for fixing the stage with the bayonet mechanism. Then firmly fix the stage with screws.

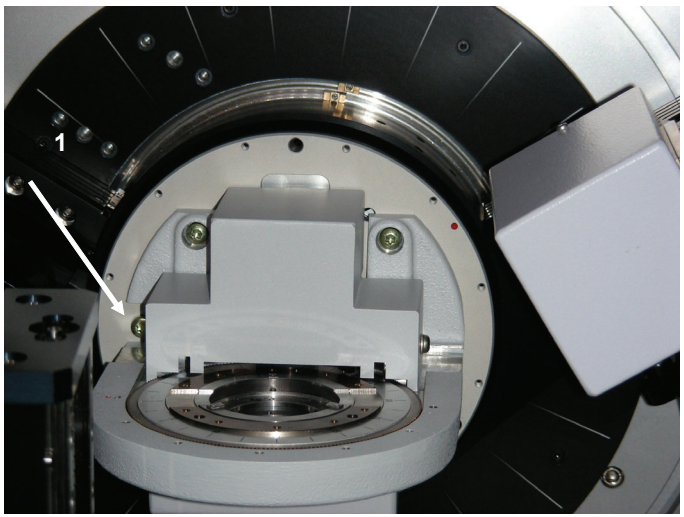



Figure 117: Centering screw (1) of the stage.

**Note**

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.

## 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 in Figure 118. For more detail refer to the DIFFRAC.SUITE User Manual (Doc-M88-EXX191).

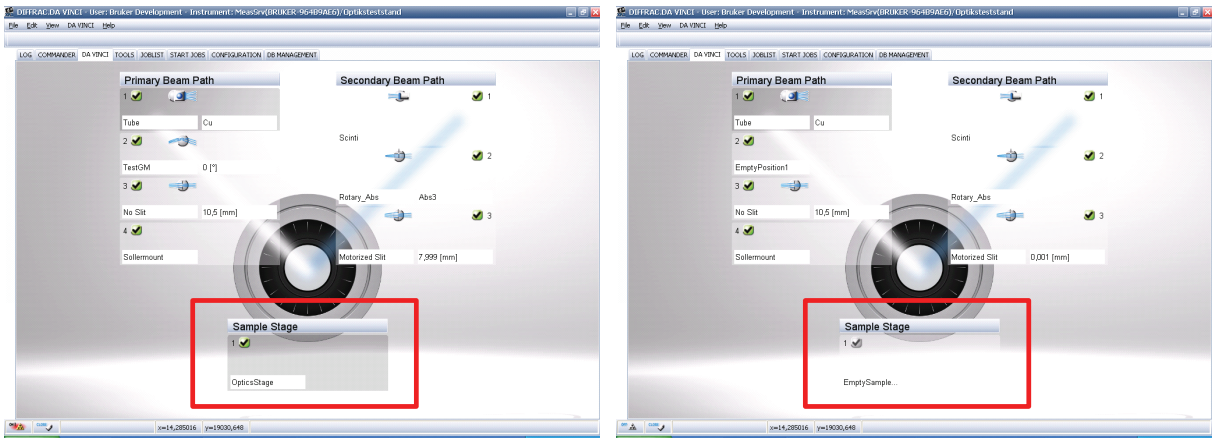



Figure 118: DAVINCI plug-in (Left: A stage is mounted; Right: No stage is mounted).

# Mounting Detectors

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

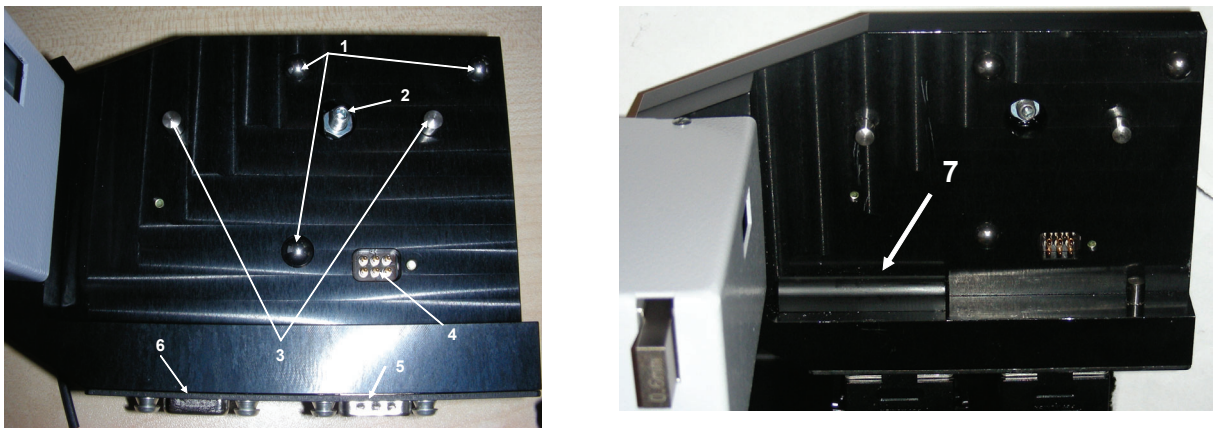



Figure 119: Universal Detector Mount (UDM) (1 three positioning balls mainly used for LYNXEYE mounting, 2 fixing screw, 3 guiding pins, 4 Chip-Readout interface, 5 socket for cable between UDM and Goniometer, 6 socket for cable UDM and secondary optical bench, 7 positioning rail UDM).

