



# Automated Multipurpose X-ray Diffractometer Instruction Manual



This manual describes the correct use of the product as well the usage precautions to be observed. To obtain full-expected performance from the product, thoroughly read this manual.

Also, store this manual at an easily accessible place so that you can promptly refer to it whenever it is necessary.

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

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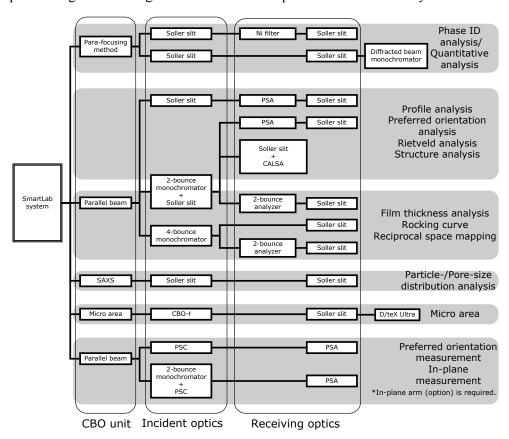
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Item No. 2080A211/B211/B212/A411/B411 Manual No. ME11573A06 2014. 11 (6th Edition)

# **Overview**

SmartLab, Automated Multipurpose X-ray Diffractometer, is an X-ray diffraction system equipped with a high-accuracy theta-theta goniometer featuring a horizontal sample mount. By changing slits (selection slits), the operator can use the para-focusing optics to measure polycrystalline samples, the parallel beam optics incorporating a multilayer mirror suitable for high-precision measurement to measure polycrystalline and thin film samples, or the SAXS optics to measure nano-scale samples. A high-resolution optics system provided with a 2- or 4-bounce monochromator on the incident side and a 2-bounce analyzer or CALSA on the receiving side can be easily installed simply by exchanging units. Adding an in-plane arm allows various in-plane measurements. SmartLab is capable of performing a broad range of measurements required for thin film analysis.



SmartLab structure and measurement examples

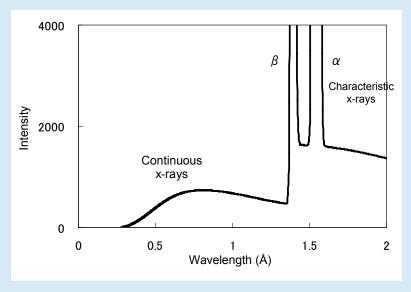
The following five types of SmartLab are described in this manual.

- 2080A211 Rotating anode type
- 2080B211 Sealed tube type
- 2080B212 Sealed tube type with in-plane arm
- 2080A411 Rotating anode type
- 2080B411 Sealed tube type

#### Column

#### 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 a Ge or Si monochromator crystal.

Table Wavelengths of characteristic X-rays

| Tai     | eget             |                 | Wavelength (Å) |        |
|---------|------------------|-----------------|----------------|--------|
| Element | Atomic<br>number | Kα <sub>2</sub> | $Ka_1$         | Kβ     |
| Cr      | 24               | 2.294           | 2.290          | 2.085  |
| Fe      | 26               | 1.940           | 1.936          | 1.757  |
| Co      | 27               | 1.793           | 1.789          | 1.621  |
| Cu      | 29               | 1.544           | 1.541          | 1.392  |
| Mo      | 42               | 0.7135          | 0.7093         | 0.6323 |
| Ag      | 47               | 0.5638          | 0.5594         | 0.4970 |
| W       | 74               | 0.2138          | 0.2090         | 0.1844 |

# **Product Features**

### High-accuracy theta-theta goniometer

A theta-theta goniometer enables omega scans, 2-theta/omega scans, and 2-theta scans with the sample oriented horizontally (deviation from horizontal orientation may occur if sample orientation is adjusted). The horizontal positioning of a sample minimizes the distortion effects caused by weight in the case of a large wafer while reducing the possibility of a dropped sample. Additionally, the two axes are equipped with encoders to enable control of each axis at a resolution of 0.0001°.

### **Attachment**

XY attachment for automated XY mapping or an RxRy attachment for sample tilt alignment to be conducted before in-plane or reciprocal space map (RSM) measurement. A newly developed connector allows easy changing of attachments.

## X-ray generator

Even with a horizontal sample mount goniometer with a moving X-ray source, the product can incorporate a state of the art high-intensity 9 kW rotating anode X-ray generator. When combined with a multilayer mirror, this X-ray generator produces a high intensity X-ray beam (approx. 6 to 7 times the intensity provided by a sealed-tube system) equal to an 18-kW rotating anode X-ray generator, while reducing power consumption by 50% due to the high-brightness focal spot. The system also offers lower operating costs compared to earlier models. A 3 kW sealed tube X-ray generator can also be used with the SmartLab system. The specifications of each generator are summarized in the following table

**Table Generator specifications** 

|                        | Max. load | Max. voltage | Max. current | Target metal |
|------------------------|-----------|--------------|--------------|--------------|
| 9 kW rotating anode *1 | 9 kW      | 45 kV        | 200 mA       | Cu           |
| 3 kW sealed tube *2    | 3 kW      | 45 kV *3     | 60 mA        | Cu           |

\*1:2080A211/2080A411

\*2:2080B211/2080B212/2080B411

\*3:60kV (Option:4621S102)

### **Cross beam optics (CBO)**

This unit allows easy switching between the direct beam for para-focusing (Bragg-Brentano) optics for phase ID analysis and quantitative analysis of powder samples and a monochromatic parallel beam using a multilayer mirror for profile analysis of powder samples, measurement of preferred orientation, measurement of thin film samples, RSM measurement, and rocking curve measurement, simply by changing a selection slit. Similarly, other selection slits allow easy switching between small angle X-ray scattering (SAXS) optics for nano-structural measurements and small aperture optics for micro area analysis.

## Alignment mechanism (monochromators and analyzers)

You can select crystal index and type based on the resolution required for measurement. The 2-bounce monochromators, 4-bounce monochromators, and 2-bounce analyzers have built-in adjustment mechanisms that enable automatic adjustment via control software (hereinafter referred to as "SmartLab Guidance"). Crystal adjustment positions are preserved even after the unit is removed, enabling data measurements without readjustment, simply by installing the previously adjusted crystal.

## Receiving analyzer system

Since the receiving slit box # 2 has a translatable slit position, the following receiving analyzer systems can be used for different applications simply by replacing the parts and using the automatic adjustment function of SmartLab Guidance.

- Double-slit analyzer with two variable slits on receiving side Para-focusing,, small-angle scattering, and reflectivity measurement geometries, etc.
- Parallel-slit analyzer (PSA)
   Profile measurements of powder samples using parallel beam optics and requiring high intensity and high precision, thin film measurements, and measurement of preferred orientation, etc.
- 2-bounce analyzer
   Reflectivity measurements requiring high resolution, RSM measurements, and rocking curve measurements, etc.

### Incident optics system

The following mechanisms enable switching between para-focusing optics, parallel beam optics, 2- or 4-bounce monochromator high resolution optics, small-angle scattering optics, and micro area measurement optics.

Additionally, for various in-plane measurements, a parallel slit collimator (PSC) that controls resolution in the in-plane direction can be installed on the incident parallel slit adaptor and 2-bounce monochromator.

- CBO unit
   To select the incident beam.
- Standard incident optics unit

  To select Soller slit, 2-bounce monochromator (with Soller slit), or 4-bounce monochromator.
- Standard incident slit box
   Equipped with variable slit and length-limiting slit.

### Receiving optics system

The following mechanisms enable selection of a broad range of resolution characteristics for specific purposes.

A parallel slit analyzer (in-plane PSA) that increases the resolution in the in-plane direction can be used for various in-plane measurements.

- Standard receiving slit box # 1
   For installation of a Kβ X-ray filter for measurements using para-focusing optics.
- Standard receiving optics unit # 1
   For installation of various analyzers.
- Standard receiving optics unit # 2
  For installation of parallel slit such as Soller slits.
- Standard receiving slit box # 2
   With a slit position alignment mechanism.
- Standard attenuator
   For the adjustment of X-ray intensity.

## **Optics switching system**

Optics alignment results are retained by SmartLab Guidance. When you change optics the previously stored alignment results are used eliminating the need for realignment.

## **Optical device detection**

SmartLab Guidance can check the conditions of the following optical devices used for measurement.

- Selection slit in CBO unit
- Type of incident Soller slit or crystal monochromator
- · Width of incident slit
- Length of length-limiting slit or type of collimator
- Width of receiving slit
- · Type of analyzer
- Type of receiving Soller slit
- Type of detector
- Presence/absence of diffracted beam monochromator, etc.

SmartLab Guidance checks whether the necessary optical devices are installed and displays a message 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 Guidance)**

SmartLab Guidance used to control SmartLab also guides the user through required measurement procedures and condition-setting processes, in addition to providing conventional SmartLab control functions. 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.

Each Package Measurement was prepared by specialists with expertise in the specific field of measurement methodology. Even users with limited experience with X-ray diffraction or X-ray reflectivity measurements can perform measurements in the same way a specialist would. The software also allows customization of alignment and measurement conditions for special measurement needs. Manual control is also possible. The software is designed to meet a wide range of user needs.

## **Two-slit SAXS optics**

The two-slit SAXS optics incorporating a multilayer mirror can perform measurements with better accuracy and S/N ratios than conventional three-slit SAXS optics. SmartLab Guidance handles previously difficult adjustments of small-angle scattering optics. NANO-Solver, the analysis package for pore and particle size distribution analysis, corrects scattered image distortion resulting from optics based on the deconvolution of the slit function.

## In-plane optics (2080B212)

The use of the in-plane arm and RxRy attachment enables measurements of in-plane diffraction by maintaining grazing incidence conditions during sample rotation. This allows the measurement of diffraction from lattice planes perpendicular to the sample surface. High-precision measurement of orientation, crystallinity, and distortion of thin films are possible without interference by X-rays scattered or diffracted from the substrate. Reflection pole figure measurements using the in-plane geometry eliminate the blind region at the pole figure edge typically encountered with traditional out-of-plane reflection pole figure measurements. Complete crystal orientation information is obtained from the total-area pole figure. Conditions for optics alignment and sample alignment are set automatically by SmartLab Guidance Package Measurements.

