

Red arrows show the hardware parts we have.



Instruction Manual

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Before Using the Product

Read this manual cover to cover before attempting to use SmartLab SE. Carefully read the *Guidelines for* safe use of the X-ray diffractometer described in the beginning of this manual.

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Overview

SmartLab SE, automated multipurpose X-ray diffractometer (hereinafter referred to as SmartLab SE), is an X-ray diffraction system equipped with a theta-theta goniometer featuring a horizontal sample mount.

- Out of various optics available, the optics suited for the sample or the measurement purpose can be selected simply by exchanging the unit.
- <u>In addition to</u> the fixed sample platform for powder sample measurements, the sample section is capable of combining with a variety of attachments such as for sample surface plane measurements or In-situ measurements can be installed.
- The optics and the sample section are controlled via the integrated software package SmartLab Studio II (hereinafter referred to as SmartLab Studio II).

SmartLab Studio II guides the user through the procedures of optical device settings, optical and sample alignment, and measurement condition settings, and also executes analysis after the measurement.

- Since the software for measurement and analysis are not separate, the analysis result can be obtained in a seamless procedure.
- All optical devices are equipped with a sensor. This lets SmartLab Studio II guide you to prepare the optical device configurations that is suitable for the measurement purpose.
- Measurement by conventional manual operation (Pro Control) is also available.

Taking advantage of these features, SmartLab SE allows even inexperienced users to perform measurements in the same conditions as a specialist would, just by selecting a measurement purpose.

Features

Horizontal sample mount theta-theta goniometer

A theta-theta goniometer enables omega scans, 2-theta/omega scans, and 2-theta scans with the sample kept oriented horizontally*. In the case of samples such as a large wafer, the horizontal positioning of a sample minimizes the distortion effects caused by the weight of the sample and also reduces the possibility of a dropped sample.

* Deviation from horizontal orientation may occur if sample orientation is adjusted.

Sample section

The sample section consists of three units: attachment platform, attachment and sample holder.

The attachment platform is selectable either from the fixed sample platform for powder samples or universal Z attachment platform with Z-axis adjustment mechanism. On the universal Z attachment platform, a variety of attachments or attachment bases such as for sample rotation/spinning, and for In-situ measurement can be installed.

The attachment bases which are installed on the universal Z attachment platform can combine with different attachment heads such as for mapping measurement and random orientation measurement. These attachment bases, attachments, and attachment heads can be easily changed.

X-ray generator

The specifications of the generator are summarized in the following table. The maximum rated load depends on the type (target and focus) of X-ray tube. For details, refer to the instruction manual of the X-ray tube.

	Max. load	Variable range of tube voltage	Variable range of tube current
Casled tube	2 I-W/	20 kV to 60 kV	2 mA to 60 mA
Sealed tube	JKW	(1 kV step)	(1 mA step)

Table Specifications of X-ray generator

Cross beam optics (CBO)

Cross beam optics (CBO) is a system that enables switching between two types of beams depending on the sample and the measurement purpose just by changing the selection slits. The CBO unit has the following types: CBO, CBO-E, and CBO- α .

CBO

Switches between divergent beam and parallel beam*.

- CBO-E Switches between divergent beam and convergent beam.
- CBO-α

Switches between divergent beam and monochromatized divergent beam.

 The parallel beam obtained by CBO can be used to SAXS optics for nano-structural measurements or micro-area measurement optics.

Incident optics section

The incident optics section consists of three units. By changing the units, various geometries such as para-focusing, parallel beam (including SAXS and micro area measurement), convergent beam, and monochromatized divergent beam can be used. Each type of CBO units contains the multilayer mirror and the 2-bounce monochromator that have an adjustment mechanism to be controlled via SmartLab Studio II (automatic alignment).

- Incident optics unit #1 Incident beam path dedicated for para-focusing optics or CBO units can be selected.
- Incident optics unit #2 Soller slit, 2-bounce monochromator (with Soller slit), or CBO-f can be selected.
- Incident slit Variable incident slit box, including the length-limiting slit, or collimator can be selected.

Receiving optics section

The receiving optics section consists of six optical units. By changing the units, backgrounds can be reduced and the resolution suited for the measurement purpose can be easily obtained.

Scattered radiation protector

This is used in combination with the receiving slit box.

Receiving slit # 1

Filter box, receiving slit box, and can be selected. The $K\beta$ filter can be installed on the both items.

- Receiving optics unit # 1 Parallel slit analyzers can be selected.
- Receiving optics unit # 2 Soller slits can be selected.
- Receiving slit # 2 A virtual receiving slit which limits the detection area of the detector.
- Attenuator For the adjustment of X-ray intensity.

Detector

In the detector section, the D/teX Ultra250 1D detector is equipped. Detecting elements are arranged to effectively detect and integrate X-ray intensities, letting measurements in broad range be completed in a short period time. D/teX Ultra 250 and diffracted beam monochromator for D/teX can be used in combination depending on the measurement purpose. D/teX Ultra 250 can also be changed to HyPix-400 2D detector.

Optics switching system

Optics alignment results are retained by SmartLab Studio II. Even when the optics are changed, the previously stored alignment results are used, eliminating the need for realignment.

Optical device detection

SmartLab Studio II can check the conditions of the following optical devices used for measurement:

- · Selection slit in CBO unit
- · Type of incident Soller slit or 2-bounce monochromator
- Width of incident slit
- · Type of length-limiting slit
- Presence/absence of collimator
- · Width of receiving slit
- · Type of analyzer
- · Type of receiving Soller slit
- Type of detector and scan mode

· Presence/absence of diffracted beam monochromator,

SmartLab Studio II checks whether the necessary optical devices are installed and displays a guidance for the optical configurations suitable for the user's intended application. The measurement data file stores parameters for the optical devices during measurement to improve data reproducibility and traceability.

Control software (SmartLab Studio II)

"SmartLab Studio II" integrates all optical alignments, measurements, and analyses in a single software package.

In hardware control, the program guides the user through required measurement procedures and condition-setting processes. Optical device configurations, optics alignment methods, sample alignment methods, and measurement conditions specific to various measurement needs are grouped in units called Package Measurements. By selecting an appropriate Package Measurement for the analysis purpose, the user is guided through the procedures of optics alignment, sample alignment, and data measurement. The program provides the optimal alignment and measurement conditions for the desired analysis. The software also allows customization of alignment and measurement conditions for special measurement needs. Pro control is also possible. The software is designed to meet a wide range of user needs.

In data analysis, the hardware configuration and the data analysis are linked. After the data collection in the measurement plugin, the analysis can be easily started just by selecting the desired analysis plugin. Flow bars which list the functions necessary for each application are also made ready. User can save analysis results and create reports just by proceeding through the analysis steps along the flow bar. Data transfer between different applications is designed to be easily performed. The flow bar is designed to be easy to use even for users familiar with X-ray diffraction measurements.





General view of SmartLab SE

①Control PC	PC used to control SmartLab SE.
②Power key	Power key used to start and stop SmartLab SE.
③Emergency off switch (EMO) *1	Press this switch in the event of an emergency to cut off the power supply to the main unit.
(4)Door	This door is opened to change samples and optical devices.
⑤X-rays on lamp	Lights when X-rays are generated.
©Observation window ^{*2}	Window used to observe the inside of the main unit.
⑦Power-On indicator	Lights when SmartLab SE is powered on.
®X-rays-On indicator	Lights when X-rays are generated.
Door-Lock indicator	Lights when the door is locked
①Alarm indicator	Flashes when an error occurs.

*1 Emergency off switch (EMO) is optional.

*2 The observation window used in the SmartLab SE is made of high lead content glass. Please be very careful to handle it. (See "Observation window")



Rear view of SmartLab SE

⁽¹⁾ Power input connector	Connect from external power supply.
⁽¹⁾ Circuit breaker	Circuit breaker to apply the power to the main unit.
(13)Inlet and outlet for cooling water	Connect the cooling water hoses from the water chiller here.
⁽¹⁴⁾ Connectors for water chiller	Connect the control cable of the water chiller (bypass valve) here.
(5)Control cable hole	Pass the control cables for SmartLab SE through this hole.
^(f) Cooling water filter unit ^{*1}	Use this filter unit when not using the water chiller.

*1 The cooling water filter unit is optional.



Gomometer

①Theta_s arm	Arm for controlling X-ray beam incident angle.
②X-ray generator section	X-ray generating device.
③Incident optics section	Optical device for achieving desired incident X-ray conditions.
④Theta_d arm	Arm for controlling the X-ray detector angle.
©Receiving optics section	Optical device for achieving desired X-ray receiving conditions.
[©] Detector section	X-ray detector.
⑦Sample section	Adjusts the position and orientation of the sample to be measured.