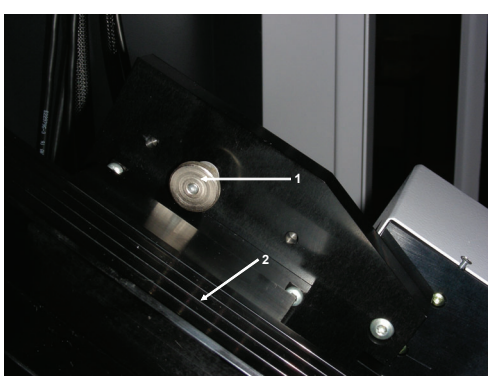



Figure 120: Backside of the universal detector mount (1 fixing screw; 2 track).

The detectors which can be mounted this way are:

- Scintillation counter
- SOL-XE
- LYNXEYE

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

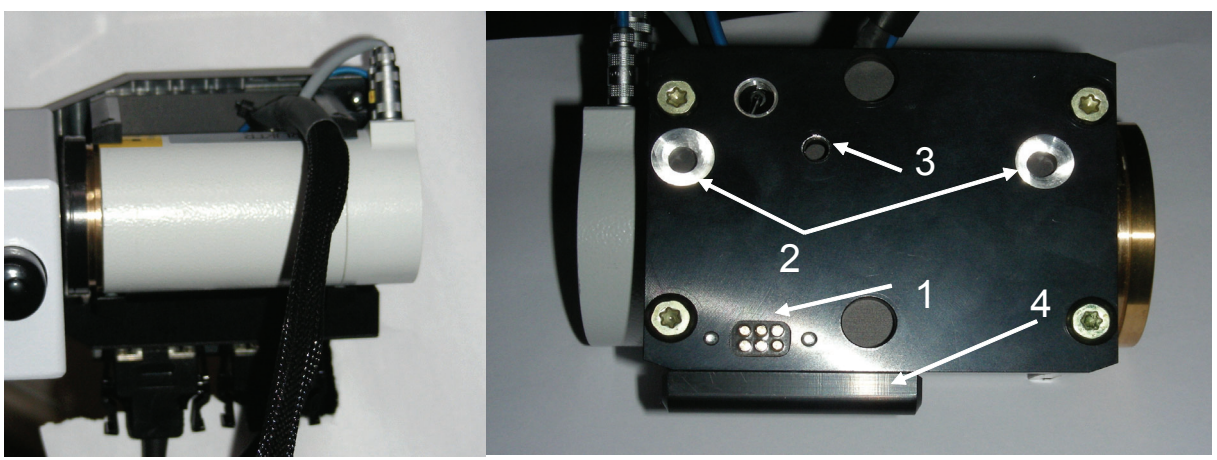



Figure 121: Scintillation detector is fixed on a detector holder which is mounted on the UDM; backside of the detector holder (1 Recognition Chip, 2 holes for guiding pins, 3 thread for fixing screw, 4 positioning rail detector holder).

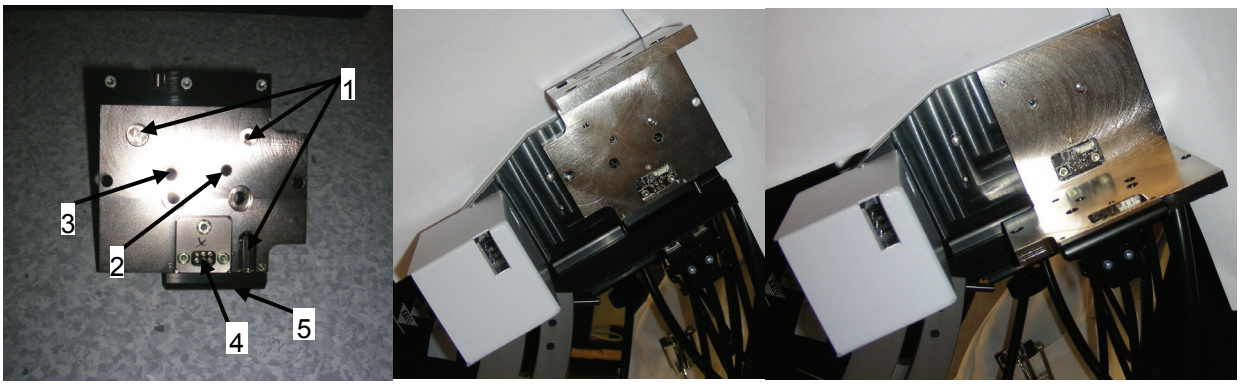



Figure 122, Figure 123: Left: LYNYEYE detector holder (1 reference surfaces,2 thread for fixing screw, 3 hole for guiding pin, 4 Recognition Chip, 5 positioning rail), Mittle/Right: LYNX-EYE detector holder mounted on the universal detector mount, (right: 0° position, left: 90° position) .