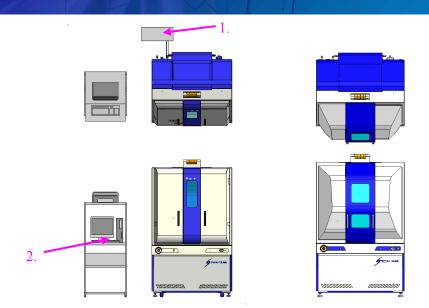
### Column

#### 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  $K\beta$  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  $K\beta$  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.

| Taygot | Excitation      | Optimun<br>(equivalent | 0                       |
|--------|-----------------|------------------------|-------------------------|
| Target | voltage<br>(kV) | Intensity at maximum   | P/B ratio at<br>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                   |

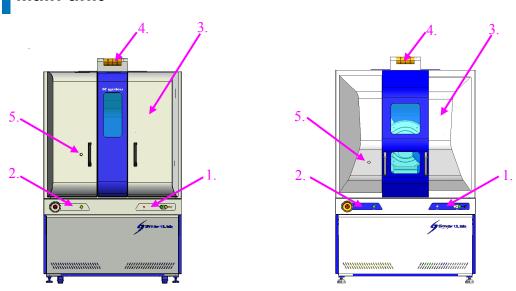
# **Names of Parts**



General view of SmartLab (Left: 2080A211/2080B211/2080A411/2080B411, Right: 2080B212)

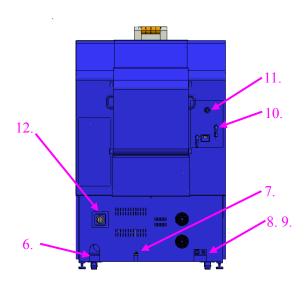
| 1. Rotary pump | Pump for maintaining the vacuum in the X-ray generator. (2080A211/2080A411) |
|----------------|---|
| 2. Control PC  | PC used to control SmartLab.  |

# Main unit



Front view of SmartLab (Left: 2080A211/2080B211/2080A411/2080B411, Right: 2080B212)

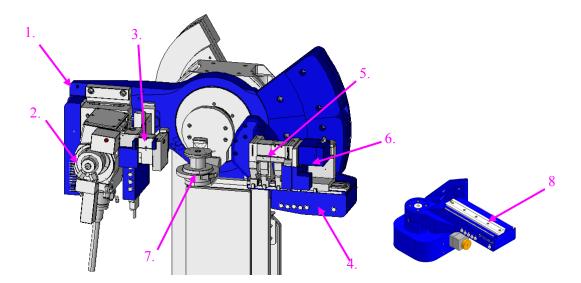
| 1. Main panel         | Panel used to start and stop SmartLab.                     |
|-----------------------|--|
| 2. Operating panel    | Panel used to turn the internal light on/off.              |
| 3. Door               | This door is opened to change samples and optical devices. |
| 4. X-ray warning lamp | Lights when X-rays are generated.                          |
| 5. Door-lock button   | Lock/Unlock the door.                                      |



#### Rear view of SmartLab

| 6. Power input connector         | Connect to external power supply.            |
|----------------------------------|--|
| 7. LAN cable hole                | Pass the LAN cable of the control PC for     |
| 7. Er ii v edote note            | SmartLab through this hole.                  |
|                                  | Connect the power cable of the rotary pump   |
| 8. Connectors for rotary pump    | here.  |
|                                  | (2080A211/2080A411)                          |
| 9. Connectors for water          | Connect the control cable of the water       |
| circulation pump                 | circulation pump (short-cut valve) here.     |
| 10. Inlet and outlet for cooling | Connect the cooling water hoses of the water |
| water                            | circulation pump here.                       |
| 11 Ding for rotory numn          | Connect the rotary pump pipe here.           |
| 11. Pipe for rotary pump         | (2080A211/2080A411)                          |
| 12 Circuit brooker               | Circuit breaker for the power supply to the  |
| 12. Circuit breaker              | main unit.                                   |

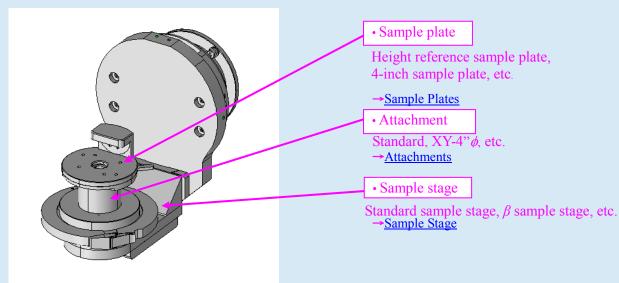
# Goniometer



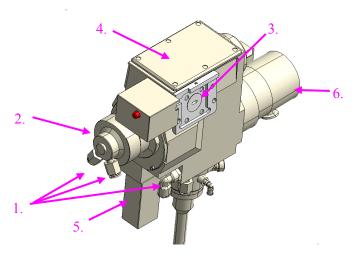
### Goniometer

| 1. Theta_s arm      | Arm for controlling X-ray beam incident angle.                     |
|---------------------|--|
| 2. X-ray tube       | X-ray generating device.   |
| 3. Incident optics  | Optical device for achieving desired incident X-ray conditions.    |
| 4. Theta_d arm      | Arm for controlling the X-ray detector angle.                      |
| 5. Receiving optics | Optical device for achieving desired X-ray receiving conditions.   |
| 6. Detector         | X-ray detector.  |
| 7. Sample stage     | Adjusts the position and orientation of the sample to be measured. |
| 8. In-plane arm     | Theta_d arm used for in-plane measurements. (2080B212)             |

## **Names of Parts** Theta s arm Selection slit PB, BB, SA, etc. CBO unit (for Cu target) • Incident length-limiting slit 10, 15 (mm), etc. Incident slit box • Incident parallel slit adaptor/monochromator Soller slit 5.0 deg, Ge(220)x2 CBO-f etc. Incident optics unit (standard incident optics unit) Theta darm • $K\beta$ filter Receiving slit box # 1 • Receiving optical device adaptor/analyzer PSA 0.114 deg, Ge(220)x2 etc. → Receiving optics unit # 1 • Receiving parallel slit adaptor • Soller slit/in-plane PSA Soller slit 5.0 deg, In-plane PSA 0.5 deg etc. → Receiving optics unit # 2 90000 • Scintillation counter, D/teX ultra /diffracted beam monochromator for Cu →<u>Detector</u> Sample attachment • Sample plate Height reference sample plate, 4-inch sample plate, etc.

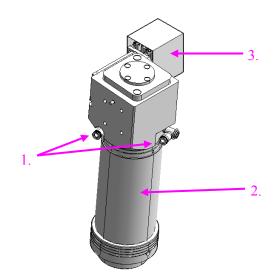


# X-ray tube



**Rotating anode X-ray tube (2080A211/2080A411)** 

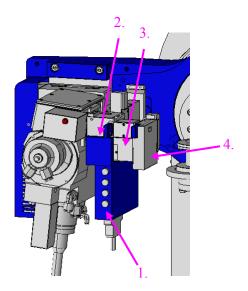
| 1. Cooling water pipe          | Connect the X-ray generator cooling water pipe. |
|--------------------------------|---|
| 2. Rotary target               | X-ray generating source.                        |
| 3. Shutter                     | Controls X-ray emissions.                       |
| 4. Filament replacement window | Window for replacing the filament.              |
| 5. Ion gauge                   | Measures the vacuum in the X-ray generator.     |
| 6. Turbo molecular pump (TMP)  | Vacuates the X-ray generator.                   |



Sealed X-ray tube (2080B211/2080B212/2080B411)

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

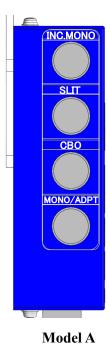
# Theta\_s arm

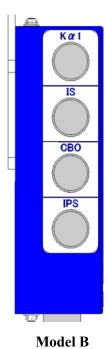


Theta\_s arm

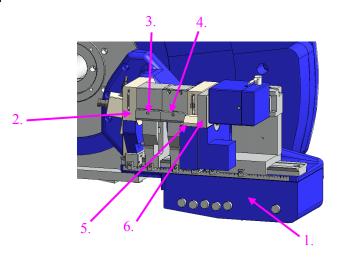
| 1. Incident connector box       | The control cable of the optical device connects here. |  |  |  |
|---------------------------------|--|--|--|--|
| 2. Cross beam optics (CBO) unit | Optics unit for switching optics.                      |  |  |  |
| 3. Incident optics unit         | Install a soller slit, a monochromator, etc. here.     |  |  |  |
| 4. Incident slit box            | Variable slit on the incident side.                    |  |  |  |

The incident connector box differs depending on the type of SmartLab model. The names and appearance of the connector ports of each model are shown below.





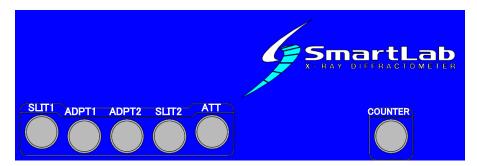
# Theta\_d arm



Theta\_d arm

| 1. Receiving connector box   | The control cables for the optical devices connect here. |
|------------------------------|--|
| 2. Receiving slit box # 1    | Variable slit on receiving side.                         |
| 3. Receiving optics unit # 1 | Receiving optical devices (analyzer) are installed here. |
| 4. Receiving optics unit # 2 | Soller slits, etc. are installed here                    |
| 5. Receiving slit box # 2    | Variable slit on receiving side.                         |
| 6. Attenuator                | Adjusts X-ray intensity.                                 |

The receiving connector box differs depending on the type of SmartLab model. The names and appearance of the connector ports of each model are shown below.

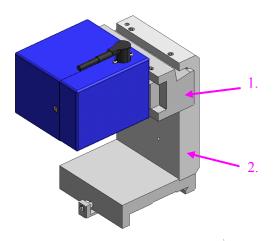


Model A



Model B

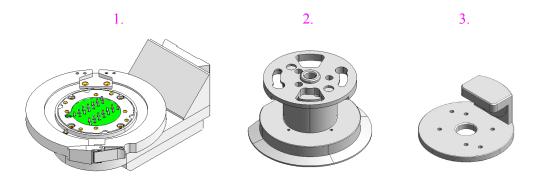
# **Detector mounting bracket**



#### **Detector mounting bracket**

| 1. Base             | Used to secure the detector in place. |
|---------------------|---------------------------------------|
| 2. Detector adaptor | Used to attach the detector.          |

# Sample stage and attachments



### Sample stage and attachments

| 1. Sample stage | Sample attachment unit.                           |
|-----------------|---|
| 2. Attachment   | Attachment to be mounted to the sample stage.     |
| 3. Sample plate | Plate on which sample holder or sample is placed. |

# **Software Configuration**

# **Control software**

| 9 | Sma |
|---|-----|

SmartLab Guidance Used to control SmartLab and perform measurements.

## Data processing software

| <u></u> | Standard data processing | Performs basic data processing such as smoothing, background removal, etc. |
|---------|--------------------------|--|
|         | PDXL                     | Performs basic data processing.  |

## Data display software (option)

| M         | Thin Film data processing (9240H162) | Performs a brief analysis of rocking curve as well as basic data processing.                  |  |  |
|-----------|--------------------------------------|---|--|--|
| <i>\$</i> | Profile viewer (9240H162)            | Superimposes multiple measurement data for display.   |  |  |
|           | 3D Explore (9240H403)                | Displays two-dimensional data such as RSM, pole figure, etc. Performs simple data processing. |  |  |

# Analysis software (option)

|          | PDXL(9240J702, 9240J703, 9240J710)                    | Performs the additional functions such as phase ID analysis, quantitative analysis, Rietveld analysis, etc.                               |
|----------|---|---|
|          | GlobalFit: reflectivity analysis (9240H155)           | Performs reflectivity analysis.   |
| <b>O</b> | NANO-Solver(9289H901)                                 | Performs particle size/pore diameter distribution analysis.   |
| <b>M</b> | Pore size analysis (9240H231)                         | Performs analysis of particle size/pore diameter distribution of thin film, accounting for diffuse scattering from surface and interface. |
| W        | GlobalFit: extended rocking curve analysis (9240H145) | Performs simulation and analysis of rocking curve.  |
|          | QA-Tex(9240H601)                                      | Performs quantitative analysis of the preferred orientation of a sample.  |
|          | CSDA(9240H651)  | Performs crystallite size distribution analysis.  |

### Column

#### 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  $\theta$  of the incident X-ray beam and X-ray absorption coefficient  $\mu(/cm)$  in the following formula to obtain x cm, the effective thickness.