Theta_s arm



Incident beam path/CBO unit \rightarrow Incident optics unit # 1

Selection slit (PB, BB. SA, etc.) $\rightarrow \underline{CBO \text{ unit}}$

Soller slit 2.5° / CBO-f \rightarrow Incident optics unit # 2

Standard incident slit/Length-limiting slit/Collimator adapter → Incident slit

Theta_d arm



Sample section (Universal Z attachment platform)



10 SmartLab SE: Automated Multipurpose X-ray Diffractometer

X-ray generator section



X-ray generator section

①Cooling water piping	Connect the X-ray generator cooling water tube.
②X-ray tube	X-ray generating source.
③Shutter unit	Controls X-ray emissions.

Incident optics section



Incident optics section

①Incident connector box	Connect the control cables of the optical devices here.
②Incident optics unit # 1	Install an incident beam path or a CBO unit, etc. here.
③Incident optics unit # 2	Install an incident Soller slit, monochromator, etc. here.
④Incident slit	The slit on the incident side.

Receiving optics section



Receiving optics section

①Receiving connector box	The control cables for the optical devices connect here.
②Receiving slit	Install a filter box or a variable slit.
③Receiving optics unit # 1	Install a parallel slit analyzer.
④Receiving optics unit # 2	Install a parallel slit.
⑤Attenuator	Adjusts X-ray intensity.

Detector section





Detector section

①Detector	Detects X-rays.
②Detector adapter	A detector holder There are two types: fixed type and detachable type.



Sample section



Sample section

①Fixed sample platform	Sample platform for glass sample holder or aluminum sample holder. Other attachments cannot be used.
②Universal Z attachment platform	Attachment platform with Z axis alignment mechanism. Various attachments can be installed.
③Attachment base	Attachment base to be mounted on the universal Z attachment platform.
(4)Attachment head	Attachment head to be mounted on the attachment base.
⑤Sample plate	Plate on which sample holder is placed. Attach this plate to the attachment head.

Software Configuration

Measurements and analyses are controlled via the SmartLab Studio II integrated X-ray analysis software.

Control software

XRD Measurement plugin

Used to control SmartLab and perform measurements.

Basic data processing

Data Manager plugin	Performs basic data processing such as smoothing, background subtraction, etc.
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Analysis

Powder XRD plugin Basic: A00002241/A00002250 Qualitative analysis: A00002242/A00002251 Quantitative analysis: A00002243/A00002252 Comprehensive analysis: A00002244/A00002253 Rietveld analysis: A00002245/A00002254 Structure determination: A00002246/A00002255 Retained austenite analysis: A00009287/A00009288 Respirable silica analysis: A00009289/A00009290	Performs powder diffraction data analysis. This plugin contains a basic part and seven optional parts.
XRR plugin A00002238/A00002247	Performs X-ray reflectivity data analysis to evaluate thickness, density, and interface roughness of thin film samples.
HRXRD plugin A00002239/A00002248	Performs simulation and analyses of rocking curve and reciprocal space mapping.
MRSAXS plugin A00004213/A00004214	Performs particle size/pore diameter distribution analysis of powder, solution, or thin film.
Stress plugin A00004211/A00004212	Calculates stress based on 1D or 2D data obtained by SmartLab.
Texture plugin Basic: A00008482/A00008483 ODF Analysis: A00008484/A00008485	Basically displays pole figure data and performs analysis, and optionally performs ODF analysis as well.
XRD DSC plugin A00007091/A00007793	Displays and analyzes XRD patterns and DSC charts.
PDF plugin A00008319/A00008320	Calculates radial distribution function and pair distribution function.
Data Visualization plugin A00008321/A00008322	Displays and analyzes massive data obtained from various measurements of XY mapping, temperature control, and XRD-DSC.
Cluster analysis A00004215/A00004216	Classifies measurement datasets into several groups based on their similarity.
Audit trail & ER/ES system A00004217	An audit trail & electronic record/electronic signature system that supports CFR21 Part 11 (U.S. FDA) and Annex 11 (EU GMP).

Turning On and Off SmartLab SE

This chapter describes the procedures for turning on and off SmartLab SE.

Turning on SmartLab SE

1 Make sure that the power key is inserted on the front side of the main unit.

If it is not, insert the power key into the main unit.

- **2** Turn the power key clockwise to the position (further to the right than the position).
- **3** The key automatically moves to the position and the Power-On indicator turns on.

The key cannot be removed at this state.











Power-On indicator

Turning off SmartLab SE

If SmartLab SE is unused for a while, turn off SmartLab SE according to the following procedure.

1 Make sure that the status of SmartLab SE are the following.

- · Measurements have been completed.
- · X-ray generation is turned off.
- All controls have ended.
- **2** Turn the power key counterclockwise to the \bigcirc position.





3 Make sure that the Power-On indicator has turned off.





Tip

This turn-off procedure completely shuts down the SmartLab SE internal power supply.

Opening and Closing the Door

In order to prevent human exposure to X-ray radiation, the shutter and door are equipped with an interlocking function. To ensure safety, the door is automatically locked while the shutter is open for measurements, etc. Conversely, while the shutter is close, the door is unlocked and can be safely opened anytime.

Opening the door



While the shutter is open for measurements, etc., the door is locked and cannot be opened.

1 Make sure that the shutter is close and the Door-Lock indicator is off.



2 Open the door.



The shutter cannot be opened while the door is open.

Closing the door

- **1** Close the door.
- **2** When the door is properly closed, the instrument is then ready for operation.

Emergency off switch (EMO)

Press this switch in the event of an emergency to cut off the power supply to the main unit.

To cancel the emergency stop status, turn the button clockwise. After confirming safety, turn the power key to the $\[\]$ position to restart SmartLab SE. (see <u>Turning on SmartLab SE</u>).



After restarting SmartLab SE, restart the control PC and SmartLab Studio II.



Emergency off switch (EMO) (optional:A00010189)

Optical Device

This section introduces the names of optical devices and describes how they are installed.

Connecting and removing optical device cable

By connecting the cable from each optical device to the corresponding connector on the connector box, SmartLab Studio II recognizes the optical configuration.

Connecting optical device cable

- **1** Check the optical device name on the cable end and also check the name on the corresponding connector of the connector box.
- **2** Hold the part of the cable end where the optical device name is indicated. Connect it to the corresponding connector of the connector box so the name on the cable end faces upward. (The cable end slides and then is securely connected to the connector.)



Removing optical device cable

1 Hold the sliding part of the cable end, and remove the cable from the connector.



Incident optics unit # 1

The optical device used to select the type of incident beam is installed on the incident optics unit #1.

The incident beam path for para-focusing optics or CBO unit can be switched.

Incident beam path

The incident beam path is used only when the monochromatized divergent beam for para-focusing optics is selected. This cannot be used with the CBO unit.



Incident beam path

CBO unit

Just by changing the selection slits, the beams can be switched between divergent beam and the beams formed by using a multilayer mirror (parallel/convergent/monochromatized divergent). Parallel beam can also be formed into a narrow beam for small-angle scattering measurements or a point beam for micro area measurements by using the appropriate selection slits. Fine adjustments of the multilayer mirror can be performed via SmartLab Studio II.

Unit name	Abbreviation	Illustration
CBO (for Cu only) (optional: C484-0001-3-31) Applicable only to Cu wavelength, and enables the switching between divergent and parallel beams.	CBO (Cu)	
CBO-E (for Cu only) (optional: C484-0001-3-38) Applicable only to Cu wavelength, and enables the switching between divergent and convergent beams.	СВО-Е	
CBO-α (for Cu only) (optional: A00004622) Applicable only to Cu wavelength, and enables the switching between divergent and monochromatized divergent beams.	CBO-α	
CBO-Cu/Co (for Cu and Co) (optional: C267-0017) Applicable to Cu and Co wavelengths, and enables the switching between divergent and parallel beams.	CBO (Cu/Co)	
CBO-Mo (for Mo only) (optional: A00001716) Applicable only to Mo wavelength, and enables the switching between divergent and parallel beams.	CBO-Mo	

Table CBO unit

1 Attach the CBO unit to the shutter. Press the CBO unit against the surface in the back and secure the unit in place with the Allen wrench provided.



2 Connect the cable into the CBO connector of the incident connector box.

The connector name is indicated on the cable end.





3 Insert a selection slit into the CBO unit.

Optics name	Corresponding CBO	Abbreviation	Illustration	
For para-focusing	CBO(Cu) CBO(Cu/Co) CBO-E CBO-α	BB	i	<
For parallel beam (included in the CBO(Cu) and CBO(Cu/Co) units)	CBO-Mo CBO(Cu) CBO(Cu/Co)	РВ	i	<
For convergent beam (included in the CBO-E unit)	CBO-E	СВ	i	0
For monochromatized divergent beam (included in the CBO-α unit)	CBO-α	DB		
For SAXS (optional: 2680J111)	CBO(Cu) CBO(Cu/Co)	SA	: E 4	<
For micro area measurements (optional: 2680G111)	CBO(Cu) CBO(Cu/Co)	МА	:	
For micro area measurements (optional: 2431C304)	CBO(Cu) CBO(Cu/Co)	PB 0.5		\bigcirc
For para-focusing (Co) (included in the CBO(Cu/Co) unit)	CBO(Cu/Co)	BB Co	:	
For parallel beam (Mo) (included in the CBO(Mo) unit)	CBO(Mo)	РВ Мо	i	

Table Selection slits

The type of selection slit installed can be identified by SmartLab Studio II.