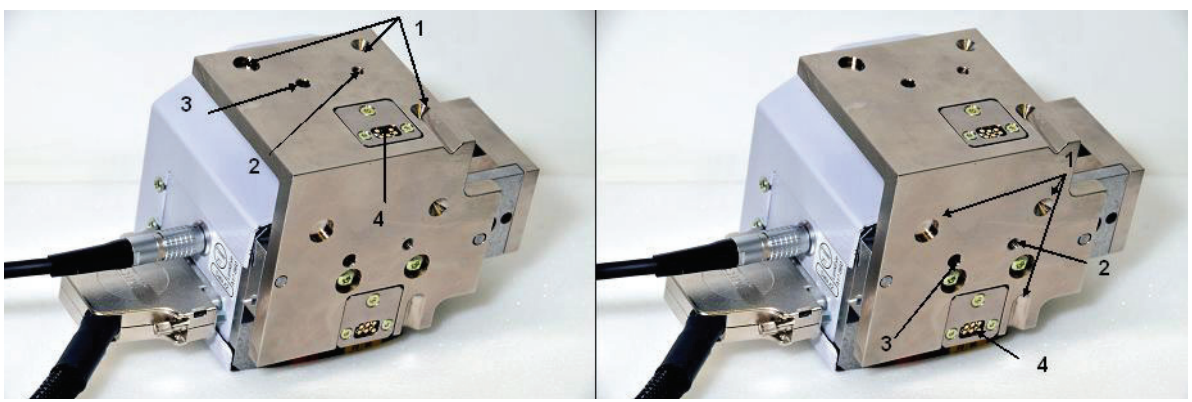



Figure 124: LYNXEYE with detector holder (1. reference surfaces, 2. thread for fixing screw, 3. hole for guiding pin, 4. Recognition chip).

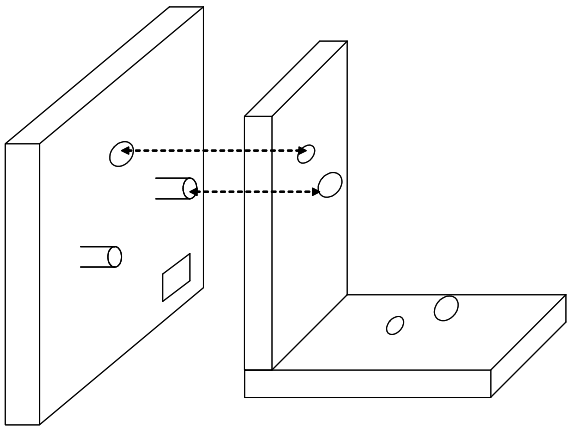


Figure 125: Mounting of the detector holder on the universal detector mount (LYNXEYE).

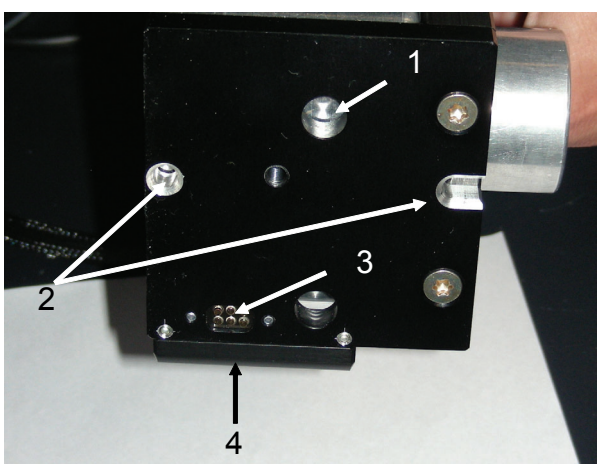



Figure 126: SOL-XE with detector holder (1 thread for fixing screw, 2 holes for guiding pins, 3 Recognition chip, 4 positioning rail detector holder).

When mounting the detector holder keep the detector holder parallel to the surface of the universal detector mount. Place the positioning rails on the detector holder into the corresponding position on the Universal detector mount (UDM). The guiding pins of the universal detector mount must fit into the corresponding holes in the detector holder as shown in Figure 125. Also the threads for the fixing screw must be at the right position.

**Note**

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.

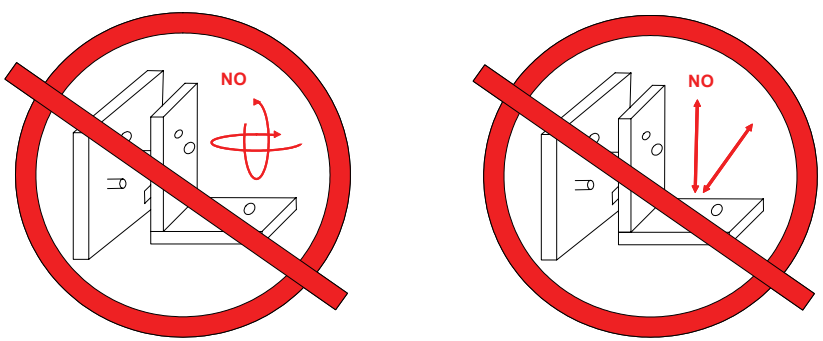



Figure 127: If the detector holder is close to the surface of the universal detector mount: Do not rotate or translate it. If you rotate or translate the detector holder the chip interface can be damaged.