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

| 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 |

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  $\theta = 90^{\circ}$ .

| Su             | Substance                                |            | Mass abs | sorption co | efficient μ/, | o (cm²/g) | Thickness for 90% utilization rate (mm) |             |        |        |
|----------------|--|------------|----------|-------------|---------------|-----------|---|-------------|--------|--------|
|                | botance                                  | $(g/cm^3)$ | MoKα     | CuK α       | CoK α         | CrK α     | $MoK\alpha$                             | $CuK\alpha$ | CoK α  | CrKα   |
|                | $\alpha$ -Al <sub>2</sub> O <sub>3</sub> | 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  |
|                | $\alpha$ -SiO $_2$                       | 2.65/2     | 3.71     | 34.5        | 53.1          | 108       | 2.34                                    | 0.252       | 0.164  | 0.080  |
| Powder         | CaCO <sub>3</sub>                        | 2.71/2     | 8.02     | 70.9        | 107           | 205       | 1.06                                    | 0.120       | 0.079  | 0.041  |
|                | $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> | 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 <sub>2</sub> 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 |
|                | Al                                       | 2.70       | 5.16     | 48.6        | 74.8          | 152       | 0.826                                   | 0.088       | 0.057  | 0.028  |
|                | α-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  |
| . S.yinor      | Vinyl chloride                           | 1.41       | 6.73     | 62.0        | 94.2          | 186.2     | 1.21                                    | 0.132       | 0.086  | 0.044  |
| Single crystal | Si                                       | 2.42       | 6.44     | 60.6        | 93.3          | 189       | 0.738                                   | 0.079       | 0.051  | 0.025  |
| substance      | $\alpha$ -SiO $_2$                       | 2.65       | 3.71     | 34.5        | 53.1          | 108       | 1.17                                    | 0.126       | 0.082  | 0.040  |

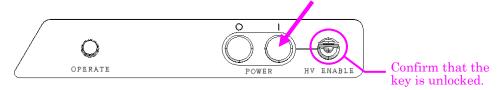
# **Turning On and Off SmartLab**

Described below are the procedures for turning on and off SmartLab.

### Turning on SmartLab

1 Make sure that the HV ENABLE key on the main panel is unlocked (the key is in the horizontal position).

If it is locked (the vertical position), turn the key clockwise to unlock it.



#### Main panel

- 2 Press the "|" ON button.
- **3** Make sure the OPERATE lamp lights up in green.



The OPEARATE lamp blinks while SmartLab is starting up and when an error occurs. For more information, see Appendix Using the OPERATE Lamp.

## Starting SmartLab Guidance

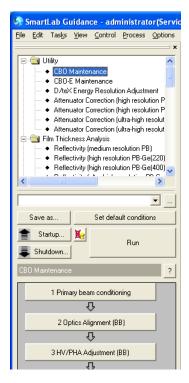
- **1** Click the Start button. Select All Programs → Rigaku → SmartLab Guidance, then select SmartLab Guidance.
- 2 The login window will appear. Enter the registered user name and password in the Login name and Password boxes, and click the OK button. (When launching the software for the very first time, enter administrator in the Login name box and click the OK button without entering a password.)
- **3** The control axes are initialized after the software is launched.



For detailed information on initialization, refer to Section 14 of the "Reference Manual" Help Topic of the online help section of the *SmartLab Guidance software*.

### **Startup**

Click Package Measurement under the Task menu and click Startup on the displayed Package Measurement flow bar.



Startup



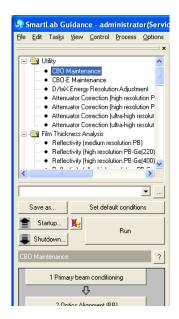
When not using the water circulation pump, make sure the cooling water is flowing properly. If the cooling water is not flowing or the amount, pressure of water are incorrect, an alarm will be issued and remain active for three minutes after the startup procedure is initiated. Adjust the water volume to shut off the alarm within three minutes, then repeat the startup procedure. For information on how to adjust the water volume, refer to the instruction manual of the water circulation pump.



For the startup procedure, refer to Section 2.1 of the "CBO Maintenance Package Measurement" Help Topic of the online help section of the *SmartLab Guidance software*.

### **Shutdown**

Click **Package Measurement** under the **Task** menu and click **Shutdown** in the displayed **Package Measurement** flow bar.



Shutdown

#### **CAUTION**



When not using the water circulation pump, wait three minutes following shutdown (i.e., three minutes after the yellow lamp located at the upper section of SmartLab goes out), then halt the supply of cooling water. (Halting the cooling water supply in less than three minutes may result in damage to SmartLab.)





For more information on the shutdown procedure, refer to Section 2.6 of the "CBO Maintenance Package Measurement" Help Topic of the online help section of the *SmartLab Guidance software*.



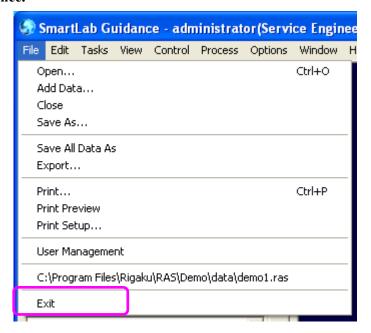
While SmartLab is not used, it is recommended to turn off only the X-ray, and do not turn off the vacuum unit. In this case, the controller remains powered; therefore SmartLab can be started from SmartLab Guidance. If SmartLab is unused for a while (e.g. during the New Year Holidays), press the "O" OFF button to turn the power of SmartLab off.



## **Exiting SmartLab Guidance**

After turning off the X-rays, exit SmartLab Guidance.

**1** Click the Exit command under the File menu to exit SmartLab Guidance.



## **Turning off SmartLab**

If SmartLab is unused for a while (New Year Holidays, etc.), turn off SmartLab according to the following procedure.

- **1** Check the SmartLab status (e.g., confirm that measurements have been completed).
- **2** Press the "O" OFF button on the main panel.



main panel

3 Make sure the OPERATE lamp has turned off.



This procedure completely shuts down the SmartLab internal power supply.

# **Opening and Closing the Door**

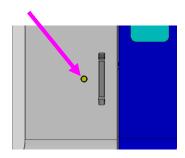
To prevent human exposure to X-ray radiation, the shutter and door are equipped with an interlocking function. To ensure safety, when changing samples or optical devices during X-ray generation, open and close the door by the following procedure:

## Opening the door



Under normal conditions, the door is locked and cannot be opened.

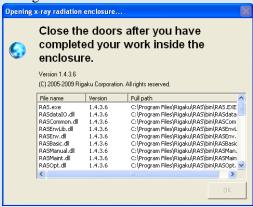
**1** Press the DOOR LOCK button on the door.



- 2 The shutter will close and the door will be unlocked.
- **3** The DOOR LOCK button will flash, and you will hear beeping.
- 4 Open the door.



The shutter cannot be opened when the door is unlocked. The following message also appears when the door is unlocked. SmartLab Guidance cannot be operated until the door is locked again.



## Closing the door

- **1** Close the door of the radiation enclosure.
- **2** Press the DOOR LOCK button on the door.
- **3** When the door is properly locked, the DOOR LOCK button stops flashing, and the beeping stops. The instrument is then ready for operation.

## **Emergency stop 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, press the "|" ON button on the main panel to restart SmartLab. (see <a href="Turning on SmartLab">Turning on SmartLab</a>, <a href="Starting SmartLab Guidance">Startup</a>).



After restarting SmartLab, also restart SmartLab Guidance.



**Emergency stop switch (EMO)** 

### Turning on/off the internal lamp (LAMP)

Press once the LAMP button to turn on the internal lamp. Press again to turn it off.



**LAMP** button

# **Optics**

This section introduces the names of parts and describes how they are installed. For information on how to use the  $K\alpha_1$  unit, refer to the instruction manual of the  $K\alpha_1$  unit.

## **CBO** unit (for Cu target)

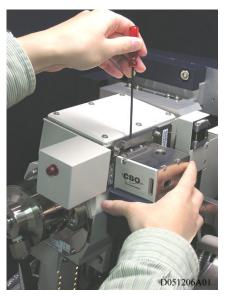
The CBO unit is designed for characteristic X-rays ( $K\alpha$  X-rays) generated by the standard Cu target. Changing selection slits allows switching between divergent and parallel/convergent beams using a multilayer mirror. A parallel beam can also be converted into a narrow beam for small-angle scattering measurements or a small beam for micro area measurements.

Fine adjustments of the multilayer mirror can be performed from SmartLab Guidance.

Table CBO unit

| Unit name   | Abbreviation | Illustration |
|---|--------------|--------------|
| CBO unit (for Cu target only) Applicable to Cu wavelength, and enables the switching between divergent and parallel beams.                          | CBO(Cu)      |              |
| CBO unit (for Cu and Co targets) (option) Applicable to both Cu and Co wavelengths, and enables the switching between divergent and parallel beams. | CBO(Cu/Co)   |              |
| CBO-E unit (for Cu target only) (option: 2220C401) Applicable to Cu wavelength, and enables the switching between divergent and convergent beams.   | СВО-Е        |              |

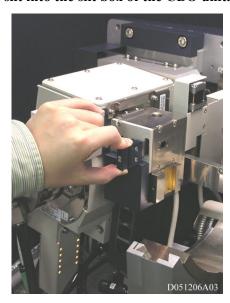
**1** Attach the CBO unit to the shutter. Press the CBO unit against the surface in the back and secure the unit in place with the Allen wrench provided.



2 Insert the cable into incident connector box.
(Name of cable and connector: CBO)



## **3** Insert a selection slit into the slit box of the CBO unit.



**Table Selection slits** 

| Name of optics   | Corresponding CBO              | Abbreviation | Illustration |
|--|--------------------------------|--------------|--------------|
| For para-focusing  | CBO(Cu)<br>CBO(Cu/Co)<br>CBO-E | ВВ           |              |
| For para-focusing (Co) (included in the CBO (Cu/Co) unit)        | CBO(Cu/Co)                     | ВВ Со        |              |
| For parallel beam (included in the CBO(Cu) and CBO(Cu/Co) units) | CBO(Cu)<br>CBO(Cu/Co)          | РВ           |              |
| For convergent beam (included in the CBO-E unit)                 | СВО-Е                          | СВ           |              |
| For micro area measurements (option: 2431C302)                   | CBO(Cu)<br>CBO(Cu/Co)          | PB 0.3       |              |
| For small-angle scattering measurements (option: 2680J111)       | CBO(Cu)<br>CBO(Cu/Co)          | SA           |              |
| For micro area measurements (option: 2680G111)                   | CBO(Cu)<br>CBO(Cu/Co)<br>CBO-E | MA           |              |
| For small-angle scattering high-intensity measurements (option)  | CBO(Cu)<br>CBO(Cu/Co)          | SA 0.1       |              |



The type of selection slit installed can be identified from SmartLab Guidance.

Table Selection slits for micro area measurements (option)

| Name of optics                   | Size            | Abbreviation | Illustration |
|----------------------------------|-----------------|--------------|--------------|
| Parallel beam (2431C302)         | <i>ϕ</i> 0.3 mm | PB 0.3       |              |
| Parallel beam (2431C301)         | φ0.1 mm         | PB 0.1       |              |
| Point source X-rays*1 (2431C202) | φ0.3 mm         | BB 0.3       |              |
| Point source X-rays*1 (2431C201) | <i>¢</i> 0.1 mm | BB 0.1       |              |

<sup>\$1</sup> : Switchable to the point source X-rays only using the sealed tube type (2080B211/2080B212/2080B411).



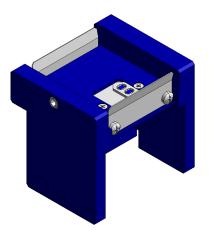
The type of selection slit installed can be identified from SmartLab Guidance. Above selection slits is used in combination with a collimator as a double pinhole collimator.

## Incident optics unit (standard incident optics unit)

Install an optical device to condition the incident X-ray beam. Select an optical device from three types: parallel slit, 2-bounce monochromator, and 4-bounce monochromator.

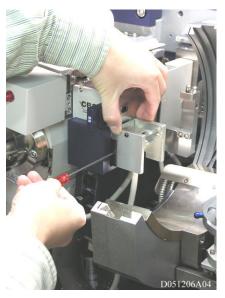
#### Incident parallel slit adaptor (IPS adaptor)

This adaptor is used to install a parallel slit.



Incident parallel slit adaptor

1 Install the incident parallel slit adaptor 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.



Connect the cable to the incident connector.