Tip

Incident optics unit # 2

The optical device used to condition the incident X-ray beam is installed on the incident optics unit #2.

As the optical device, either the incident Soller slit or the 2-bounce monochromator can be selected.

Incident parallel slit adapter (IPS adapter)

This adapter is used to install an incident Soller slit.



Incident parallel slit adapter

1 Install the incident parallel slit adapter to the CBO unit by sliding it down from the top section of the CBO unit. Secure it in place with the Allen wrench provided.



2



Connect the cable to the IPS connector on the incident connector box.

3 Install the incident Soller slit on the IPS adapter and secure it in place with the Allen wrench provided.



The connector name is indicated on the cable end.

Туре	Aperture	Abbreviation	Illustration	
Open (optional: 2680D115)	Open	Soller_Slit_open		<
Soller slit (optional: 2680D116)	5°	Soller_Slit_5.0 deg		
Soller slit (standard)	2.5°	Soller_Slit_2.5 deg		<
2D slit (optional) For Cu: A00007629 For Cr: A00007753 For Co: A00007754 For Fe: A00007755 For Mo: A00007756	0.5 mm (Kβ filter included)	2D_Slit		<

Table Incident Soller slit

Tip

The type of installed incident Soller slit on the adapter can be identified by SmartLab Studio II.

Ge(220) 2-bounce monochromator

This optical device produces a monochromatized X-ray beam using a 2-bounce channel cut crystal. Fine alignments of the channel cut crystal are performed via SmartLab Studio II. A dedicated parallel slit can be installed next to the channel cut crystal (the side closer to the sample).



Ge(220) 2-bounce monochromator (optional: 2680B113)

1 Attach the Ge(220) 2-bounce monochromator to the CBO unit by sliding it down from the top section of the CBO unit. Secure it in place with the Allen wrench provided.



2 Connect the cable to the IPS connector on the incident connector box. The connector name is indicated on the cable end.



3 Install a parallel slit on the Ge(220) 2-bounce monochromator. Secure it in place with the Allen wrench provided.


Туре	Aperture	Abbreviation	Illustration
Open (included with the 2-bounce monochromator)	Open	Soller_slit_open	P
Soller slit (optional: 2680D213)	5.0°	Soller_slit_5.0 deg	a la ma la
Soller slit (optional: 2680D212)	2.5°	Soller_slit_2.5 deg	a la la

Table Parallel slits for Ge(220) 2-bounce monochromator



The type of parallel slit installed on the adapter can be identified by SmartLab Studio II.

CBO-f unit

This optical device is used in measurements of micro area and micro amount of sample. The parallel beam in the longitudinal direction is converged onto the sample.

Fine alignment of the CBO-f unit is performed via SmartLab Studio II.



CBO-f unit (optional: 2220C302)

1 Install the CBO-f unit to the CBO unit by sliding it down from the top section of the CBO unit, and secure it in place with the Allen wrench provided.



2 Connect the cable to the IPS connector on the incident connector box. The connector name is indicated on the cable end.



Incident slit

This slit is installed on the Zs axis on the theta_s arm.

Standard incident slit box

This slit box adjusts the slit width to control the beam divergence angle (for the para-focusing method) or the beam width (for the parallel beam method). For SAXS measurements, this slit guards against parasitic scattering. A length-limiting slit can also be inserted to limit the beam length along the longitudinal axis.



Standard incident slit box

1 Attach the standard incident slit box to the Zs axis. Secure it in place with the Allen wrench provided.



2 Connect the cable to the IS connector on the incident connector box. The connector name is indicated on the cable end.



3 Insert a length-limiting slit to control beam length along the longitudinal axis.



Length of limiting slit	Abbreviation	Illustration	
15 mm (optional: A00007633)	15		
10 mm	10		<
5 mm	5		<
2 mm	2		
0.5 mm (optional: C295-0069)	0.5		•

Table Length-limiting slits

Tip

The type of inserted length-limiting slit can be identified by SmartLab Studio II.

PB Collimator adapter

This adapter is used when combining a collimator with parallel beams formed by a multilayer mirror or the beam formed by CBO-f. Collimators can be installed by using the collimator adapter.



PB Collimator adapter (optional: A00004692)



1 Install the PB collimator adapter to the Zs axis and secure it in place with the Allen wrench provided.

2 Connect the cable to the IS connector on the incident connector box. The connector name is indicated on the cable end.



3 Insert the collimator into the collimator adapter.

Table Collimators

Pinhole diameter	Abbreviation	Illustration
Long type φ0.8 mm (optional: 2431E107)	0.8	C Be
Long type φ0.5 mm (optional: 2431E106)	0.5	
Short type φ0.8 mm (optional: A00003336)	0.8	10 Cra
Short type φ0.5 mm (optional: A00003335)	0.5	





Use long type collimators only with the combinations of items below.

- Standard attachment head + collimator sample plate
- XY-20 mm attachment head + collimator sample plate (The total amount of the X, Y motion must be less than 10 mm. $\sqrt{(X^2+Y^2)} < 10$ mm)
- RxRy attachment head + collimator sample plate

When attachment heads and long type collimator are used at the same time, some combination of items may cause a collision between the end of the collimator and the attachment main body, sample, or sample plate, resulting in damage to them.

Use short type collimators for combination of items other than those listed above.

Point optics unit

This optics unit is used for measurements using point beams. It is added after switching the X-ray tube from line focus to point focus.



Point optics unit (optional: A00007634)

Point slit adapter

This adapter is a base to mount a selection slit. The point focus selection slit (BB φ 0.5 mm) is used.



Selection slit (BB φ 0.5 mm) and point slit adapter

Incident attenuator

This attenuator is used to adjust X-ray intensities at the incident side.



Incident attenuator

BB collimator adapter

This adapter is a base to mount the BB collimator. The $K\beta$ filter can be inserted into the BB collimator adapter.



BB collimator adapter

Pinhole diameter	Abbreviation	Illustration
Long type φ0.8 mm (optional: 2431E107)	0.8	
Long type φ 0.5 mm (included in point optics unit)	0.5	
Short type φ0.8 mm (optional: A00003336)	0.8	· Ca
Short type φ0.5 mm (optional: A00003335)	0.5	ica

Table	Collimator
Table	Commator

Incident K_β filter

This is the K β filter for the incident side. Place the K β filter in the absorber case and insert it into the BB collimator adapter on the X-ray source side.



Incident Kß filter and absorber case

Use long type collimators only with the combinations of items below.

- Standard attachment head + collimator sample plate
- XY-20 mm attachment head + collimator sample plate
 - (The total amount of the X, Y motion must be less than 10 mm. $\sqrt{(X^2+Y^2)} < 10$ mm)
- RxRy attachment head + collimator sample plate

When attachment heads and long type collimator are used at the same time, some combination of items may cause a collision between the end of the collimator and the attachment main body, sample, or sample plate, resulting in damage to them.

Use short type collimators for combination of items other than those listed above.

Receiving slit # 1

This unit is installed on the sample side of the theta_d arm.

Filter box

A K β filter 1D can be inserted to remove $K\beta$ X-rays from diffracted beam.



Filter box

1 Attach the filter box to the rail of the theta_d arm. Secure it in place with the Allen wrench provided.





2 Connect the cable to the RS1 connector on the receiving connector box. The connector name is indicated on the cable end.

3 A K β filter 1D can be inserted.



Receiving slit box

In para-focusing optics, this slit box adjusts the slit width to control the convergence angle of the beam. In parallel beam optics and SAXS optics, it functions as the first slit in a double-slit receiving system. The slit width and the vertical position are adjusted in SmartLab Studio II. K β filter 1D can also be inserted to eliminate the K β beam contained in the diffraction beam.



Receiving slit box (optional: C484-0001-3-30)

1 Attach the receiving slit box to the rail of the theta_d arm. Secure it in place with the Allen wrench provided.





2 Connect the cable to the RS1 connector on the receiving connector box.

3 The $K\beta$ filter 1D can be inserted.



- **4** The installation position can be read from the ruler on the theta_d arm.

Scattered radiation protector

The scattered radiation protector is used to reduce the scattered X-ray beam entering from outside the X-ray optical path. It is effective especially on reducing the background on the low angle side. It is used in combination with the receiving slit box.



(The end section can be removed)

Scattered radiation protector (optional: A00000511)



Scattered radiation protector (optional:A00008462)



1 Attach the scattered radiation protector to the rail of the theta_d arm. Secure it in place with the Allen wrench provided.