**Note**

The LYNXEYE detector holder only uses the rear guiding pin.

If the detector holder is properly placed fix the holder with the fixing screw at the backside of the universal detector mount.

## 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 Figure 128. For more detail refer to DIFFRAC.SUITE User Manual (Doc-M88-EXX191).

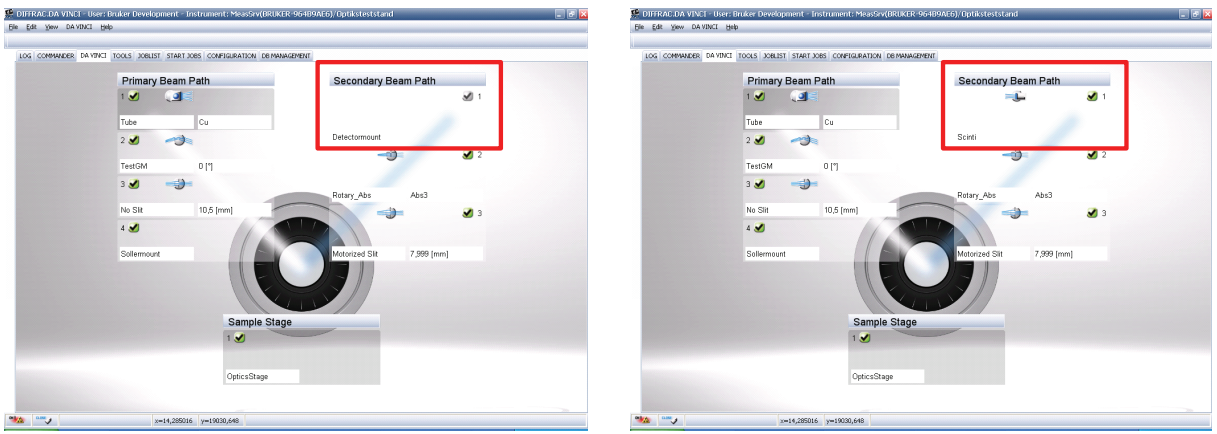



Figure 128: DAVINCI plug-in (Left: No detector is mounted; Right: A scintillation counter is mounted).

# Typical Scans

## 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)<br>(Omega) | θ/θ: Source arm of goniometer<br>θ/2θ: Sample | Around horizontal axis of goniometer | θ/θ: Surface of sample<br>(horizontal line through centre of goniometer)<br>θ/2θ: direction of beam path |
| TwoTheta (2θ) | θ/θ: Source arm and/or Detector arm<br>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) |

### 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.

## 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”).

## 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.

## 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.

## 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.

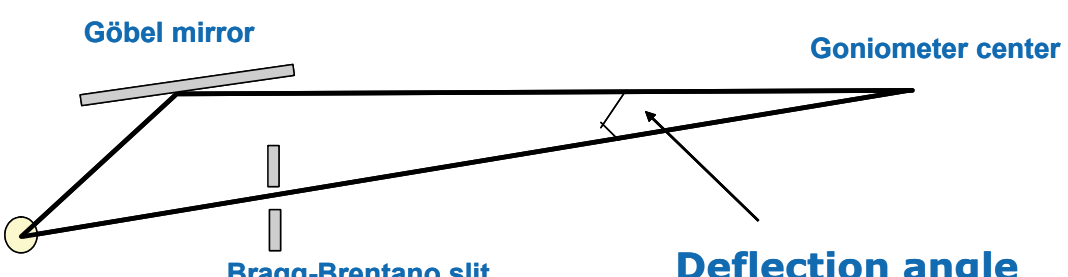
## General procedure for optimizing reference positions and deflection angles

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.

## The 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.



## 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”.

## 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.

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.

# The 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 and 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:

a.) first set up of the diffractometer at customer side

b.) change of the adapter plate

c.) change of the x-ray tube

d.) change from point focus to line focus

## 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 optics), Montel mirrors and primary side monochromators (eg. Johansson 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. Move Theta and 2Theta to 0°. Make sure that the beam is not blocked by the sample holder. Use suitable absorbers and Kß filter. The measured countrate should be in the range of $10^3$cps to $10^5$ cps (counts per second). Use no detector slit and no anti scatter slit. Open fixing screw of the focus position. Use suit-

able detector settings (for Cu radiation usually 40kV/40mA). Start the ratemeter in DIFFRAC.COMMANDER and open the shutter. Adjust focus position in steps of ¼ turns to achieve maximum intensity recorded with the ratemeter. Check focus position with all other monochromatic optics. One focus position should fit for all monochromatic optics which need this tube and focus type (point/line).

# Glass slit alignment

In order to ensure reliable operation of the diffractomer, 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.

## Zero Point Definition of the θ Scale

### Measurement

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

### Consequence

The basic alignment of the θ scale is o.k. if the angle $\theta_{Max}$ 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 $\theta_{Max}$. 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.