(Name of cable and connector: MONO/ADPT (Model A) or IPS (Model B))



**3** Install the parallel slit on the adaptor and secure it in place with the Allen wrench provided

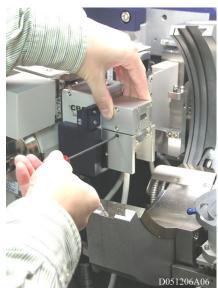


Table Incident parallel slits

| Туре   | Divergence<br>limit angle | Abbreviation          | Illustration |
|--|---------------------------|-----------------------|--------------|
| Open (option: 2680D115)  | None                      | Soller_Slit_open      |              |
| Soller slit  | 5°                        | Soller_Slit_5.0 deg   |              |
| Soller slit<br>(option: 2680D114)                                | 2.5°                      | Soller_Slit_2.5 deg   |              |
| In-plane PSC<br>(Parallel Slit Collimator)<br>(option: 2680D113) | 1.0°                      | In-plane_PSC_1.0 deg  |              |
| In-plane PSC (option: 2680D112)                                  | 0.5°                      | In-plane_PSC_0.5 deg  |              |
| In-plane PSC (option: 2680D111)                                  | 0.15°                     | In-plane_PSC_0.15 deg |              |

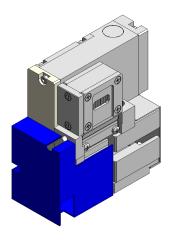


The type of installed incident parallel slit can be identified from SmartLab Guidance.

#### 2-bounce monochromator (option)

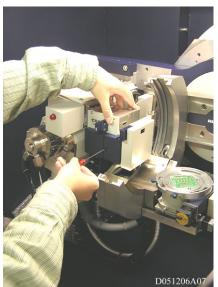
This optical device produces a monochromatized X-ray beam using a 2-bounce channel cut crystal.

Fine alignment of the channel cut crystal is performed from SmartLab Guidance. A dedicated parallel slit can be installed after the channel cut crystal.



2-bounce monochromator

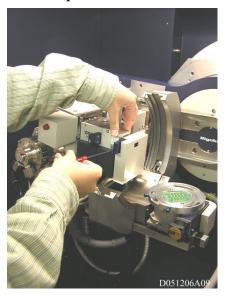
**1** Attach the 2-bounce monochromator to the CBO unit by sliding it down from the top section of the CBO unit. Secure it in place with the Allen wrench provided.



Connect the cable to the incident connector.
(Name of cable and connector: MONO/ADPT (Model A) or IPS (Model B))



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



**Table 2-bounce monochromators** 

| Crystal                  | Diffraction plane | Abbreviation | Illustration |
|--------------------------|-------------------|--------------|--------------|
| Ge<br>(option: 2680B111) | 220               | Ge(220)x2    |              |
| Ge<br>(option: 2680B112) | 400               | Ge(400)x2    |              |



The type of installed crystal can be identified from SmartLab Guidance.

Table Parallel slits for 2-bounce monochromator

| Туре                              | Divergence | Abbreviation         | Illustration |
|-----------------------------------|------------|----------------------|--------------|
| Open (provided with the unit)     | None       | Soller_slit_open     |              |
| Soller slit<br>(option: 2680D213) | 5.0°       | Soller_slit_5.0 deg  |              |
| Soller slit<br>(option: 2680D212) | 2.5°       | Soller_slit_2.5 deg  |              |
| In-plane PSC (option: 2680D211)   | 0.5°       | In-plane_PSC_0.5 deg |              |

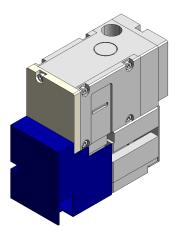
Tip

The type of parallel slit installed on the adaptor can be identified from SmartLab Guidance.

#### 4-bounce monochromator (option)

This optical device produces a monochromatized X-ray beam using two 2-bounce channel cut crystals.

Fine alignment of the channel cut crystals can be performed from SmartLab Guidance.



4-bounce monochromator

**1** Attach the 4-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.



Connect the cable to the incident connector.

(Name of cable and connector: MONO/ADPT (Model A) or IPS (Model B))



**Table 4-bounce monochromators** 

| Crystal                  | Diffraction plane | Abbreviation | Illustration |
|--------------------------|-------------------|--------------|--------------|
| Ge<br>(option: 2680B211) | 220               | Ge(220)x4    |              |
| Ge<br>(option: 2680B212) | 440               | Ge(440)x4    |              |



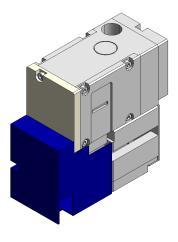
2

The type of installed crystal can be identified from SmartLab Guidance

### **CBO-f unit (option: 2220C301)**

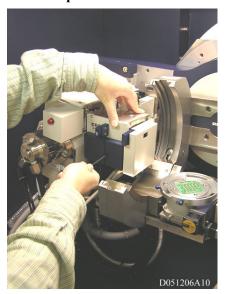
Converges the parallel beam in the longitudinal direction onto the sample. The optical device is used for the measurements of micro amount of a sample and of micro area.

Fine alignment of the CBO-f unit is performed from SmartLab Guidance.



**CBO-f** unit

1 Install the CBO-f unit to the theta\_s arm from the top, and secure it in place with the Allen wrench provided.



Connect the cable to the incident connector.

(Name of cable and connector: MONO/ADPT (Model A) or IPS (Model B))





2

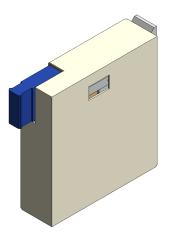
The type of installed crystal can be identified from SmartLab Guidance.

## Incident slit box

This variable slit box 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 small-angle scattering 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 incident connector.

(Name of cable and connector: SLIT (Model A) or IS (Model B))



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



Table Length-limiting slits

| Length of limiting slit                           | Abbreviation | Illustration |
|---|--------------|--------------|
| 15 mm   | 15           |              |
| 10 mm   | 10           |              |
| 5 mm  | 5            |              |
| 2 mm  | 2            |              |
| 0.5 mm<br>micro area optics<br>(option: 2680G111) | 0.5          |              |



The type of inserted length-limiting slit can be identified from SmartLab Guidance.

### **Collimator holder (option)**

Some types of pinhole collimators can be installed for micro area measurements.  $K\beta$  filter for Cu can also be installed (option).

Table Collimator holder (option)

| Name                            | Usage   | Illustration |
|---------------------------------|---|--------------|
| PB collimator holder (2431D201) | Used in combination with multilayer mirror or CBO-f unit. |              |
| BB collimator holder (2431D101) | Used in point focus optics.                               |              |

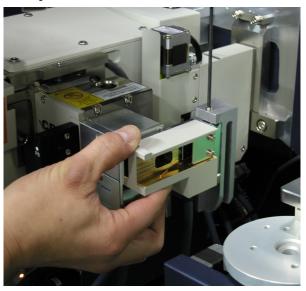
Table Incident filter (option)

| Name                        | Illustration |
|-----------------------------|--------------|
| Absorber case (2431H101)    |              |
| Kβ filter for Cu (2431H201) |              |



 $K\beta$  filter for various wavelength can be supplied. Please contact the sales representative who you purchased the instrument from.

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



Connect the cable to the incident connector.(Name of cable and connector: SLIT (Model A) or IS (Model B))





3 Insert the pinhole collimator into the collimator holder.

**Table Pinhole collimators (option)** 

| Pinhole diameter        | Abbreviation | Illustration |
|-------------------------|--------------|--------------|
| φ 0.2 mm<br>(2431E103)  | 0.2          |              |
| φ 0.1 mm<br>(2431E102)  | 0.1          |              |
| φ 0.05 mm<br>(2431E101) | 0.05         |              |

### CAUTION



Do not use the pinhole collimator (option) with the XY-4" phi attachment. When using the pinhole collimator with the XY-4" phi attachment, the end of the pinhole collimator collides with the sample and/or the sample plate from the translation of X and Y axes, and thus the pinhole collimator, the sample, and the sample stage may become damaged.



The type of inserted pinhole collimator can be identified from SmartLab Guidance.

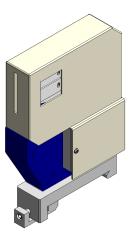
# Receiving slit box # 1

This slit box is installed on the sample side of the theta\_d arm.

#### Standard receiving slit box # 1

For para-focusing optics, this slit box controls the anti-scatter slit width to reduce the level of background scatter from the sample. For parallel beam optics, it functions as the first slit in a double-slit receiving system.

The slit width can be controlled from SmartLab Guidance. A K $\beta$  filter can be inserted to remove K $\beta$  X-rays from characteristic X-rays.



Standard receiving slit box # 1

**1** Attach the standard receiving slit box # 1 to the rail of the theta\_d arm. Secure it in place with the Allen wrench provided.

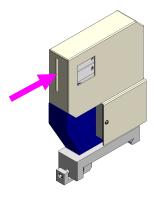


**2** Connect the cable to the receiving connector box.

(Name of cable and connector: SLIT1 (Model A) or RS1 (Model B))

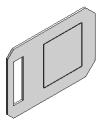


**3** A K $\beta$  filter may be inserted.





Check whether a  $K\beta$  filter is inserted from SmartLab Guidance.



K

ß filter (Ni foil)



The K $\beta$  filter reduces the K $\beta$  characteristic X-rays by approximately 97.5% while only reducing the K $\alpha$  characteristic X-rays by approximately 50%. Therefore almost all K $\beta$  X-rays can be removed. (Used for measurements based on the para-focusing method.)



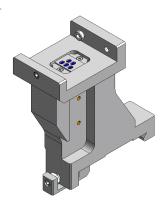
**4** The installation position can be read from the scale on the theta\_d arm.

# Receiving optics unit # 1

This unit attaches after receiving slit box # 1. A parallel slit analyzer or 2-bounce crystal analyzer can be installed on receiving optics unit # 1.

### Receiving optical device adaptor (ROD adaptor)

This adaptor is used to install a parallel slit analyzer (PSA). A parallel slit analyzer is installed so that the plates inside are mounted horizontally thereby providing angular resolution for the theta d axis.



Receiving optical device adaptor

**1** Attach the receiving optical device adaptor to the rail of the theta\_d arm. Secure it in place with the Allen wrench provided.



Connect the cable to the receiving connector.(Name of cable and connector: ADPT1 (Model A) or ROD (Model B))



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

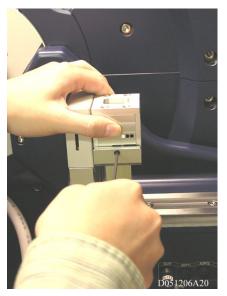


Table Parallel slit analyzers

| Aperture angle               | Length | Abbreviation  | Illustration |
|------------------------------|--------|---------------|--------------|
| Open                         | 45 mm  | PSA_open      |              |
| 1.0°<br>(option: 2680F113)   | 45 mm  | PSA_1.0 deg   |              |
| 0.5°                         | 45 mm  | PSA_0.5 deg   |              |
| 0.114°<br>(option: 2680F111) | 90 mm  | PSA_0.114 deg |              |
| Open                         | 90 mm  | PSA_open      |              |



The standard configuration of SmartLab cannot incorporate either the PSA 0.114 deg or PSA open (90 mm) in conjunction with a receiving parallel slit. In order to do this, the configuration must be modified by, moving the receiving parallel slit adaptor, standard slit box # 2, attenuator, and detector (counter) to the right.

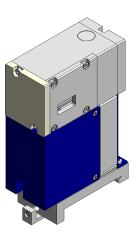


The type of parallel slit analyzer installed on the adaptor can be identified from SmartLab Guidance.

### 2-bounce analyzer (option)

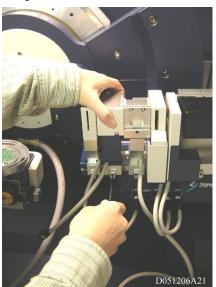
The 2-bounce channel cut analyzer provides increased resolution on the 2-theta axis by only allowing diffracted X-rays that satisfy the diffraction condition for the analyzer crystal to enter the detector.

Fine alignment of the channel cut crystal can be performed from SmartLab Guidance.