Receiving optics unit # 1

This unit is installed next to the receiving slit # 1 (the side closer to the detector). A parallel slit analyzer can be installed on receiving optics unit # 1.

Receiving optical device adapter (ROD adapter)

This adapter is used to install a parallel slit analyzer (PSA). A parallel slit analyzer is installed so that the slits inside are parallel to the scanning direction of theta_d axis, that is, in the 2 theta resolution direction.



Receiving optical device adapter

1 Attach the receiving optical device adapter to the rail of the theta_d arm. Secure it in place with the Allen wrench provided.





2 Connect the cable to the ROD connector on the receiving connector box.

The connector name is indicated on the cable end.



3 Attach a parallel slit analyzer to the ROD adapter. Secure it in place with the Allen wrench provided.



Aperture	Length	Abbreviation	Illustration	
Open (standard)	45 mm	PSA_open	QO	<
0.5° (optional: 2680F112)	45 mm	PSA_0.5 deg		<
0.228° (optional: 2680F117)	90 mm	PSA_0.228 deg		0
0.114° (optional: 2680F111)	90 mm	PSA_0.114 deg		

Table Parallel slit analyzers



In principle, when the PSA 0.114 deg or PSA 0.228 is installed, do not use the receiving parallel slit. To use the receiving slit box, the receiving parallel slit adapter, the attenuator, and the detector must be moved to the right.

Tip

The type of parallel slit analyzer installed on the adapter can be identified by SmartLab Studio II.

Receiving optics unit # 2

This unit is installed next to the receiving optics unit #1 (the side closer to the detector). A receiving Soller slit can be installed in receiving optics unit #2.

Receiving parallel slit adapter (RPS adapter)

This adapter is used to install a receiving Soller slit. The receiving Soller slit is installed so that the slits are parallel to the scanning direction of theta d axis, that is, in the axis divergence direction.



Receiving parallel slit adapter

1 Attach the receiving parallel slit adapter to the rail of the theta_d arm. Secure it in place with the Allen wrench provided.





2 Connect the cable to the RPS connector on the receiving connector box. The connector name is indicated on the cable end.

3 Attach the receiving Soller slit to the receiving parallel slit adapter. Secure it in place with the Allen wrench provided.



Туре	Aperture	Abbreviation	Illustration
Soller slit 5° (optional: 2680D116)	5°	Soller_Slit_5.0 deg	
Soller slit 2.5° (standard)	2.5°	Soller_Slit_2.5 deg	

Table	Rec	eiving	Sol	ler	slit
THENTE		CAT AAA	- NOA		DAAL

Tip

The type of receiving Soller slit currently installed on the adapter can be identified by SmartLab Studio II.

Receiving slit # 2

The virtual receiving slit functions as the receiving slit #2. Limiting the detection area of the detector and using it as a receiving slit is called the virtual receiving slit. In para-focusing optics, it controls the 0D measurement resolution. In parallel beam optics, it functions as the second slit in a double-slit receiving system.

Slit width and vertical position are adjusted in SmartLab Studio II.

Attenuator

The attenuator is used to adjust the diffracted X-ray beam intensity using the several types of metal films on the rotating disc. Attach it in front of the detector on the theta_d arm.

The type of attenuator can be changed via SmartLab Studio II, and also can be automatically changed according to the detected intensity during measurements.



Attenuator (optional: 2680S102)

1 Attach the attenuator to the rail of the theta_d arm. Secure it in place with the Allen wrench provided.





2 Connect the cable to the ATT connector on the receiving connector box. The connector name is indicated on the cable end.

Tip

The attenuator types for actual measurements are as follows: Open, 1/70, 1/1000, 1/10000. The attenuator types for alignments are as follows: 9 kWBB, 9 kWBB, 3 kWBB, 3 kWPB.

Detector

D/teX Ultra250

D/teX Ultra250 is a semi-conductor 1D detector in which small detecting devices are arranged. Since D/teX Ultra250 detects and integrates X-ray intensity efficiently, the diffraction peaks in a broad range can be detected with a high speed scan. As a characteristic of a semi-conductor detector, it has high energy resolution which can reduce background level, enabling the detection of diffraction peaks of minor components easily.

In standard configuration, D/teX Ultra250 and its adapter are fixed on the goniometer arm and cannot be removed. If the optional monochromator is used with the detector, a detachable detector adapter will be provided.

Whether D/teX Ultra250 is installed or not can be identified by SmartLab Studio II.



D/teX Ultra250

1 Attach the detector to the rail of the theta_d arm. Secure it in place with the Allen wrench provided.





When using the detector with para-focusing optics, set the installation position of the detector adapter to 350 mm



2 Connect the cable to the DETECTOR connector on the receiving



Detector adapter (detachable type)

This adapter enables attaching/detaching D/teX Ultra250 when an optional monochtomator is used together.



Detector adapter (Optional, intended for using the monochromator and detector together)

1 Attach the detector adapter to the rail of the theta_d arm. Secure it in place with the Allen wrench provided.





When using the detector with para-focusing optics, set the installation position of the detector adapter to 350 mm



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2 Connect the cable to the DETECTOR connector on the receiving connector box.

The connector name is indicated on the cable end.



3 Install the D/teX Ultra250 to the detector adapter, and secure it in place with the Allen wrench provided.



Diffracted beam monochromator dedicated for D/teX

The diffracted beam monochromator (hereafter called DBM) for D/teX is used to detect only the K α X-rays, and remove the K β X-rays, which interfere with detecting the diffraction peaks of minor components. By using this monochromator with D/teX Ultra250, measurement is available in an even higher energy resolution and detects the diffraction peaks of minor components efficiently.

DBM for D/teX

This monochromator is used to monochromatize the diffracted beam received in a 1D measurement to use only the K α X-rays.



DBM for D/teX and D/teX Ultra250 (optional: C484-0001-3-39)

1 Install D/teX Ultra250 on the DBM for D/teX. Secure it in place with the Allen wrench provided.



Attach only the receiving slit box to the rail of the theta_d arm.

If receiving optics unit #1, receiving optics unit #2, or attenuator is attached,

3 Install the DBM for D/teX (with the D/teX Ultra250 installed) to the detector adapter. Secure it in place with the Allen wrench provided.





2

remove them.

When using the detector with para-focusing optics, set the installation position of the detector adapter to 350 mm.



DBM for D/teX 0D BB

This monochromator is dedicated for para-focusing optics, and used to monochromatize the diffracted beam received in a 0D measurement to K α X-rays.



DBM for D/teX 0D BB and D/teX Ultra250 (optional: A00002334)

1 Install D/teX Ultra250 on the DBM for D/teX 0D BB. Secure it in place with the Allen wrench provided.





Install the DBM for D/teX 0D BB (with the D/teX Ultra250 installed) to the detector adapter. Secure it in place with the Allen wrench provided.

3 Insert the Receiving slit 2(RS2 0.3/0.15mm) into the DBM for D/teX 0D BB.





2

When using the detector with para-focusing optics, set the installation position of the detector adapter to 350 mm.



Rigaku

DBM for D/teX 0D BB/PB

This monochromator is used to monochromatize the diffracted beam received in a 0D measurement to K α X-rays. By rotating the dial on the top of the monochromator housing, the angle of the monochromating graphite crystal position can be changed between Bent (para-focusing geometry) and Flat (parallel beam geometry).



DBM for D/teX 0D BB/PB and D/teX Ultra250 (optional: A00002314)

1 Install D/teX Ultra250 on the DBM for D/teX 0D BB/PB. Secure it in place with the Allen wrench provided.


2 Install the DBM for D/teX 0D BB/PB (with the D/teX Ultra250 installed) to the detector adapter. Secure it in place with the Allen wrench provided.



3 When using the DBM for D/teX 0D BB/PB with para-focusing optics, insert the receiving slit #2 (RS2 0.3/0.15mm).

When using it with parallel beam optics, remove the receiving slit #2.



Note

When using the detector with para-focusing optics, set the installation position of the detector adapter to 350 mm.



HyPix-400

HyPix-400 is a multi-dimensional semiconductor hybrid pixel array detector with an ultra-high dynamic range and high-sensitivity. 0D, 1D and 2D measurements can be done by the seamless detection mode.

Horizontal mount

When HyPix-400 is attached to the dedicated holder (included with HyPix-400) and installed on SmartLab SE, it can be positioned horizontally on the theta_d arm.

Vertical mount

When HyPix-400 is attached to the HyPix-400 holder (optional: A00003065) and installed on SmartLab SE, it can be positioned vertically on the theta_d arm.

For more details, refer to the instruction manual of HyPix-400.



HyPix-400 (optional: A00010204)



Horizontal mount (using dedicated holder)



Vertical mount (using optional holder) (optional: A00003065)

Installation of HyPix-400

Attachment Platform

Fixed sample platform and universal Z attachment platform

The fixed sample platform is installed on the goniometer as a standard component. It is used for fixing the sample in position during a measurement. The universal Z attachment platform (optional: A00010190) can be selected instead of the fixed sample platform.