## Zero Point Definition of the 2θ Scale

**Measurement**

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

# Standard and TWIN/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/TWIN configuration**: fully automated, software-controlled switching between different beam paths. Note that the primary TWIN optics requires a 560 mm measurement diameter.



**Caution!**

Each of the two configurations, standard and TWIN/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.



**Note**

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.

# 1. Standard Configuration

**Characteristics:**

Measurement diameter 500mm, sometimes 560mm

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

## Dedicated Bragg-Brentano Setup (Standard)

**Characteristics (cf. Figure 129:)**

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 0-D and 1-D detectors are shown in Figure 129 and Figure 130 respectively.

See also special setup with Johansson monochromator on page 70.

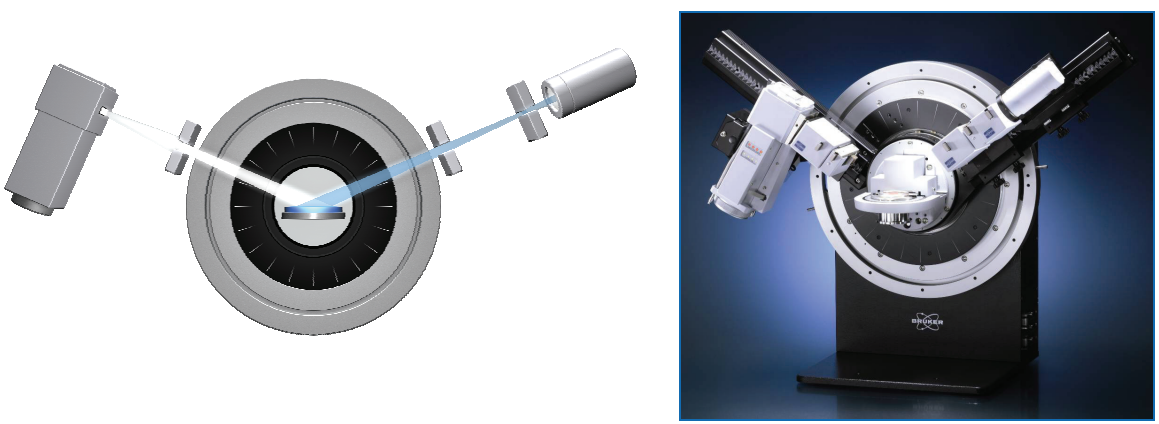


Figure 129: Basic Bragg-Brentano setup for phase identification and structure determination. Plug-in slits and Scintillation counter are mounted in this example.

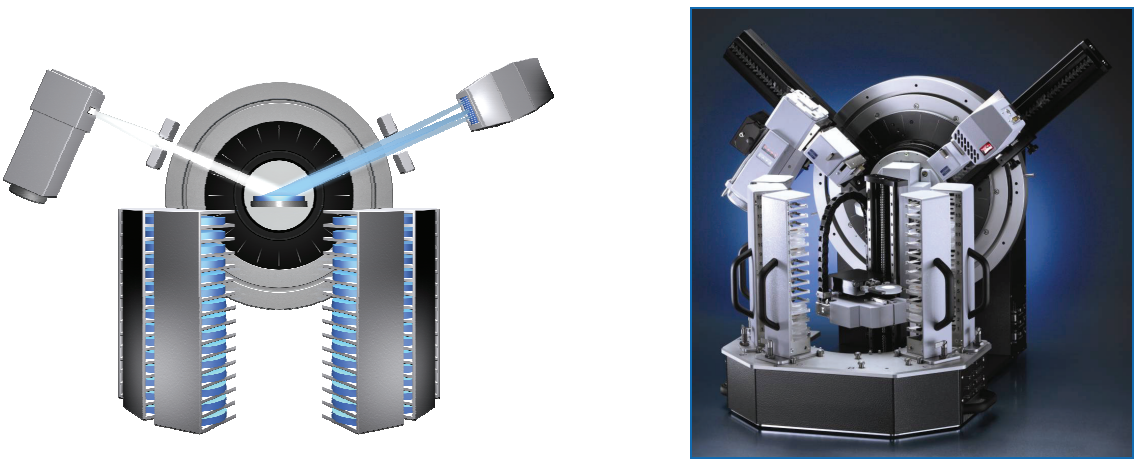


Figure 130: Bragg-Brentano setup for high throughput – AUTO-CHANGER and LYNXEYE.

## Transmission Setup (Standard)

**Characteristics (cf. Figure 131):**
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 in Figure 131. Note the mounted goniometer podium and the position of the air-scatter screen.

See also special setup with Johansson monochromator on page 70.