2-bounce analyzer

**1** Attach the 2-bounce analyzer to the theta\_d arm. Secure it in place with the Allen wrench provided.



2 Connect the cable to the receiving connector.
(Name of cable and connector: ADPT1 (Model A) or ROD (Model B))



**Table Crystal types** 

| Crystal                  | Diffraction plane | Abbreviation | Illustration |
|--------------------------|-------------------|--------------|--------------|
| Ge<br>(option: 2680C111) | 220               | Ge(220)x2    |              |
| Ge<br>(option: 2680C112) | 400               | Ge(400)x2    |              |



The type of crystal currently installed can be identified from SmartLab Guidance.

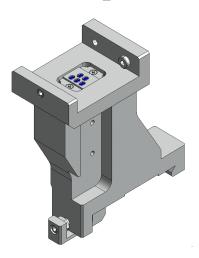
# Receiving optics unit # 2

This unit attaches to before receiving slit box # 2. A parallel slit can be installed in receiving optics unit # 2.

### Receiving parallel slit adaptor (RPS adaptor)

This adaptor is used to install a parallel slit.

A parallel slit is installed so that the plates inside are mounted vertically controlling axial divergence for the theta d axis.



Receiving parallel slit adaptor

**1** Attach the receiving parallel slit adaptor to the rail of the theta\_d arm. Secure it in place with the Allen wrench provided.



Connect the cable to the receiving connector.(Name of cable and connector: ADPT2 (Model A) or RPS (Model B))



**3** Attach a parallel slit to the adaptor. Secure it in place with the Allen wrench provided.



Table Receiving parallel slits

| Туре   | Aperture angle | Abbreviation           | Illustration |
|--|----------------|------------------------|--------------|
| Soller slit  | 5.0°           | Soller_slit_5.0 deg    |              |
| Soller slit<br>(option: 2680F114)                            | 2.5°           | Soller_slit_2.5 deg    |              |
| In-plane PSA<br>(use of PSA 1.0 deg)<br>(option: 2680F113)   | 1.0°           | In-plane_PSA_1.0 deg   |              |
| In-plane PSA<br>(use of PSA 0.5 deg)                         | 0.5°           | In-plane_PSA_0.5 deg   |              |
| In-plane PSA<br>(use of PSA 0.114 deg)<br>(option: 2680F111) | 0.114°         | In-plane_PSA_0.114 deg |              |



The type of receiving parallel slit currently installed on the adaptor can be identified from SmartLab Guidance.

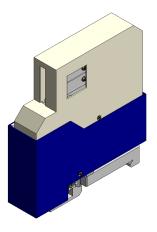
# Receiving slit box # 2

This slit box is installed in from of the detector on the theta\_d arm.

#### Standard receiving slit box # 2

The standard receiving slit box # 2 contains the variable receiving slit and controls measurement resolution in the para-focusing and double-slit receiving configurations.

If a double-slit receiving system is used, it will function as the second slit. Slit width and vertical position can be controlled from SmartLab Guidance.



Standard receiving slit box # 2

**1** Attach the standard receiving slit box # 2 to the rail of the theta\_d arm. Secure it in place with the Allen wrench provided.





When using the para-focusing method, set the installation position to 300 mm.



**2** Connect the cable to the receiving connector.

(Name of cable: SLIT2 (Model A) or RS1/RS2 (Model B))

(Name of connector: SLIT2 (Model A) or RS2 (Model B))



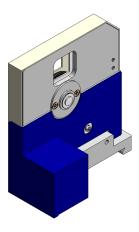
# **Attenuator**

The attenuator adjusts the intensity of the detected X-rays.

#### Standard attenuator

The system uses several types of attenuators on a rotating disc. Attenuators for optics alignment are also built-in.

The type of attenuator can be switched from SmartLab Guidance. Attenuators can be switched automatically for various measurements based on detected intensity.



Standard attenuator

**1** Attach the standard attenuator to the rail of the theta\_d arm. Secure it in place with the Allen wrench provided.



### **2** Connect the cable to the receiving connector.

(Name of cable and connector: ATT)



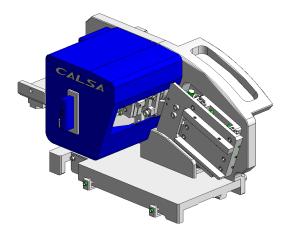


The attenuator types used during actual measurements are as follows: Open, 1/70, 1/1000, 1/10000. The attenuator types used during alignments are as follows: 9 kWBB, 9 kWPB, 3 kWBB, 3 kWPB for alignment.

# Receiving optics unit CALSA (option: 2680K201)

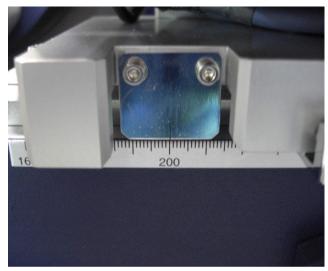
CALSA is a multi-crystal analyzer, which is used by installing onto the theta\_d arm. It achieves the ultrahigh resolution of the X-ray diffraction beam from a sample.

The positions of the analyzer crystals and detector can be aligned from SmartLab Guidance.



**CALSA** 

- 1 Remove all the units installed on the theta d arm.
- **2** Attach CALSA on the rail of the theta\_d arm and set the installation position to 200 mm. The position can be read from the scale on the theta\_d arm.



**3** Secure it in place with the Allen wrench provided.



**4** For Model A: Connect the ADPT1, ADPT2, and SLIT2 cables to the ADPT1, ADPT2, and SLIT2 connectors on the receiving connector box.

For Model B: Connect the ROD, RPS, and RS2 cables to the ROD, RPS, and RS2 connectors on the receiving connector box.



## Receiving parallel slit

Install the parallel slit in a manner such that the stacking direction of the foils should be the same as the direction of the theta\_d axis scan (the direction of axial divergence).



Table Receiving parallel slit

| Туре   | Aperture angle | Abbreviation           | Illustration |
|--|----------------|------------------------|--------------|
| Soller slit  | 5.0°           | Soller slit 5.0 deg    |              |
| Soller slit<br>(option: 2680F114)                            | 2.5°           | Soller slit 2.5 deg    |              |
| In-plane PSA<br>(use of PSA 1.0 deg)<br>(option: 2680F113)   | 1.0°           | In-plane PSA 1.0 deg   |              |
| In-plane PSA<br>(use of PSA 0.5 deg)                         | 0.5°           | In-plane PSA 0.5 deg   |              |
| In-plane PSA<br>(use of PSA 0.114 deg)<br>(option: 2680F111) | 0.114°         | In-plane PSA 0.114 deg |              |



The type of the receiving parallel slit installed on the adaptor can be identified from SmartLab Guidance.

Rigaku

#### **CALSA** beam conditioner

The slit, attenuator, and knife edge are prepared to be used with CALSA. Insert the beam conditioner into CALSA to condition the X-ray beams.



Table Beam conditioner

| Туре                     | Abbreviation            | Use  | Illustration |
|--------------------------|-------------------------|--|--------------|
| Knife edge               | KE                      | Cuts off the scattering X-rays from the sample in data measurements. |              |
| reference Receiving slit | ARS                     | Used for the 2-theta axis alignment in optics alignment.             |              |
| Attenuator               | ATT 9 kW<br>or ATT 3 kW | Used to attenuate the X-ray intensity in optics alignment.           |              |



The type of the beam conditioner inserted into CALSA can be identified from SmartLab Guidance.

#### **Detector (D/teX Ultra)**

Detects X-rays diffracted from CALSA.



D/teX Ultra

**1** Connect the power cable and LAN cable to D/teX Ultra.



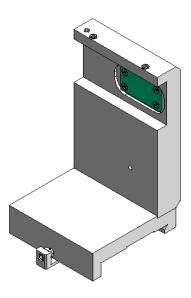
**2** Attach D/teX Ultra to the detector adaptor of CALSA. Secure it in place with the Allen wrench provided.



# **Detector**

# **Detector adaptor**

Used to attach the detector.



**Detector adaptor** 

**1** Attach the detector adaptor to the rail of the theta\_d arm. Secure it in place with the Allen wrench provided.



Connect the cable to the receiving connector.

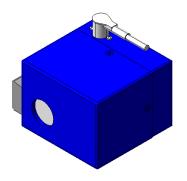
(Name of cable and connector: COUNTER (Model A) or DETECTOR (Model B))



# Scintillation counter (SC-70S)

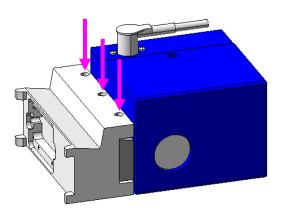
The scintillation counter is attached to the detector adaptor. It detects X-rays received by the receiving optics.

Check whether the scintillation counter is installed from SmartLab Guidance.

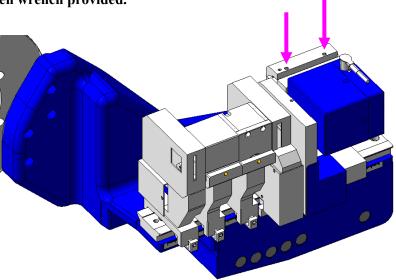


**Scintillation counter (SC-70S)** 

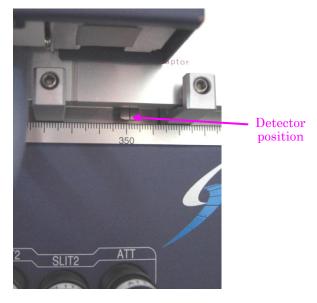
**1** When detecting X-rays from the receiving optics directly (in absence of the diffracted beam monochromator), install the scintillation counter to the base with the Allen wrench provided.



**2** Attach the base to the detector adaptor. Secure it in place with the Allen wrench provided.



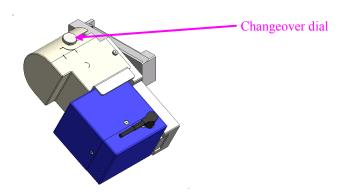
**3** The installation position can be read from the scale on the theta\_d arm.



## DBM unit for Cu (option: 2726H112)

This unit is used to monochromatize the diffracted beam such that only  $K\alpha$  X-rays are detected.

Change the curvature of the monochromating crystal from Bent (para-focusing geometry) to Flat (parallel beam geometry) by rotating the dial on the top of the monochromator housing.

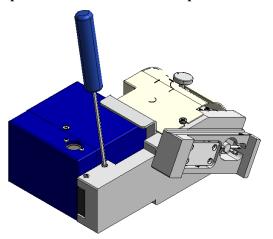


DBM (diffracted beam monochromator) unit for Cu

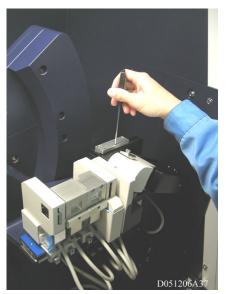


Diffracted beam monochromator unit using Cu for para-focusing optics only (Bent only) can be supplied (2726H113).

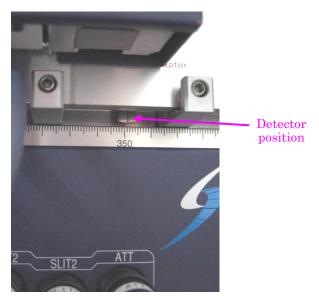
**1** Install the scintillation counter on the diffracted beam monochromator unit. Secure it in place with the Allen wrench provided.



**2** Install the unit on the detector adaptor. Secure it in place with the Allen wrench provided.



**3** The unit installation position can be read from the scale on the theta\_d arm.





When using the monochromator in Bent mode with para-focusing optics, the unit must be installed at a specific position for a specific target given in the table below. When using the monochromator in Flat mode, the exact position is not important.

| Type of target | Installation position |
|----------------|-----------------------|
| Cu             | 351.5 mm              |
| Co             | 359.8 mm              |
| Fe             | 364.7 mm              |
| Cr             | 376.1 mm              |

#### 4 The monochromator slit (RSm) may be inserted into the unit.



Check whether the monochromator slit (RSm) is inserted from SmartLab Guidance.