When using the fixed sample platform, attachment bases other than sample holders cannot be installed.

When using the universal Z attachment platform, various attachment bases can be installed. With this platform, the Z-axis adjustment mechanism enables adjusting the height of the sample surface upon using the attachment bases.



Fixed sample platform



Universal Z attachment platform (optional: A00010190)

Direct beam stop

In measurements using D/teX Ultra250, the direct beam stop should be always installed to prevent the direct beam from irradiating directly on the active area.



Direct beam stop

The direct beam stop is installed on the detector side of the fixed sample platform or the universal Z attachment platform. When installing the direct beam stop, move the theta_d arm to 60° (2theta/theta=120°).



Installation of direct beam stop

When the direct beam stop is not being used, such as when aligning the position or exchanging an attachment, follow the steps below and move the direct beam stop to the retracted position.

- ① Loosen the thumb screw.
- ② Slide the direct beam stopper inside.
- ③ Tighten the thumb screw.



Retraction of direct beam stop

Knife edge

The knife edge is effective to reduce backgrounds in a 1D measurement with D/teX Ultra250.



Knife edge (optional: 2431G102)

The knife edge is installed to the upper side of the fixed sample platform or the universal Z attachment platform.



Installation of knife edge

Follow the steps below to retract the knife edge when exchanging the measurement sample.

- 1 Loosen the screw.
- 2 Turn the knife edge clockwise.



(In-use position)

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Retraction of knife edge
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Attachment Base

Attachment bases are used in combination with attachment heads and sample plates on the universal Z attachment platform. For details, refer to the instruction manual of each attachment base.

Mounting attachment base to the goniometer

To install the attachment bases on the universal Z attachment platform, follow the steps below.

- **1** Slide the attachment base down the rail of the universal Z platform.
- **2** Make sure that the attachment base is level, and then secure it by tightening a lever on the side of the universal Z attachment platform.



Installation of attachment bases

- **3** Connect the attachment base cable to the attachment connection port of SmartLab.
 - ① Raise the attachment connection port cover to open.
 - ② Connect the cable, with the indication "ATTACHMENT" side upward.
 - ③ Tighten the two screws of the connector.
 - 4 Lower the attachment connection port cover to close.



Standard attachment base

This can be used in combination with attachment heads, and is included with the universal Z attachment platform (optional: A00010190).



Standard attachment base

β attachment base

This can be used in combination with attachment heads.

The rotational axis (β) on the sample surface plane enables performing measurements while rotating the sample thereby averaging the variation of particle sizes.



 β attachment base (optional: 2430B101)

φ attachment base

This can be used in combination with attachment heads. The movement of rotational axis (ϕ) on the sample surface plane can be controlled in micro angles.



φ attachment base (optional: 2430E101)

Attachment

Attachments can be installed on the universal Z attachment platform. Attachment base is not required for the attachments listed below. For details, refer to the instruction manual of each attachment.

ASC-6/ASC-10 attachment

Consecutive measurements can be performed by switching multiple samples mounted on the turret. The rotational axis (β s) on the sample surface plane enables performing measurements while rotating the sample thereby averaging the variation of particle sizes.

The ASC-6 attachment (optional: 2430H101) for six samples or the ASC-10 attachment (optional: 2430H201) for ten samples can be selected. The vertical transmission geometry (optional: 2455H105) is also available in ASC-6.





ASC-6 (optional: 2430H101) ASC-10 (optional: 2430H201)

αβ attachment

The $\alpha\beta$ attachment controls the sample surface plane rotational axis (β) and the sample tilt axis (α). Since the sample oscillation axis (γ) and the transmission geometry is also available, the reflection/transmission pole figure measurements can be performed. Residual stress measurements are also available.



αβ attachment (optional: A00001668)

χφ attachment

The $\chi \phi$ attachment controls the sample surface plane rotational axis (ϕ) and the sample tilt axis (χ) in micro angles. Thin film measurements are available by using the reflectivity rate of thin films. Reflectivity pole figure measurements and residual measurements are available as well.



Xq attachment (optional: 2430D101)

Multipurpose high temperature attachment

The multipurpose high temperature attachment is designed to keep a sample at a high temperature during X-ray diffraction measurements. Changes in crystal structure and composition of a sample under specific temperature can be observed. It enables measurement in a vacuum environment, specific atmospheric condition, or inert gas environment.



Multipurpose high temperature attachment (optional: 2311B900)

Corrosion resistant high-temperature attachment Reactor-X

The Reactor X corrosion resistant high-temperature attachment (hereinafter called Reactor X) is designed to keep a sample at a high temperature during X-ray diffraction measurements. Reactor X uses an infrared heater to heat the sample, providing a high rate of temperature increase, and allows good temperature-holding characteristics in measurements. The sample stage is made of SiO₂, which allows measurements in various ambient environments.



Corrosion resistant high-temperature attachment Reactor-X (optional: A00006734)

DSC attachment

The DSC attachment is designed to perform measurements of differential scanning calorimetry (DSC) and X-ray diffractometry (XRD) simultaneously while heating/cooling a sample. With small amount of sample, it measures thermal changes in transition, melting, crystallization and dehydration while measuring the change in X-ray diffraction intensity simultaneously.



DSC attachment (optional: 8230X002)

Anton Paar attachment

Measurements can be performed using the Anton Paar attachments equipped with temperature adjustment mechanism (HTK1200N, HTK16, HTK2000, XRK900, TTK600, and CHC Plus). For more details on how to use the attachment, temperature range and pipe arrangement, refer to the instruction manual of each attachment.

Table Anton Paar attachment

Name	Illustration	
HTK1200N attachment (optional: 2312A220 / 2312A230)		<
HTK16N attachment (optional: 2312A200)		
HTK2000N attachment (optional: A00004448)		C
XRK900 attachment (optional: A00004451 / A00004452 / A00004453)	Care Contraction	
TTK600 attachment (optional: A00007064 / A00007065 / A00007066 / A00007067)		<
CHC plus attachment (optional: A00007057 / A00007059)		

Attachment Head

Depending on measurement purposes, you can change attachment heads with various adjustment axes. Attachment heads can be installed on standard, β , and ϕ attachment bases (all optional).

Mounting attachment head

- **1** Unlock the front latch.
- **2** Open the clasps to the right and left sides. Then remove the attachment head currently installed.



- **3** Make sure the right and left clasps are open.
- **4** Install an attachment head to be used by aligning the groove of the attachment head with the pin on the front.
- **5** Close the clasps and lock the latch.



Standard attachment head

This attachment head has no movable axis. It is included with the Universal Z attachment platform (optional: A00010190). It is used for measurements of powder and bulk samples.



Standard attachment head

RxRy attachment head

This attachment head has Rx and Ry axes that permit tilt adjustments of two axes intersecting at right angles. It is used for reflectivity measurements and RSM measurements.



RxRy attachment head (optional: 2680A213)

XY-20 mm attachment head

This attachment head has +/-10 mm X and Y translation axes which are perpendicular to each other. It is used to position the sample for micro area measurements. This attachment cannot be used with 8-inch wafer sample plates.



XY-20 mm attachment head (optional: 2680A211)

XY-4 inch φ attachment head

This attachment head has $\pm - 50$ mm provides X and Y translation axes which are perpendicular to each other. It is used for full-map measurement of 4-inch wafers. This attachment can be used only with the 4-inch XY mapping sample plate.4.



XY-4 inch φ attachment head (optional: 2680A212)

Capillary spin attachment head

This attachment head is used to spin the sample filled in a capillary during measurements.



Capillary spin attachment head (optional: 2430C101)

Battery cell attachment head

This attachment head is used to charge/discharge batteries during measurements. It is used in X-ray diffraction measurements of positive and negative electrode materials.



Battery cell attachment head (optional: A00006816)

Anton Paar attachment head

This is the Anton Paar dome-shaped sample heating unit. For more details on how to use the attachment head, temperature range and pipe arrangement, refer to the instruction manual of each attachment head.

Table Anton Paar attachn	ient	head
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Name	Illustration
DHS1100 attachment head (optional: A00007051)	
DCS500 attachment head (optional: A00007052)	



The type of attachment head installed can be identified by SmartLab Studio II.

Sample Plate

Select a sample plate based on the sample to be measured. Sample plates are placed directly on the attachment head.

Height reference sample plate

The height reference sample plate is used by inserting a glass sample holder or an aluminum sample holder.

The height reference sample plate is placed directly on the attachment head. In a sample alignment, a center slit and Si powder reference sample are inserted. It is included with universal Z attachment platform (optional: A00010190).



Height reference sample plate

Transmission SAXS sample plate

The transmission SAXS sample plate is used by inserting the transmission SAXS sample holder. The transmission SAXS sample plate is placed directly on the attachment head. It is included with the SAXS unit (optional: 2680J111).