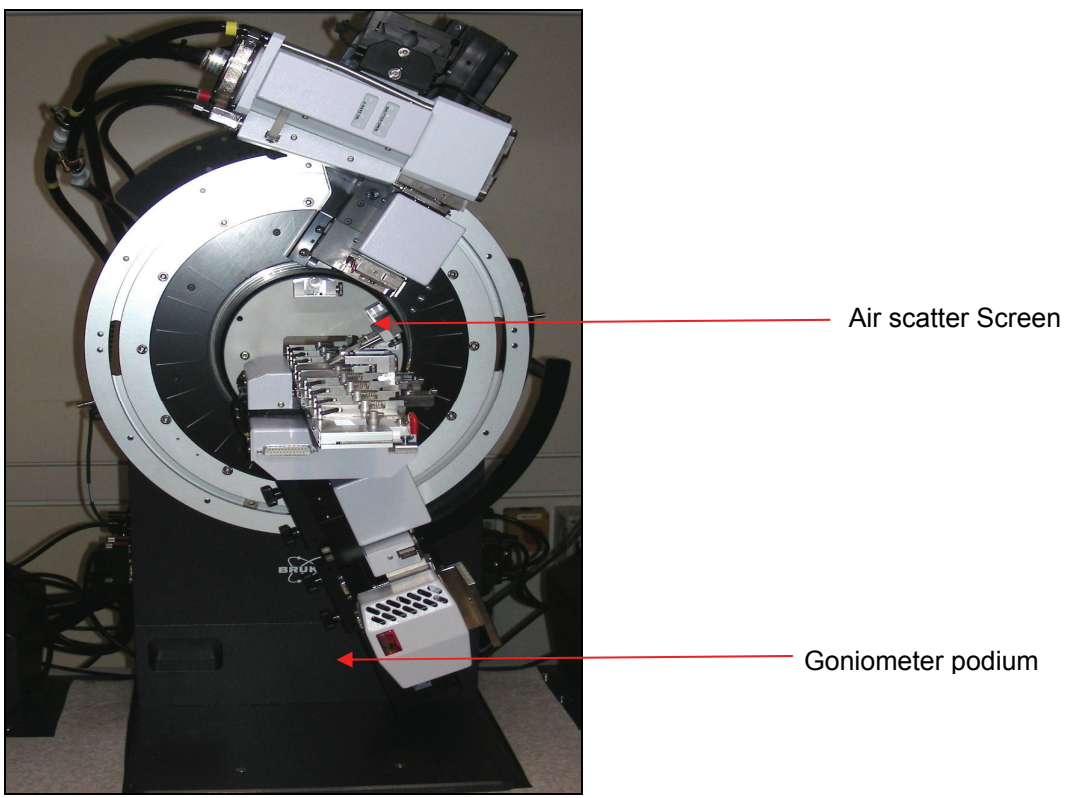



Figure 131: Typical setup for transmission measurement.

## X-ray Reflectometry Setup (Standard)

**Characteristics (cf. Figure 132):**
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

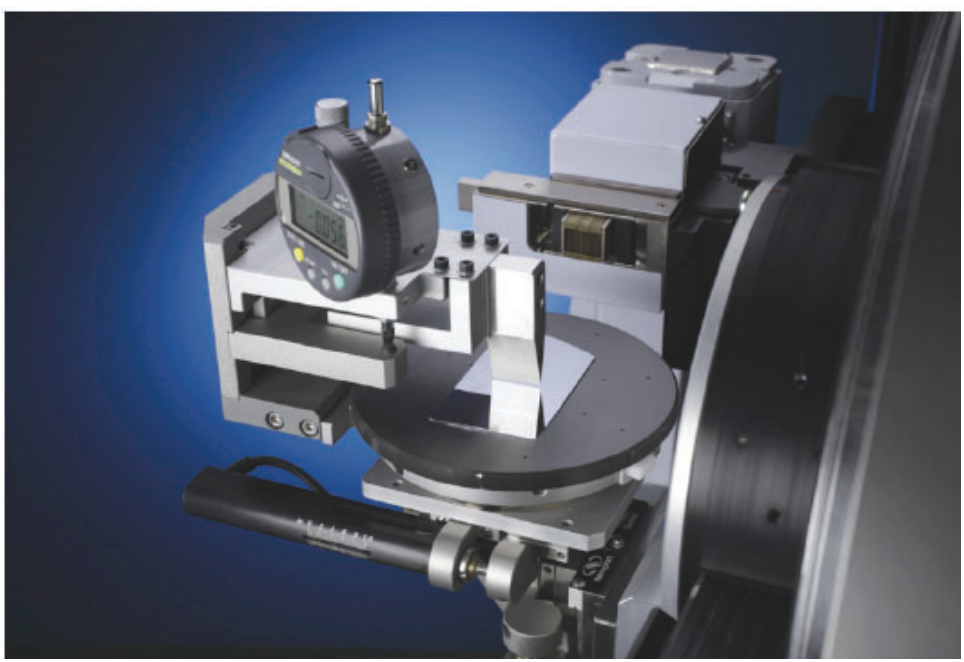


Figure 132: The sample area of a reflectometry setup, showing a compact XYZ stage and a knife edge.

## Non-Ambient Setup (Standard)

**Characteristics (cf. Figure 133):**
Measurement diameter starting from 500mm.

Sample space requirements approximately the same as a standard sample carrier.



Figure 133: Non ambient setup with VÅNTEC-1.