Use the RSm only when using the monochromator in Bent mode for the para-focusing method. The RSm is not used when using the monochromator in Flat mode.

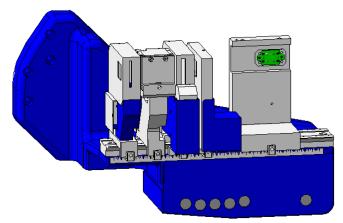
# D/teX Ultra (option)

D/teX Ultra is a 1D detector in which small detecting devices are arranged. Since D/teX Ultra detects X-ray intensity efficiently, the diffraction peaks of minor components can be detected with a high speed scan.

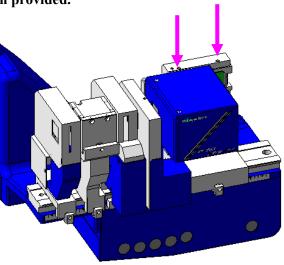
D/teX Ultra should be installed to the detector adaptor. Check whether D/teX Ultra is installed from SmartLab Guidance.



- 1 When detecting X-rays from the receiving optics directly (in absence of the diffracted beam monochromator), install D/teX Ultra to the base with the Allen wrench provided.
- 2 Remove the receiving optical device adaptor (see <u>Receiving optical</u> <u>device adaptor (ROD adaptor)</u>), move the receiving parallel slit adaptor (see <u>Receiving parallel slit adaptor (RPS adaptor)</u>), standard receiving slit box 2 (see <u>Standard receiving slit box # 2</u>), and standard attenuator (see <u>Standard attenuator</u>) to the incident side, and then install the detector adaptor.



3 Install the base to the detector adaptor, and secure it in place with the Allen wrench provided.



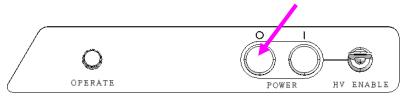


When the measurement is made in para-focusing optics, install the detector adaptor at position 351.5 mm



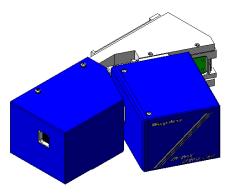
-Detector position

4 Connect the power cable and communication cable to the connector on the back of D/teX Ultra. Before connecting the cables, press the "O" OFF button to turn off the power of SmartLab.



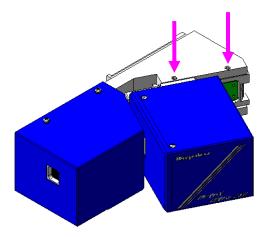
# DBM unit for Cu for D/teX Ultra (option: 2726P101)

This unit is used to monochromatize the diffracted beam such that only  $K\alpha$  X-rays are detected.

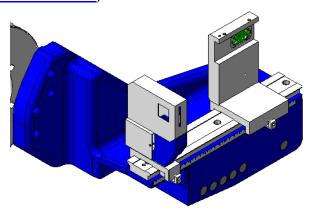


DBM (diffracted beam monochromator) unit for Cu (for D/teX Ultra)

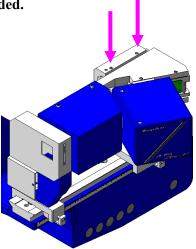
**1** Install D/teX Ultra on the diffracted beam monochromator unit. Secure it in place with the Allen wrench provided.



Remove the receiving optical device adaptor (see <u>Receiving optical</u> device adaptor (ROD adaptor)), receiving parallel slit adaptor (see <u>Receiving parallel slit adaptor (RPS adaptor)</u>), standard receiving slit box # 2 (see <u>Standard receiving slit box # 2</u>)), and standard attenuator (see <u>Standard attenuator</u>).



3 Install the unit on the detector adaptor. Secure it in place with the Allen wrench provided.





When the measurement is made in para-focusing optics, install the detector adaptor at position 351.5 mm.



Detector position

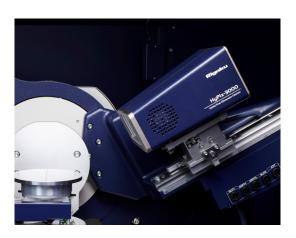
## HyPix-3000 (option: A00001644)

HyPix-3000, hybrid multi-dimensional pixel detector, has an ultra-high dynamic range and high-sensitivity. 0D, 1D and 2D measurement can be done by the seamless detection mode.

Attach HyPix-3000 to the mount holder and install the mount holder to SmartLab. The HyPix-3000 can be positioned vertically by using the vertical mount holder for SmartLab (option).



HyPix-3000





Installation of HyPix-3000

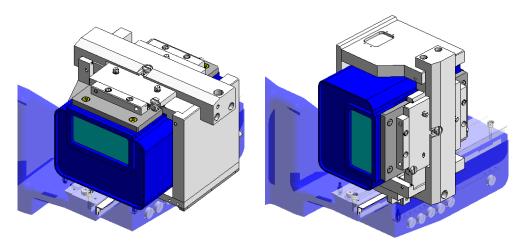
(left: Horizontal mount holder (standard), right: Vertical mount holder (option))



For detailed information on HyPix-3000, refer to the "SmartLab + HyPix-3000 System Instruction Manual" Help Topic of the online help section of the *SmartLab Guidance software*.

# PILATUS (option: 5742A300)

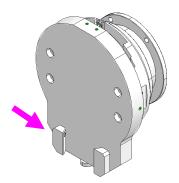
PILATUS is a low-noise high-sensitivity semi-conductor hybrid 2D detector. Attach PILATUS to the PILATUS holder and install the PILATUS holder to SmartLab, PILATUS can be placed either horizontally or vertically.



Installation of PILATUS (left: horizontal, right: vertical)

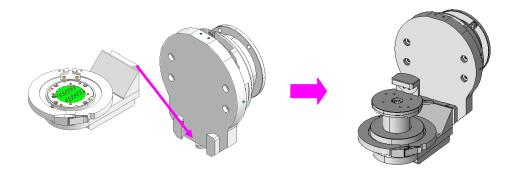
# Sample Stage

To move the sample up and down, Z axis is installed on the SmartLab goniometer. This mechanism enables the sample surface to be adjusted when using a sample stage or an attachment.

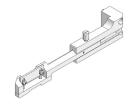


Sample height alignment unit

Sample stages can be installed to the sample height alignment unit. To install a sample stage, join the stage with the rail of the sample height alignment unit as shown in the following figure, and slide it from top down. Make sure that the stage is placed horizontally and secure it in place with the Allen wrench provided.



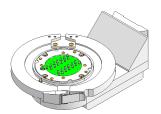
The direct beam stopper (option: provided with 5741A300) and knife edge (option: 2431G101) can also be installed to the sample height alignment unit. They will reduce the background intensity in high-speed measurements using D/teX Ultra.



Direct beam stopper

# Standard sample stage

This can be used in combination with attachments.

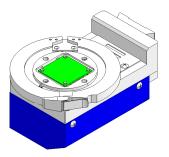


Standard sample stage

# $\beta$ sample stage (option: 2430B101)

This can be used in combination with attachments.

The rotational axis  $(\beta)$  on the sample surface plane enables to perform measurements by rotating the sample thereby reducing preferred orientation.

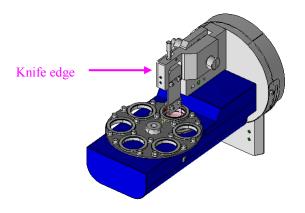


 $\beta$  sample stage

# ASC-6 (option: 2430H101)

Consecutive measurements can be performed by switching the six samples mounted on the turntable.

The rotational axis ( $\beta$ s) on the sample surface plane enables to perform measurements by rotating the sample thereby averaging the variation of particle sizes.



**Auto sample changer (ASC-6)** 

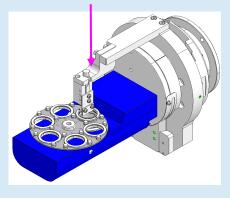
Table Standard accessories

| Name  | Illustration |
|---|--------------|
| Al sample holder with base 0.5 for ASC (2455E445) |              |
| Height alignment jig for ASC                      |              |



General-purpose knife edge (option: 2431G101) will be required to perform measurements in the reflection geometry using a knife edge. If you own the optional accessories for measurements in the vertical transmission geometry, the general knife edge is not necessary.

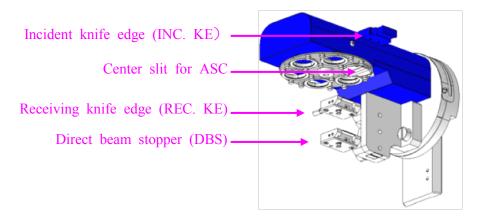
General-purpose knife edge



#### For vertical transmission geometry (option: 2455H105)

Measurements in the vertical transmission geometry can be performed when the convergent beam optics using the CBO-E unit is available. The transmission film sample holder (2455E463) is also necessary for this measurement.

Loosen the left lever and rotate the turntable 180 degrees clockwise for the vertical transmission geometry.



Switch to the vertical transmission geometry



When measurements are performed in the reflection geometry using a knife edge, install the reflection knife edge (REF. KE) to the extended base for ASC as shown below.

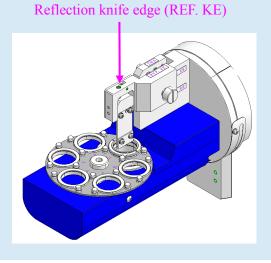
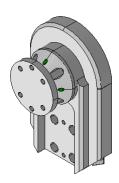


Table Accessories

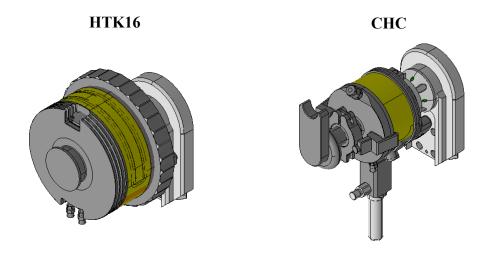
| Name                            | Illustration |
|---------------------------------|--------------|
| Transmission film sample holder |              |
| Center slit for ASC             |              |
| Incident knife edge(INC. KE)    |              |
| Extended base for ASC           |              |
| Direct beam stopper (DBS)       |              |
| Receiving knife edge (REC. KE)  |              |
| Reflection knife edge (REF. KE) |              |
| Mylar foil                      | _            |

# Anton Paar sample stage

Measurements can be performed using the Anton Paar sample stages equipped with temperature adjustment mechanism. The stages are: HTK1200N, HTK16, HTK2000, XRK900, TTK450, and CHC. For information of how to use the sample stage and pipe arrangement, refer to the instruction manual of each stage.



Anton Paar sample stage adaptor



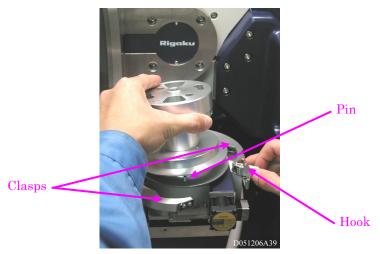
**Installation of the Anton Paar sample stages** 

# **Attachments**

You can switch between attachments with various adjust axes for different measurement purposes.

Described below is the method for changing attachments.

- 1 Click Manual Control under the Control menu. Move the chi axis to 0° and the phi axis to 0°.
- **2** Release the hook.
- **3** Open the clasps for the right and left sides. Remove the attachment.



- 4 Make sure the right and left clasps are open.
- 5 Install the attachment by aligning the groove of the attachment with the pin on the front.
- 6 Close the clasps and engage the front hook.