Transmission SAXS sample plate

Tip

 Normally the height reference sample plate and transmission SAXS sample plate should be used on top of the standard attachment head. If using the height reference sample plate or transmission SAXS sample plate with an attachment other than the standard attachment head, you must set each attachment head axis position to the original position (0° or 0 mm). To install/remove each sample plate to/from an attachment head, see_Installing and removing wafer sample plates.

β spin sample plate

The β spin sample plate is used by inserting a glass sample holder or an aluminum sample holder. It is included with the β attachment base (optional: 2430B101).



 β spin sample plate

Wafer sample plate and sample spacer

A wafer sample plate is used in measurements of wafer-shaped samples. A sample spacer is placed on the attachment head. The wafer sample plate is placed on the sample spacer. A sample is placed on top of the wafer sample plate. Wafer sample plates and sample spacers are available for 4-inch, 6-inch, and 8-inch samples.



Wafer sample plate (optional)



sample spacer (optional)



Placing a sample larger than the sample plate may damage the sample during measurement.

Magnets are generally used to secure the sample in place. For details on how to mount samples, see <u>Wafer-shaped samples</u>.



Тір т

The sample holding magnets can be used for samples measuring up to approximately 3 mm in thickness.

Sample holding magnets

The Z axis is used to align the sample surface with the center of the X-ray beam. Since the thickness range for which the Z axis can be adjusted is 9 mm, you must select the appropriate sample spacer based on the thickness of the sample to be measured.

Fabl	e	Sam	ple	SI)a	cers
	-	~	P	~ •		

Measurable sample thickness	Abbreviation	Illustration
0 to 3 mm (optional: 2680A406)	0-3 mm	
3 to 12 mm (optional: A00001187)	3-12 mm	0000
12 to 21 mm (optional: A00001188) 0 to 9 mm (*1)	12-21 mm 0-9 mm (blue stamped mark)	6°00.0

*1 When the XY-4 inch ϕ attachment head is used, the thickness indicated by the blue stamped mark on the sample spacer is available.

Table Wafer sample plates

Name	Illustration
4-inch wafer sample plate (optional: 2680A304)	
6-inch wafer sample plate (optional: 2680A302)	
8-inch wafer sample plate (optional: 2680A303)	
4-inch XY mapping sample plate (included with XY-4 inch ϕ attachment head)	\otimes

Table List of possible combinations of attachment head, sample spacer, and sample plate

Attachment head	Sample spacer	Sample plate	Measurable sample thickness
Standard attachment head RxRy	0-3 mm4-inch wafer sample plate3-12 mm6-inch wafer sample12-21 mmplate(*1)(black stamped mark)8-inch wafer sample		0-3 mm 3-12 mm 12-21 mm
attachment head	4-inch wafer sample p		
XY-20 mm	6-inch wafer sample plate(*1)		21-30 mm
attachment head	8-inch wafer sample plate(*1)		
XY-4 inch ϕ attachment head	0-9 mm (blue stamped mark)(*2)	0-9 mm (blue stamped mark)(*2)4-inch XY mapping sample plate	
	4-inch XY mapping sample plate		9-18 mm

*1 Both X and Y must be 0 mm when a sample is placed on the XY-20 mm attachment head.

*2 When the XY-4 inch ϕ attachment head is used, the thickness indicated by the blue stamped mark on the sample spacer is available.

Atmosphere separator

The atmosphere separator is a sample plate which can isolate the powder sample from the external atmosphere. It is used by placing the included sample spacer on the attachment head, then placing the atmosphere separator on it.



Atmosphere separator (optional: 2392A100)

Installing and removing sample spacers

To mount a sample spacer to an attachment head, align the sections indicated by the arrows in the figure below, and then turn the sample spacer clockwise to secure it in place.



Installing the sample spacer on the attachment head

To remove the sample spacer from the attachment head, apply pressure to the bar indicated by the arrow in the figure below, and then turn the sample spacer counterclockwise.



Removing the sample spacer from the attachment head

Installing and removing wafer sample plates

To mount a wafer sample plate to a sample spacer, align the sections indicated by the arrows in the figure below, and then turn the sample plate clockwise to secure it in place.

The wafer sample plate can be removed by turning it counterclockwise.



Installing/removing the wafer sample plate

Tip

See <u>Installing and removing sample spacers</u> for details on direct mounting (removing) a wafer sample plate to (from) an attachment head.

Sample Holder

Sample holders are used to measure samples that require preparations, such as powder and bulk samples.

Glass sample holder

This sample holder is used to measure powder samples.



Glass sample holder

Aluminum sample holder

This sample holder is used to measure powder or small bulk samples.



Aluminum sample holder

Low background sample holder

This is a sample plate with special processing that prevents diffraction X-rays deriving from the sample holder to be irradiated. Compared with the glass sample holder, halos do not appear when using this sample holder. This is effective in measurements at low angle side or of a small amount of sample.



Low background sample holder (optional: M00016228)

Transmission SAXS sample holder

This sample holder is used to measure fibrous or film samples in transmission method. It is included with the SAXS unit (optional: 2680J111).



Transmission SAXS sample holder

Capillary holder

This sample holder is used to mount the capillary sample onto the capillary spin attachment head. The capillary sample is spun with the rotation (ω_c) axis during measurements. Attach the capillary sample to the capillary holder and mount the holder to the capillary spin attachment head. The prepared sizes of the holder are $\varphi 1.0 \text{ mm}$, $\varphi 0.9 \text{ mm}$, $\varphi 0.7 \text{ mm}$, $\varphi 0.5 \text{ mm}$ and $\varphi 0.3 \text{ mm}$. The capillary holder suited for the size of the capillary should be selected.

Capillary



Capillary holder

(optional)



Capillary spin attachment head

General-purpose sample holder

This sample holder is used for reflection and transmission measurements of powder, fibrous, and film samples.



General-purpose sample holder for reflection (optional: 2101G101)



General-purpose sample holder for transmission (optional: 2101G102)

Air-sensitive sample holder

This sample holder is used to place a sample in a simple airtight condition during measurements.



Air-sensitive sample holder (optional: 2392B101)

Accessory

Table Accessory of main unit

Name	Q'ty	Illustration	
Kβ filter	1		←
Fixed beam stop	1		<
Glass sample holder 0.2 mm depth 0.5 mm depth	2 5		←
Aluminum sample holder	2		<
Ball-end hex screwdriver (for installing optical devices)	1		<

Table Accessory of fixed sample platform

Name	Q'ty	Illustration
Center slit	1	Service and
Si powder reference sample	1	

Name	Q'ty	Illustration	2
Center slit	1		<
Si powder reference sample	1		<
Si wafer reference sample	1		
Height reference sample plate	1		 ←───
Standard attachment head	1	ES-	
Standard attachment base	1		 ←−−−−

TableAccessory of universal Z attachment platform
(optional: A00010190)

Example of Sample Mounting

Introduced below are some typical sample mounting methods.

Powder sample

Powder samples are filled into the indentation of the glass or aluminum sample holders and then inserted into the fixed sample platform or the height reference sample plate.





(Fixed sample platform) (Height reference sample plate)

Holding powder samples

Bulk sample

If the size of a bulk sample fits within the window of the aluminum sample holder, the sample should be secured in place (by using compounds, etc.) on the aluminum sample holder and then inserted into the fixed sample platform or height reference sample plate.

If the size of the sample is large, a 4-inch wafer sample plate, 6-inch wafer sample plate, or 8-inch wafer sample plate should be used to mount the sample. In this case, the sample should be placed so that the measurement sample surface is parallel to the sample plate.

For details, see Wafer sample plate and sample spacer (optional).

Sample for transmission SAXS measurements

Fibrous and film samples should be mounted by clamping it to the transmission SAXS sample holder, securing it in place, then inserting it into a sample holder (Transmission SAXS sample holder and transmission SAXS sample plate are included with the SAXS unit (optional: 2680J111)).

Wafer-shaped samples

A 4-inch, 6-inch, or 8-inch wafer sample plate is used to measure wafer-shaped samples (wafer sample plates and sample spacers are available optionally).



Wafer-shaped sample

Tip

When placing or removing the sample, we recommend removing the sample plate and sample spacer from the attachment head. When removing the sample plate and sample spacer, we recommend setting the chi axis to 0° and positioning the sample horizontally.

Place the sample at the center of the sample plate. Use the sample fixing magnets (optional: 2680R101) to hold the sample in place. The magnets can be used with samples that are up to 3 mm thick.

To perform measurement of samples thicker than 3 mm, change the sample spacer, or see Bulk sample.



Placing a sample larger than the sample plate may result in damage to the sample during measurement.



Place an 8-inch wafer on the center of the 8-inch wafer sample plate because the space available for the sample holding magnets in place is small.

Periodic Maintenance

Perform the following maintenance tasks at regular intervals to maintain the performance of SmartLab SE.

Optics and detector maintenance

Multilayer mirror	Re-adjustment is required after performing maintenance of the X-ray generator.
Attenuator correction	Re-adjustment is required after changing the wavelength or performing maintenance of the X-ray generator.
Detector	Re-adjustment is required after changing the wavelength to be measured.