## Stress and Texture Setup (Standard)

**Characteristics (cf. Figure 134, Figure 135):**
Measurement diameter starting from 500 mm (depends on sample stage)

Sample Stages: Compact Eulerian Cradle, compact XYZ, Standard, Rotary, FLIP-STICK, AUTO-CHANGER, Capillary, Non Ambient chambers

UBC collimator (50µm … 2mm)

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 compact Eulerian cradle and point spot on sample. This is achieved either point with a 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

**Texture:** always requires compact Eulerian cradle and point shaped X-ray spot on the sample

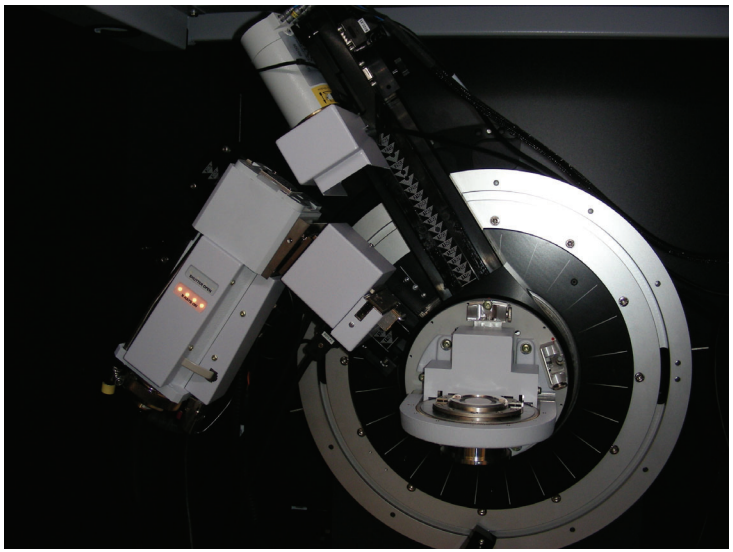


Figure 134: Residual stress detemination in isoclination mode.

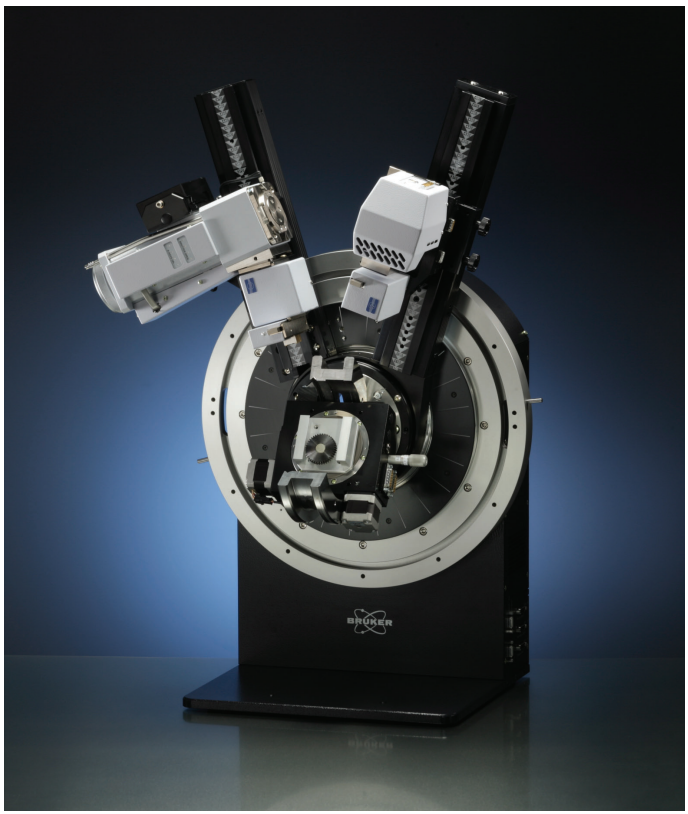


Figure 135: Example for Texture setup.

## 2. TWIN/TWIN Configuration

**Characteristics (cf. Figure 136):**

- 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 provide 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

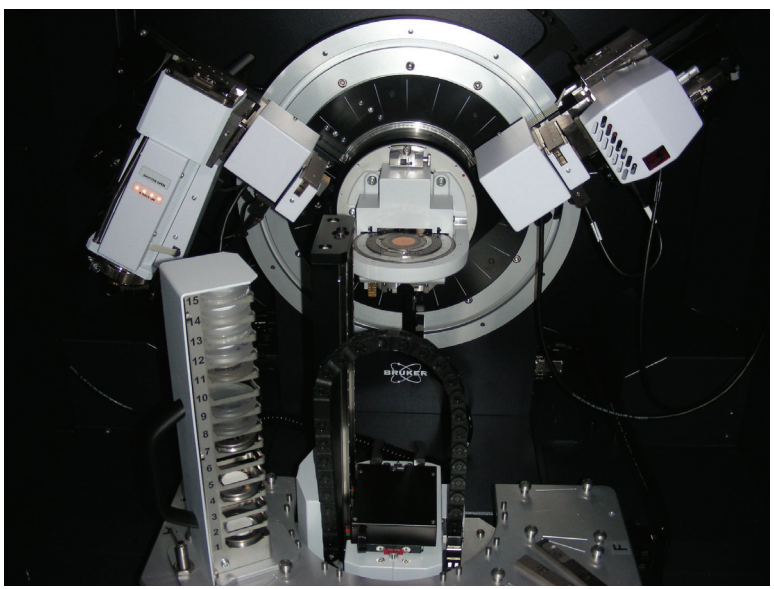


Figure 136: A TWIN/TWIN setup, which allows pure software switching between different applications (Bragg-Brentano, parallel beam, Grazing Incidence Diffraction, X-ray reflectivity, Microdiffraction). In this example combined with an AUTO-CHANGER sample stage.