**Table Types of attachments** 

| Name   | Description  | Description |
|--|--|-------------|
| Standard attachment                          | This attachment has no moving axis. It is used for reflectivity measurements and measurements of powder and bulk samples.  |             |
| RxRy attachment (option: 2680A213)           | This attachment has Rx and Ry axes that permit tilt adjustments of two axes intersecting at right angles.  It is used for RSM measurements and various types of in-plane measurements.   |             |
| XY-20 attachment (option: 2680A211)          | This attachment 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-4" phi attachment<br>(option: 2680A212)   | This attachment has +/- 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. |             |
| Capillary spin attachment (option: 2430C101) | This attachment is used to spin the sample filled in a capillary during measurements.  |             |

#### **CAUTION**



Do not use the pinhole collimator (option) with the XY-4" phi attachment. When using the pinhole collimator with the XY-4" phi attachment, the end of the pinhole collimator collides with the sample and/or the sample plate from the translation of X and Y axes, and thus the pinhole collimator, the sample, and the sample stage may become damaged.



The type of attachment installed can be identified from SmartLab Guidance.

# **Sample Plates**

Select a sample plate based on the sample to be measured.

### Height reference sample plate

Insert a glass sample holder or aluminum sample holder into the height reference sample plate.

Place the height reference sample plate directly on the attachment.

To make adjustments, insert a center slit and Si powder reference sample.

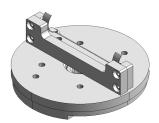


Height reference sample plate

## Transmission SAXS sample plate (option: 2680J111)

A transmission SAXS sample holder is inserted into the transmission SAXS sample plate.

The transmission SAXS sample plate is placed directly on the attachment.



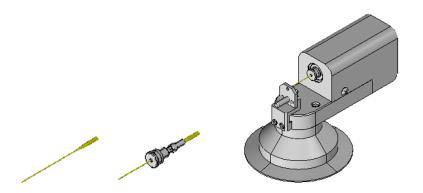
Transmission SAXS sample plate



- Normally the height reference sample plate and transmission SAXS sample plate should be used on top of the standard attachment. If using the height reference sample plate or transmission SAXS sample plate with an attachment other than the standard attachment, you must set each attachment axis to 0.
- To install/remove each sample plate to/from an attachment, see <u>Installing and removing</u> wafer sample plates.

## Capillary holder (option: 2430C101)

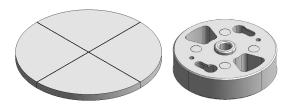
The capillary holder is used to mount the capillary sample onto the capillary spin attachment. The capillary sample is spanned with the rotation ( $\omega_c$ ) axis during measurements. Attach the capillary sample to the capillary holder and mount the holder to the capillary spin attachment. The prepared sizes of the holder are  $\varphi 1.0$  mm,  $\varphi 0.7$  mm, etc. Use the appropriate size for the capillary to be used.



Capillary spin attachment and capillary holder

## Wafer sample plates and sample spacers

A wafer sample plate is used in measurements of wafer-shaped samples. A sample spacer is placed on the attachment. 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 and sample spacer

**CAUTION** 



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

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



Magnets for securing sample in place



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

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 3 mm, you must select the appropriate sample spacer based on the thickness of the sample to be measured.

Table Sample spacers

| Measurable sample thickness                     | Abbreviation                          | Illustration |
|---|---------------------------------------|--------------|
| 0~3 mm<br>(option: 2680A406)                    | 0-3 mm                                |              |
| 3~6 mm<br>(option: 2680A405))                   | 3-6 mm                                |              |
| 6~9 mm<br>(option: 2680A404)                    | 6-9 mm                                |              |
| 9~12 mm<br>(option: 2680A403)                   | 9-12 mm                               |              |
| 12~15 mm<br>(option: 2640A402)<br>0~3 mm(*1)    | 12-15 mm<br>0-3 mm(blue stamped mark) |              |
| 15 mm~18 mm<br>(option: 2680A401)<br>3~6 mm(*1) | 15-18 mm<br>3-6 mm(blue stamped mark) |              |

<sup>\*1:</sup> Sample thickness for using XY-4"  $\phi$  attachment. Refer to the abbreviations in blue.

Table Wafer sample plates

| Name  | Illustration |
|---|--------------|
| 4-inch wafer sample plate (option: 2680A301)                      |              |
| 6-inch wafer sample plate (option: 2680A302)                      |              |
| 8-inch wafer sample plate (option: 2680A303)                      |              |
| 4-inch wafer sample plate<br>(for 18-21 mm)<br>(option: 2680A301) |              |
| 4-inch XY mapping sample plate (provided with XY-4"φ attachment)  |              |
| 4-inch XY mapping sample plate<br>(for 6-9 mm)<br>(option)        |              |

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

| Attachment  | Sample spacer   | Sample plate  | Measurable sample thickness                                   |
|---|---|---|---|
| Standard attachment<br>RxRy attachment<br>XY-20 mm attachment | 0-3 mm 3-6 mm 6-9 mm 9-12 mm 12-15 mm 15-18 mm (black stamped mark) | 4-inch wafer sample plate 6-inch wafer sample plate(*1) 8-inch wafer sample plate(*1) | 0-3 mm<br>3-6 mm<br>6-9 mm<br>9-12 mm<br>12-15 mm<br>15-18 mm |
|   | 4-inch wafer sample plate (for 18-21 mm)                            |   | 18-21 mm  |
|   | 4-inch wafer sample plate   |   |   |
|   | 6-inch wafer sample plate(*1)                                       |   | 21-24 mm  |
|   | 8-inch wafer sample plate(*1)                                       |   |   |
| XY-4" $\phi$ attachment                                       | 0-3 mm<br>3-6 mm<br>(blue stamped<br>mark)(*2)                      | 4-inch area mapping sample plate  | 0-3 mm<br>3-6 mm  |
|   | 4-inch XY mapping sample plate (for 6-9 mm)                         |   | 6-9 mm  |
|   | 4-inch XY   |   | 9-12 mm   |

<sup>\*1:</sup> Both X and Y must be 0 mm when the sample is placed on the XY-20 mm attachment.

<sup>\*2:</sup> When the XY-4"  $\phi$  attachment is used, the thickness indicated by the blue stamp mark on the sample spacer is valid.

#### Installing and removing sample spacers

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



Installing the sample spacer on the attachment

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



Removing the attachment

#### 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, 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



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

# Sample Holders

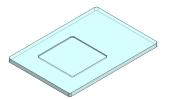
Use a sample holder when measuring samples that require preparation, such as powder and bulk samples.



For information on preparing samples, refer to Section 2.8 of the "Quick theta/2-theta measurement (BB)" or "Quick theta/2-theta measurement (PB/PSA)" Help Topic of the online help section of the *SmartLab Guidance software*.

## Glass sample holder

Use this sample holder to measure powder samples.



Glass sample holder

## Aluminum sample holder

Use this sample holder to measure powder samples or small bulk samples.



Aluminum sample holder

### Transmission SAXS sample holder (option: 2680J111)

Use this sample holder to measure fibrous or film samples by the transmission method.

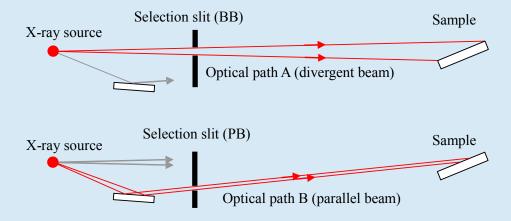


Transmission SAXS sample holder

#### Column

#### 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 B. In SmartLab, 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.

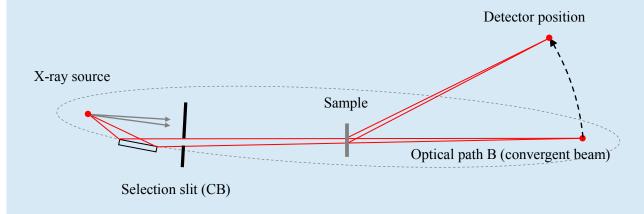


Diagram of the optics using CBO-E

# Accessories

Table Accessories

| Name  | Illustration               |
|---|----------------------------|
| Center slit   |                            |
| Si powder reference sample                              |                            |
| Si wafer reference sample                               |                            |
| Tool for installation of optical devices (Allen wrench) | Marian<br>Marian<br>Marian |

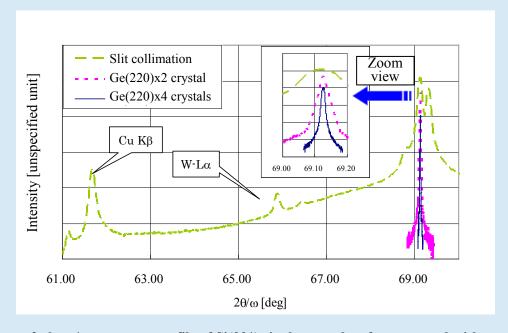
#### Column

#### **Incident monochromator crystal**

The incident monochromator crystal system uses diffraction by Ge or Si crystals for X-ray monochromatization. Ge(220)x2 crystal and Ge(440)x4 crystals are available as options for SmartLab. 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  $K\alpha_1$  X-rays. Shown below are the types and resolutions of monochromator crystals and corresponding applications.

Table Types and resolutions of incident monochromator crystals

| Type of crystal       | Resolution<br>(FWHM) | Applications   |
|-----------------------|----------------------|--|
| Ge(220)x2<br>crystal  | 0.0033°<             | For applications requiring monochromatized $K\alpha 1$ X-rays or for measurements of materials with diffraction planes with d spacings of ca. 2 Å. |
| Ge(400)x2<br>crystal  | 0.0022°<             | For measurements of materials with diffraction planes with d spacings of ca. 1.4 Å.  |
| Ge(220)x4<br>crystals | 0.0033°<             | For applications requiring high resolution over a broad 2-theta range.   |
| Ge(440)x4<br>crystals | 0.0015°<             | For applications requiring very high resolution over a broad 2-theta range.  |



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

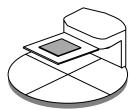
# **Examples of Sample Mounting**

Introduced below are some typical sample mounting methods.

For more information, refer to the *SmartLab Guidance Reference Manuals*, the Package Measurements Help Topics of the online help section of the *SmartLab Guidance software*.

### **Powder samples**

Place powder samples in the indentation on the glass sample holder or aluminum sample holder, then insert into the height reference sample plate.

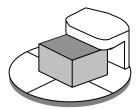


Powder sample

### **Bulk samples**

Use a 4-inchi wafer sample plate, 6-inch wafer sample plate, or 8-inch wafer sample plate to measure bulk samples. Select the appropriate sample spacer and sample plate based on sample thickness. For detailed information, refer to <u>Wafer sample plates and sample spacers</u>.

Place the sample so that the sample surface to be measured is parallel to the sample plate. A piece of clay may be used to secure the sample in place. If the bulk sample is small enough to pass through the window on the provided aluminum sample holder, you can secure the sample in the aluminum sample holder and use the height reference sample plate.



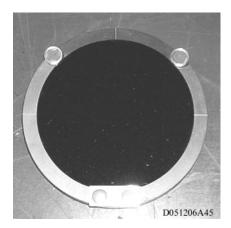
Bulk sample on height reference sample plate

### Sample for transmission SAXS measurements

For transmission SAXS measurements, clamp a fibrous or film sample to a sample holder and secure it in place, then insert the holder into a sample plate.

# Wafer-shaped samples

Use a 4-inch wafer sample plate, 6-inch wafer sample plate, or 8-inch wafer sample plate to measure wafer-shaped samples.



Wafer-shaped sample



When placing or removing the sample, we recommend removing the sample plate and sample spacer from the attachment. 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 magnets provided to hold the sample in place. The magnets can be used with samples that are up to 3 mm thick. For information on measuring samples thicker than 3 mm, see <u>Bulk samples</u>.

#### **CAUTION**



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 magnets securing the sample in place is small. Be careful while measuring of 8-inch wafers, because increasing the tilt axis (chi axis) can cause the wafer to bend under its own weight.

# **Measurements**

This section introduces basic operating procedures for typical measurements. For detailed information, refer to the *SmartLab Guidance Reference Manuals*, the Parts, and Package Measurements Help Topics of the online help section of the *SmartLab Guidance software*.