There is a mathematical possibility that the characteristics of the detector will change over time. However, you can minimize performance degradation by making the appropriate adjustments. We recommend performing detector adjustments at periodic intervals.

- D/teX Adjustment
- · HyPix Adjustment
- HyPix Calibration

To make these adjustments automatically, select **Package Part** panel and **Activities** panel, then drag and drop the appropriate package activities on the **Sequence**. For details about these parts activities, refer to the Help topic of each Parts Activities.

Cooling water

Check the amount of cooling water when using the water chiller. If the water level is low, add water. Periodic replacement of cooling water is also recommended.

Cooling water filter

This filter removes impurities from the cooling water. If the filter becomes clogged with impurities, the water pressure may not rise appropriately. We recommend changing the filter at periodic intervals.

Observation window

The observation window used in the SmartLab SE is made of high lead content glass. Pay attention to the following precautions when using or cleaning the window.



Compared to normal glass, high lead content glass is easier to have cloudy stains that persistently remain. Please pay attention to the following precautions.

- Do not use wet cloth or water to clean the glass. Immediately wipe off water with a dry cloth in case the glass becomes wet.
- If the glass becomes dirty, polish it slightly hard with a clean cloth like gauze using alcohol or a glass polisher suitable for lead glass. Wipe again afterwards with a dry cloth.
- Leaving water or dirt (fingerprints) on the glass might cause cloudy stains which may not be completely removed.
- Besides all of the above, clean the glass once a month to prevent the glass from becoming cloudy.

Maintenance parts list

Maintenance parts for SmartLab are listed below.

Parts other than those listed below may also have to be replaced due to aging degradation or consumption. If those parts are required, please contact the sales representative who you purchased the instrument from.

Consumables

Itom No.	Name	Required	Place to be used	
Illustration Q'		Q'ty	Remarks	
	O.P.		Tube shield	
MHRO00167		6	Used for X-ray tube mounting section Two O-rings included upon installation.	
B.C0000027	Silicone grease		HV cable	
RC0000927		ł	Used for XG power source side	
9407F701	X-ray tube		Tube shield	
9441F701		1	Each sealed tube	
0007 1 205	Ion exchange resin		Water chiller	
9987A205		1	If used, replaceable	
M07700001	Cooling water (purified water)	20 L	Water chiller	
MQZZ00001	and the second s		The amount depends on the water chiller.	
400007640	Filter element for cooling water filter unit		Cooling water filter unit	
A00007049		1	If used	

Maintenance parts

	Name	Required Q'ty	Place to be used
Item No.	Illustration		Remarks
A00008316	Fan	1	The upper area of radiation enclosure
			Optional item for temperature control attachments
E00003625 E00003626	Fan	1	The internal chiller
			Optional item for the internal chiller
RC0002330	Cooling water hose (Tetron braided hose)	15 m	Between water chiller and CW_UNIT
			Depends on the environment
MHWW00006	Cooling water hose (Pisco tube)	1 set	Between CW_UNIT and tube shield
			Cooling water tube for X-ray tube
RC0046834	Power key (2 keys/set)	1	Radiation enclosure
	A A		Spare key in case of lost

Troubleshooting

Troubleshooting

#	Problem	Remedy		
1	The door of SmartLab SE cannot be opened.	The door may be locked. Confirm that the Door-Lock indicator is lighting up in green. Click the door lock button of the SmartLab Stud II to unlock the door, then you can open the door.		
2	An error occurs during SmartLab Studio II startup. SmartLab SE startup procedure fails to complete properly.	There may be a problem in the connection between SmartLab SE and the controller. Confirm that the Power-On indicator is lighting up, and the Alarm indicator is turning off. Also confirm that the connectors are appropriately connected. After checking these items, shut down SmartLab Studio II, then restart it. When restarting SmartLab SE as well, be sure to restart SmartLab Studio II.		
3	The goniometer stops abruptly during measurement or other operation.	The goniometer may have stopped due to excessive force applied to the theta_s or theta_d arm. Turn the power key on the front panel to OFF to stop SmartLab SE. Check the arms to see if any item obstructs their operation, then restart SmartLab SE.		
4	The X-ray generator stops during measurement or other operations.	In the SmartLab Studio II, click System Status in the Utility tab, the part name where the alarm has been generated will be displayed in red. Check the cause.		
		Target Cooling water Cooling water Image: Cooling water HVPS Operating time of the ion exchange resin (0.00 to 4,500.00), hr 0.00 Radiation enclosure and X-ray shutter Cooling water - electrical conductivity (0 to 0), uS/m: Version Cooling water - water flow (3.8 to 5.0), L/min: Cooling water - water flow (3.8 to 5.0), L/min: 3.6 Cooling water - water temperature (0 to 43), 'C: 21 To reset the alarm status, click Alarm in the Utility tab, and then click Reset. Reset Clear Save As. Message No. Date Part Name Message Error Code Operator Mageered		
	1 2016-11-09 09:25:12 RCD3 ALARM_RCD3_000 ALARM_RCD3_000 Administrator System Status Maintenance Accessories Alarm X-ray Status			

* If a problem other than those described above occurs or if the problem cannot be corrected by taking the countermeasure described, please contact the sales representative who you purchased the instrument from
Operations of Alarm Indicator and Alarm Sound

The Alarm indicator is usually turned off, but will begin flashing in red at set intervals and alarm sound will be heard in the event of an abnormality. To reset the generated alarm, you must restart SmartLab SE. After shutting down SmartLab Studio II, turn the power key to the "O" position, then turn it to the "]" position to restart SmartLab SE.

If the Alarm indicator does not turn off, flashing in red instead after startup, please contact the sales representative who you purchased the instrument from. Before contacting, please refer to following tables to identify the alarm being generated.

* When the door is closed: Alarm sound is heard. The indicator is flashing.
When the door is opened: Alarm sound is not heard. The indicator is flashing.

Error	Pattern
XG error	$400 \text{ ms} \qquad 50 \text{ ms} \\ 0 \text{ ne long beep and two short beeps (repeats)} $
Communication error	200 ms 1000 ms One beep (repeats)

Table Pattern of the alarm indicator flashing and alarm sound for errors





Table	Causes	of	alarm
	Chabeb		

Cause	Description
XG error	An error has occurred in the X-ray generator (XG). See # 4 in Troubleshooting.
Attachment connecting cover is open	The cover of the attachment connection cable is open in the radiation enclosure. Close the cover.
Communication error	A communication error has occurred in the control board for the optical devices in the incident and receiving optics. The cables for the incident and receiving optics may be disconnected or broken.

-

Characteristic X-rays

X-rays are generated by accelerating electrons to very high speeds in a vacuum and directing them against the anode (target). The X-ray spectra generated by electrons colliding against the target can be divided into two categories: a continuous spectrum indicating continuous X-rays (white X-rays) and a discrete spectrum for characteristic X-rays.



X-ray spectrum of Cu target

The wavelengths of characteristic X-rays depend on the type of target used. Typical X-ray diffractometry uses $K\alpha$ X-rays generated by several types of metal targets, as shown in the following table. $K\alpha$ X-rays contain $K\alpha$ 1 X-rays and $K\alpha$ 2 X-rays whose wavelengths are quite close. Although this does not pose serious problems for ordinary measurement of powder samples for phase identification (ID) analysis, optimal results can be achieved by using only $K\alpha$ 1 X-rays in certain cases when making measurements for crystal structure analysis with powder samples or when performing precise measurements of thin film samples. In recent years, it has become possible to use just $K\alpha$ 1 X-rays by employing an incident optical system comprised of a multilayer mirror and Ge or Si monochromator crystals.

Г	arget		Wavelength (Å)	
Element	Atomic number	Κα2	Ka ₁	Кβ
Cr	24	2.294	2.290	2.085
Fe	26	1.940	1.936	1.757
Со	27	1.793	1.789	1.621
Cu	29	1.544	1.541	1.39
Мо	42	0.7136	0.7093	0.6323
Ag	47	0.5638	0.5594	0.4970
W	74	0.2138	0.2090	0.1844

Table Wavelengths of characteristic X-rays

Tube voltage and tube current

The intensity of characteristic X-rays is proportional to the *n*-th power of the difference between tube voltage and excitation voltage (minimum voltage required for obtaining characteristic X-rays). It is also proportional to tube current. When the tube voltage is low, the value of n approaches 2. As the tube voltage increases, the value of n becomes smaller. On the other hand, the intensity of continuous X-rays that appear as a background in the K β filter method is proportional to the square of the tube voltage and is also proportional to the tube current. This means that an optimum tube voltage value exists for measurements with each target. The following table gives the optimum voltages for different target types. For measurements using the K β filter method, it is a good way to set the tube voltage so that the P/B ratio (peak-to-background ratio) will become the largest.