## X-ray Reflectometry Setup (TWIN/TWIN)

Figure 137 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 0-D mode.

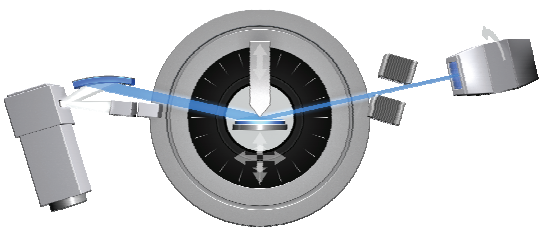


Figure 137: X-ray Reflectometry Setup (TWIN/TWIN).

## Bragg-Brentano Setup (TWIN/TWIN)

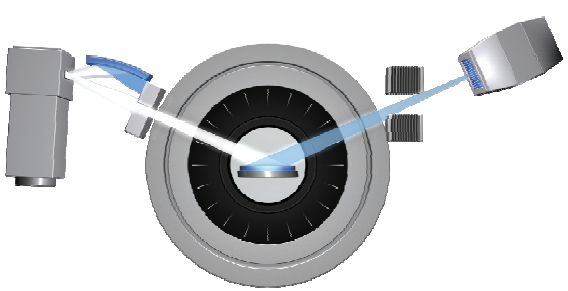


Figure 138: TWIN/TWIN Bragg-Brentano mode. Primary TWIN set to Göbel mirror and secondary side TWIN set to equatorial Soller.

## Grazing Incidence Setup (TWIN/TWIN)

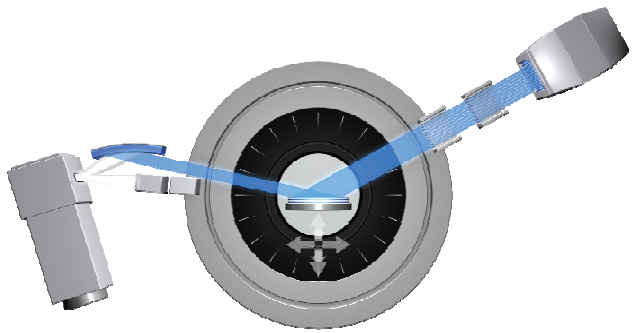


Figure 139: TWIN/TWIN - Grazing Incidence Diffraction: Primary TWIN set to Göbel mirror and secondary side TWIN set to equatorial Soller.

## Microdiffraction Setup (TWIN/TWIN)

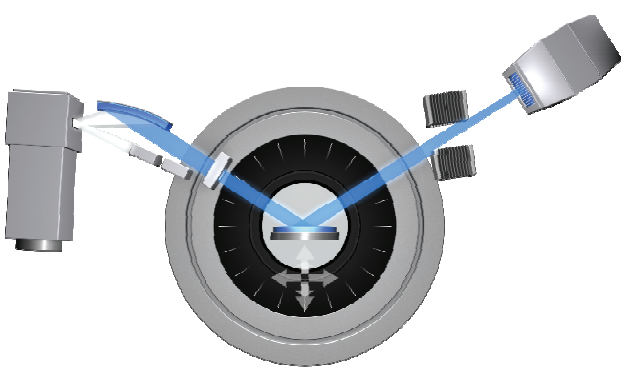


Figure 140: TWIN/TWIN – Microdiffraction: Primary TWIN set to Göbel mirror, Microslit and secondary side TWIN set to slit.

# References

| | |
|---|---|
| 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 |
| Supplement folder | DOC-M88-ZXX152 |
| AUTO-CHANGER User Manual | DOC-M88-EXX101 |
| LYNXEYE User Manual | DOC-M88-EXX095 |
| Motorized Compact XYZ stage User Manual | DOC-M88-EXX124 |
| VÅNTEC-1 User Manual | DOC-M88-EXX072 |
| SOL-XE User Manual | DOC-M88-EXX113 |
| Temperature Chambers User Manual | DOC-M88-EXX047 |
| 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 |
| Verification Booklet | DOC-M88-EXX157 |

**Artisan Technology Group** is an independent supplier of quality pre-owned equipment

## Gold-standard solutions

Extend the life of your critical industrial, commercial, and military systems with our superior service and support.

## We buy equipment

Planning to upgrade your current equipment? Have surplus equipment taking up shelf space? We'll give it a new home.

## Learn more!

Visit us at **artisantg.com** for more info on price quotes, drivers, technical specifications, manuals, and documentation.

Artisan Scientific Corporation dba Artisan Technology Group is not an affiliate, representative, or authorized distributor for any manufacturer listed herein.

We're here to make your life easier. How can we help you today?

(217) 352-9330 | sales@artisantg.com | artisantg.com

ARTISAN® TECHNOLOGY GROUP