### **Preparing for measurements**

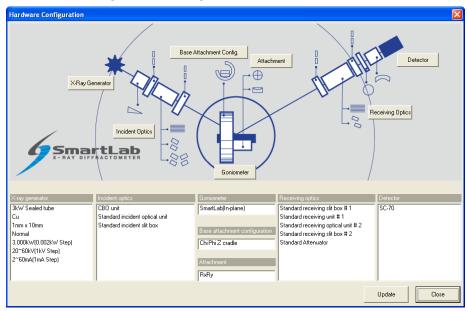
- **1** Make sure that the OPERATE lamp on the main panel is lighting up in green.
- **2** Launch SmartLab Guidance. Click Package Measurement under the Task menu.
- **3** Click the Startup button to turn on the X-ray.

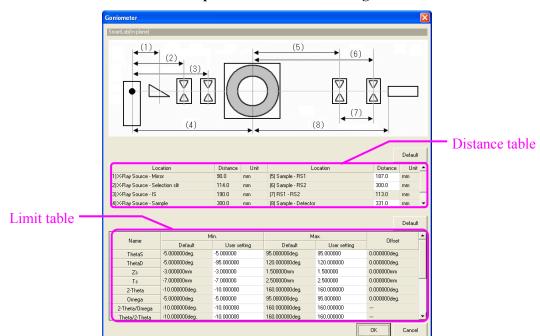


For information on the startup procedure, refer to Section 2.1 of the CBO Maintenance Package Measurement Help Topic of the online help section of the *SmartLab Guidance software*.

## Setting the hardware configuration

1 Click Hardware Configuration in the Options menu to open the Hardware Configuration dialog box.



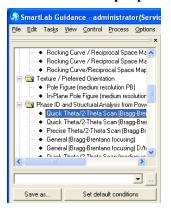


**2** Click Goniometer to open the Goniometer dialog box.

**3** Set the position of each optical device. To change the optical devices of the receiving optics, inspect the installation positions of the standard receiving slit box # 1, standard receiving slit box # 2, and detector adaptor and enter the correct corresponding values.

## **Selecting a Package Measurement**

- 1 Click Package Measurement under the Task menu.
- **2** Registered Package measurements are displayed in tree view. Select the Package Measurement that matches the purpose of the measurement.





For information on the procedures of each Package Measurements, refer to the Package Measurement Help Topic of the online help section of the *SmartLab Guidance software*.

# Periodic Maintenance

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

## **Optics maintenance**

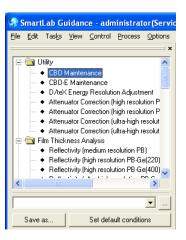
| Multilayer mirror     | You must perform re-adjustments following maintenance of the X-ray generator.                                 |
|-----------------------|---|
| Attenuator correction | You must perform re-adjustments when changing the wavelength or following maintenance of the X-ray generator. |
| HV/PHA adjustment     | You must perform re-adjustments when changing the wavelength to be measured.                                  |
| Dead-time correction  | You must perform dead-time correction measurements after HV/PHA adjustments.                                  |

The characteristics of the detector will change over time. Replacement of the detector is required when it reaches the end of its service life. However, you can minimize performance degradation by making the appropriate adjustments. We recommend performing HV/PHA adjustments and dead-time correction measurement at periodic intervals.

- HV/PHA adjustments
- Dead-time correction measurement

To make these adjustments automatically, select **Package Measurement** from the **Task** menu, then select **CBO Maintenance** Package measurement under **Utility**.

For detailed information on measurement procedures, refer to the "CBO Maintenance Package measurement" Help Topic of the online help section of the *SmartLab Guidance software*.



**Selecting Maintenance Package measurement** 

### **Cooling water**

Check the amount of cooling water when using the water circulation pump. 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.

Described below are the parts whose maintenance are required if using the rotating anode type X-ray tube.

### Target (2080A211/A411)

The target metal rotates at high speed to boost X-ray output. Pay close attention and note any abnormal noise or vibrations, and if these occur please contact the sales representative who you purchased the instrument from.

When contaminant (tungsten from the filament) from the electron gun (filament) adheres to the target metal surface, you may observe characteristic X-rays from the contamination, in addition to characteristic X-rays from the target metal. If this happens, maintenance is required. Please contact the sales representative who you purchased the instrument from.

## Filament (2080A211/A411)

As electrons are discharged, the filament will become thinner, eventually breaking the required circuit. Upon breaking, the filament will release a sudden burst of electrons that can damage the target surface. To avoid this, we recommend periodically changing the filament.

## Ion gauge (vacuum gauge) (2080A211/A411)

Since the filament in the ion gauge may come loose after many hours of use, we recommend changing the filament in the ion gauge at periodic intervals.

## Rotary pump (2080A211/A411)

Check the oil level in the vacuum pump. If the oil level is low, add oil. We also recommend periodic oil changes.

# **Troubleshooting**

| Problem  | Remedy  |  |  |  |  |
|--|---|--|--|--|--|
| The OPERATE lamp does not change color to green (remains red).   | The controller may be in the process of starting up. Wait several minutes.  If the lamp fails to turn green after several minutes, the controller may be malfunctioning. Press the OFF button on the main panel to shut down SmartLab, then press the ON button to restart SmartLab.  |  |  |  |  |
| An error occurs during SmartLab Guidance startup. SmartLab startup procedure fails to complete properly. | There may be a problem in the connection between the SmartLab and the controller. Confirm that the OPERATE lamp on the main panel is lighting up in green. Confirm the connection status of the RS232C connectors is OK.  After checking these items, shut down SmartLab Guidance, then restart it. Restart SmartLab Guidance after the SmartLab is restarted.  |  |  |  |  |
| SmartLab stops abruptly during measurement or other operation.   | Make sure the OPERATE lamp on the main panel has turned to red. SmartLab may have stopped due to excessive force applied to the theta_s or theta_d arm. Press the OFF button on the main panel to stop SmartLab. Check the arms to see if any item obstructs their operation, then restart SmartLab.  |  |  |  |  |
| The X-ray generator stops during measurement or other operations.  | Click XG Control in the Control menu to open the XG Control dialog box. Click Display in the Alarm section to open the Alarm dialog box. Check for X-ray generator alarms.  Refer to the XG status description box in the Alarm dialog box that will indicate the details of the alarm, which is brought when the pink alarm box in the Alarm dialog box is clicked.  Alarm  CWF  CWF  CWP  CWT  CWL  JAC  HYL  LVL  DUL  LCL  FCL  TRL  FCL  TRL  DOOR  LOCK  FS  XG status description:  CCWF>  The alarm box color turns from light blue to magenta in the event of the target and/or tube cooling water system failure. |  |  |  |  |
|  | The OPERATE lamp does not change color to green (remains red).  An error occurs during SmartLab Guidance startup. SmartLab startup procedure fails to complete properly.  SmartLab stops abruptly during measurement or other operation.  The X-ray generator stops during  |  |  |  |  |

<sup>\*</sup> 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.

### **Appendix** Using the OPERATE Lamp

The OPERATE lamp indicates the status of the SmartLab controller. The lamp is usually green, but will change to red and begin flashing at set intervals in the event of an abnormality. To reset the generated alarm, you must restart SmartLab. After shutting down SmartLab Guidance, press the "O" OFF button, then press the "]" ON button to restart SmartLab.

After SmartLab restarts, the OPERATE lamp will be flashing in green for 10 to 20 seconds. After that, when the lamp lights up in green, SmartLab is ready to use. If the OPERATE lamp does not turn green, flashing red instead after startup, or if the lamp turns green but changes to a flashing red immediately during use, please contact the sales representative who you purchased the instrument from. Before contacting, please refer to following tables to identify the alarm being generated.



Table Flashing pattern of the OPERATE lamp when SmartLab is starting up

| Condition                                    | Color | Continuously<br>On / flashing | Pattern          |  |  |
|--|-------|-------------------------------|------------------|--|--|
| Immediately<br>after Power ON                | Green | Flashing                      | 250 ms<br>250 ms |  |  |
| 10 seconds after<br>Power ON                 | Green | Flashing                      | 100 ms           |  |  |
| Able to be controlled from SmartLab Guidance | Green | Continuously<br>On            |                  |  |  |

Table Patterns of the OPERATE lamp flashing and alarm sounds when an alarm generated

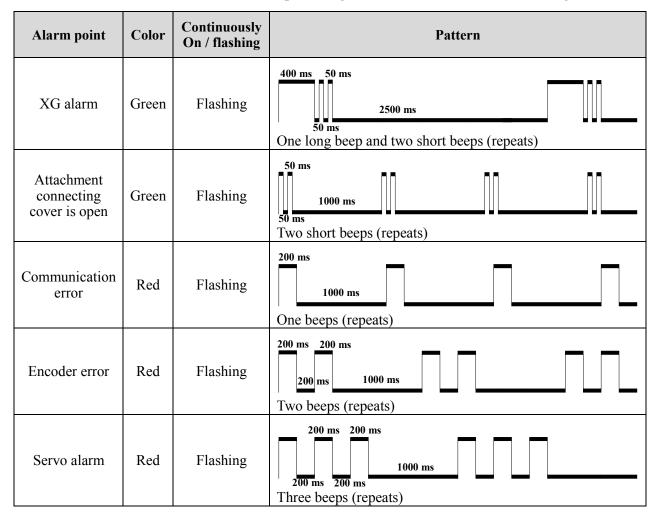


Table Causes of alarm

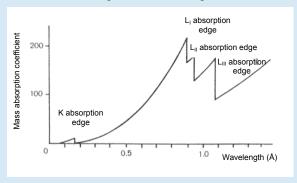
| Cause                               | Description   |  |  |  |  |
|-------------------------------------|---|--|--|--|--|
| XG alarm                            | An error has occurred in the X-ray generator (XG). See # 4 in Troubleshooting.  |  |  |  |  |
| Attachment connecting cover is open | The 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. |  |  |  |  |
| Encoder error                       | An error has occurred in the encoder used to control the goniometer. The encoder may not be adjusted correctly, or the detecting part may be stained.   |  |  |  |  |
| Servo alarm                         | The protection circuit is working as a result of motor electrical overload. See # 3 in <u>Troubleshooting</u> .   |  |  |  |  |

#### Column

#### Kß filter

The characteristic X-rays used for X-ray diffractometry generally contain  $K\alpha$  and  $K\beta$  X-rays. A substance that passes  $K\alpha$  X-rays while absorbing most  $K\beta$  X-rays to monochromatize the beam is called a  $K\beta$  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  $K\beta$  X-rays only, select an element as a  $K\beta$  filter with its absorption edge wavelength located between the  $K\alpha$  X-ray and  $K\beta$  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.

Table  $K\beta$  filters

|        | Wavelength (Å)  |                 | Metals used as a filter |   |  |                                     |                                   |
|--------|-----------------|-----------------|-------------------------|---|--|-------------------------------------|-----------------------------------|
| Target |                 |                 | Substa<br>-nces         | Wavelength<br>of<br>absorption<br>edge<br>(Å) | <b>When</b> $I_{K\beta_1}/I_{K\alpha_1} = 1/100$ |                                     |                                   |
|        | Kα <sub>1</sub> | Кβ <sub>1</sub> |                         |   | Thickness<br>(mm)                                | Mass<br>per unit<br>area<br>(g/cm²) | Κα <sub>1</sub><br>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                                |
| Mo     | 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                                |

#### LINE OF BUSINESS

#### X-ray Equipment for Science and Industry

X-ray Diffractometer and Attachments

X-ray Generators

X-ray Diffraction Cameras

X-ray Spectrometers

Piezo Goniometers

X-ray Stress Analyzers

Industrial X-ray Radiographic Equipment

Vacuum Rotary Feedthrough

#### Thermal Analysis Equipment

Differential Thermal Analyzers
Thermobalance Equipment
Thermomechanical Analyzers
Differential Scanning Calorimeters

Specifications Subject to Change without Notice

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