	Excitation	Optimur (equivalent	1 voltage 10ad) (kV)	
Target	(kV)	Intensity at maximum	P/B ratio at maximum	
Cu	8.86	40~55	25~35	
Со	7.71	35~50	25~35	
Fe	7.10	35~45	25~35	
Cr	5.98	30~40	20~30	

Penetration depth (effective thickness)

The following formula gives the ratio G_x between the intensity of diffracted X-rays from a sample of infinite thickness and the intensity of diffracted X-rays from a sample with a specific finite thickness of x (cm).

$$G_x = \frac{\int_0^x dI_D}{\int_0^\infty dI_D} = 1 - \exp\left(\frac{-2\mu x}{\sin\theta}\right)$$

Substitute the values for angle θ of the incident X-ray beam and X-ray absorption coefficient μ (/cm) in the following formula to obtain x, the effective thickness.

$$x = \frac{-\ln(1 - G_x)\sin\theta}{2\mu} = \frac{K_x\sin\theta}{2\mu} \quad \text{where} \quad K_x = -\ln(1 - G_x)\sin\theta$$

Diffracted X-ray utilization rate G_x (%)	50	75	90	95	99	99.5
K _x	0.69	1.39	2.30	3.00	4.61	6.91

 $-G_x$)

The following table shows the results of calculations of the sample thicknesses x that give a diffracted X-ray usage rate of 90% when $K_x = 2.30$ and $\theta = 90^\circ$.

Substance		Density p	Mass abs	orption coef	ficient µ/p(cm ² /g)	Thickness for 90% utilization rate (mm)				
		(g/cm ³)	MoKa	CuKa	СоКа	CrKa	MoKa	CuKa	СоКа	CrKa	
	a-Al ₂ O ₃	3.97/2	3.34	31.1	48.0	97.6	1.74	0.187	0.121	0.060	
	Si	2.42/2	6.44	60.6	93.3	189	1.48	0.157	0.102	0.050	
Powder	a-SiO2	2.65/2	3.71	34.5	53.1	108	2.34	0.252	0.164	0.080	
	CaCO ₃	2.71/2	8.02	70.9	107	205	1.06	0.120	0.079	0.041	
	a-Fe ₂ O ₃	5.26/2	27.3	216	42.7	86.5	0.160	0.020	0.103	0.051	
	ZnO	5.60/2	44.7	50.7	78.2	159	0.092	0.081	0.053	0.026	
	Ag ₂ O	7.22/2	24.1	204	300	548	0.132	0.016	0.011	0.0058	
	PbO I(tetragonal)	9.53/2	112	216	311	540	0.022	0.011	0.0078	0.0089	
The states	Al	2.70	5.16	48.6	74.8	152	0.826	0.088	0.057	0.028	
	a-Fe	7.86	38.5	308	52.8	108	0.038	0.0048	0.028	0.014	
Metal	SUS(18-8)	7.93	37.9	278	110	106	0.038	0.0052	0.013	0.014	
	Cu	8.92	50.9	52.9	81.6	166	0.025	0.024	0.016	0.0078	
	Pb	11.34	120	232	334	579	0,0085	0.0044	0.0030	0.0018	
Polymer	Polyethylene	0.93	0.59	4.0	6.1	12.5	21.0	3.09	2.02	0.989	
	Vinyl chloride	1.41	6.73	62.0	94.2	186.2	1.21	0,132	0.086	0.044	
Single	Si	2.42	6.44	60.6	93.3	189	0.738	0.079	0.051	0.025	
crystal	a-SiO2	2.65	3.71	34.5	53.1	108	1.17	0.126	0.082	0.040	

Cross beam optics(CBO)

CBO is an optical system which easily switches between the divergent beam used in measurements in para-focusing optics and the parallel beam conditioned by multilayer mirror, by replacing the selection slits. As shown in the diagram, selection slit (BB) uses X-rays passing through optical path A, while selection slit (PB) uses X-rays passing through optical path A. while selection slit (PB) uses X-rays passing through optical path B. In SmartLab SE, the multilayer mirror is designed so that optical path A and optical path B intersect at the center (goniometer center) of the sample at a distance of 300 mm from the X-ray focal point. There is no need to remove the multilayer mirror when switching from parallel beam optics to para-focusing optics. Simply change the selection slits and perform 2-theta axis adjustment one time.



Switching between para-focusing optics and parallel beam optics by changing selection slits

CBO-E keeps the CBO feature which makes the optical paths A and B intersect on the sample. The only difference is that the shape of the multilayer mirror used in the CBO-E unit is ellipsoidal while the mirror shape used for CBO is parabolic.

The ellipsoidal multilayer mirror used in CBO-E has been designed so that one focus of the ellipsoid is placed at the focus of the X-ray source and the other focus of the ellipsoid falls on the detector plane. When measuring capillary samples or samples on the transmission SAXS sample plate in the transmission geometry, the measurement can be performed very efficiently if a 1D detector like D/teX Ultra is used in combination. The CBO-E unit also switches between measurements in the reflection geometry in para-focusing optics and those in the transmission geometry in convergent optics. In both cases, D/teX Ultra enables very efficient measurements.



Incident monochromator crystal

The incident monochromator crystal system uses diffraction by Ge or Si crystals for X-ray monochromatization. Ge(220)x2 crystals and Ge(440)x4 crystals are available as options for SmartLab SE. Select the appropriate crystal based on the resolution required for measurements and the sample (d value). As an example, the Ge(220)x2 crystal monochromatizes X-rays by diffracting them twice by Ge(220) lattice planes. Passing X-rays through this crystal makes it possible to use only K α 1 X-rays. Shown below are the types and resolutions of monochromator crystals and corresponding applications.

Type of crystals	Resolution (FWHM)	Applications
Ge(220)x2 crystal	0.008°<	For applications requiring monochromatized Kα1 X-rays or for measurements of materials with diffraction planes with d spacings of ca. 2 Å.
Ge(400)x2 crystal	0.0022°<	For measurements of materials with diffraction planes with d spacings of ca. 1.4 Å.
Ge(220)x4 crystals	0.0033°<	For applications requiring high resolution over a broad 2-theta range.
Ge(440)x4 crystals	0.0015°<	For applications requiring very high resolution over a broad 2-theta range.

Table Types and resolutions of incident monochromator crystals



2-theta/omega scan profile of Si(001) single crystal wafer measured with different incident optics systems

Kβ filter

The characteristic X-rays used for X-ray diffractometry generally contain K α and K β X-rays. A substance that passes K α X-rays while absorbing most K β X-rays to monochromatize the beam is called a K β filter. The mass absorption coefficient (hereinafter referred to as "absorption coefficient") of an element becomes smaller as wavelengths become shorter, and vice versa. However, when X-rays with energy greater than the binding energy between the nucleus and electrons of an element, strike the element, the absorption coefficient suddenly increases due to the photoelectric effect. This discontinuous point in the absorption coefficient is called an absorption edge.



Change in mass absorption coefficient in accordance with wavelength (platinum)

For efficient absorption of K β X-rays only, select an element as a K β filter with its absorption edge wavelength located between the K α X-ray and K β X-ray wavelengths. Those elements will have an atomic number one or two less than that of the target element. The following table shows the target elements commonly used for X-ray diffractometry and their corresponding filters. $I_{K\beta}$ / $I_{K\alpha}$ = 1/100

	Wavele	ngth (Å)	Metals used as a filter						
	A La plat			Wavelength of	1	When $I_{K\beta}/I_{K\alpha} =$	1/100		
Target	Κα1	Kβ1	Substa -nces	absorption edge (Å)	Thickness (mm)	Mass per unit area (g/cm ²)	Kα ₁ transmissivity		
Cr	2.290	2.085	v	2.269	0.011	0.007	63		
Fe	1.936	1.757	Mn	1.896	0.011	0.008	62		
Co	1.789	1.621	Fe	1.743	0.012	0.009	61		
Cu	1.541	1.392	Ni	1.488	0.015	0.013	55		
Мо	0.7093	0.6323	Zr	0.689	0.081	0.053	43		
Ag	0.5594	0.4970	Rh	0.534	0.062	0.077	41		

Table $K\beta$ filters

Target Ka ₁	Wavele	ngth (Å)	Metals used as a filter						
				Wavelength of		When $I_{K\beta}/I_{K\alpha} =$	1/500		
	Target	$K\beta_1$ Substa -nces	Substa -nces	absorption edge (Å)	Thickness (mm)	Mass per unit area (g/cm ²)	Kα ₁ transmissivity		
Cr	2.290	2.085	V	2.269	0.017	0.010	49		
Fe	1.936	1.757	Mn	1.896	0.018	0.013	47		
Co	1.789	1.621	Fe	1.743	0.019	0.015	46		
Cu	1.541	1.392	Ni	1.488	0.023	0.020	40		
Мо	0.7093	0.6323	Zr	0.689	0.120	0.078	29		
Ag	0.5594	0.4970	Rh	0.534	0.092	0.114	27		

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