

Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

XRD – BB Instructions

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These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.

Before using the MCC, please review the MCC User Handbook available through our website.

Quick Theta/2-Theta Scan (Bragg-Brentano focusing) Package Measurement
Precise Theta/2-Theta Scan (Bragg-Brentano focusing) Package Measurement
General (Bragg-Brentano focusing) Package Measurement

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1. Package measurement flow

1.1 Quick Theta/2-Theta Scan (Bragg-Brentano focusing) Package measurement

Figure 1.1.1 shows the procedural flow for a Quick Theta/2-Theta Scan (Bragg-Brentano focusing) Package measurement.

i Tip: If the measurement is performed with the height reference sample plate, the measurement can begin immediately after completion of the optics alignment and sample positioning on the height reference sample plate, eliminating the need for sample alignment. Sample alignment must be performed if you use the wafer sample plate.

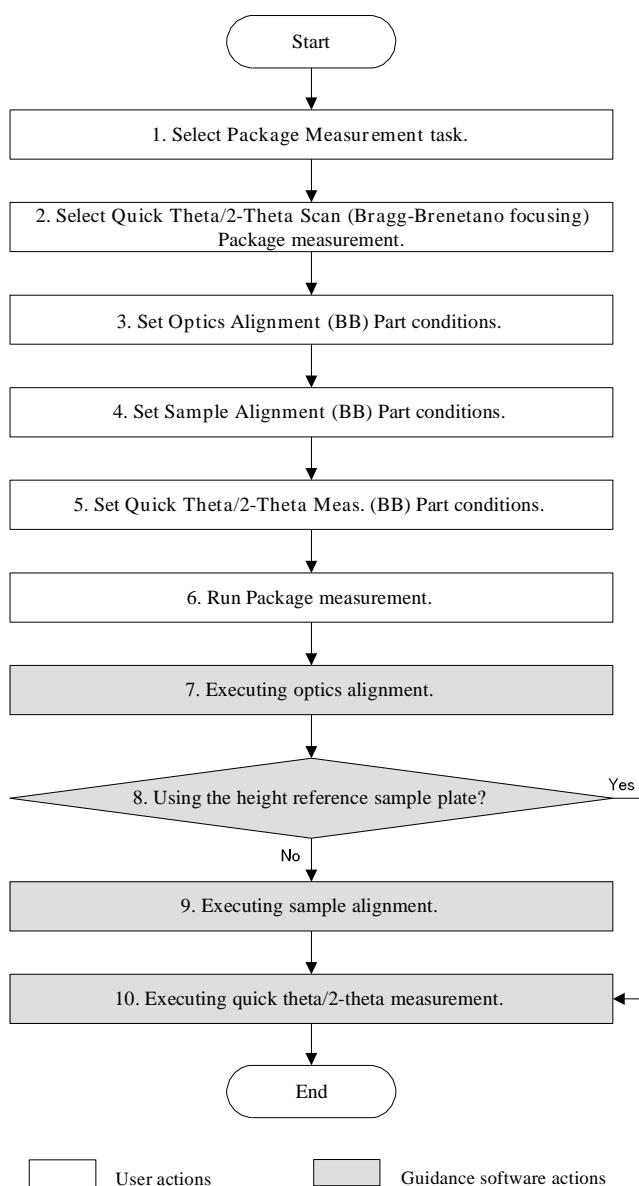
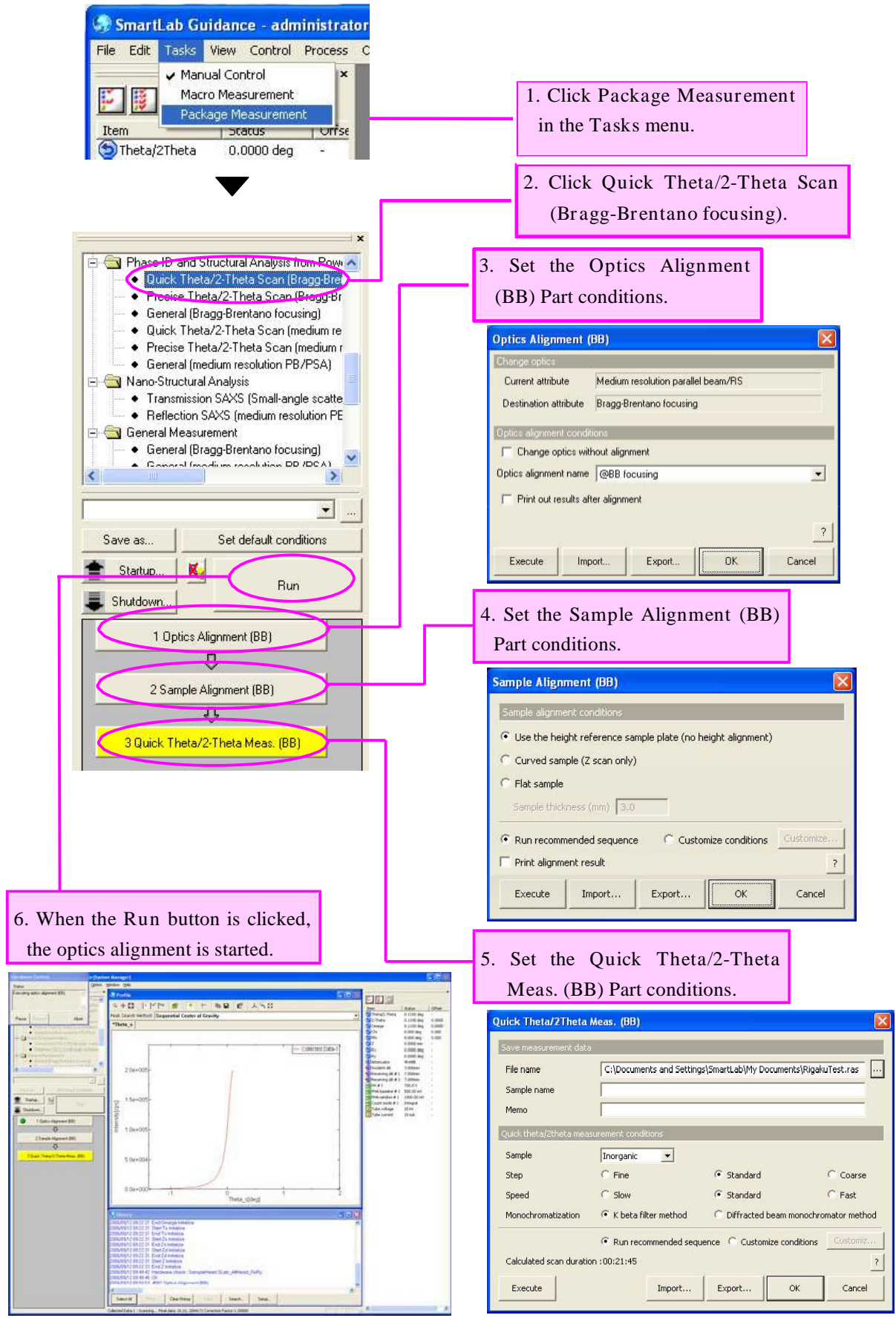


Fig. 1.1.1 Quick Theta/2-Theta scan (Bragg-Brentano focusing) Package measurement procedural flow

1. Package measurement flow



1.2 Precise Theta/2-Theta Scan (Bragg-Brentano focusing) Package measurement

Figure 1.2.1 shows the procedural flow for a Precise Theta/2-Theta Scan (Bragg-Brentano focusing) Package measurement.

i Tip: If the measurement is performed with the height reference sample plate, the measurement can begin immediately after completion of the optics alignment and sample positioning on the height reference sample plate, eliminating the need for sample alignment. Sample alignment must be performed if you use the wafer sample plate.

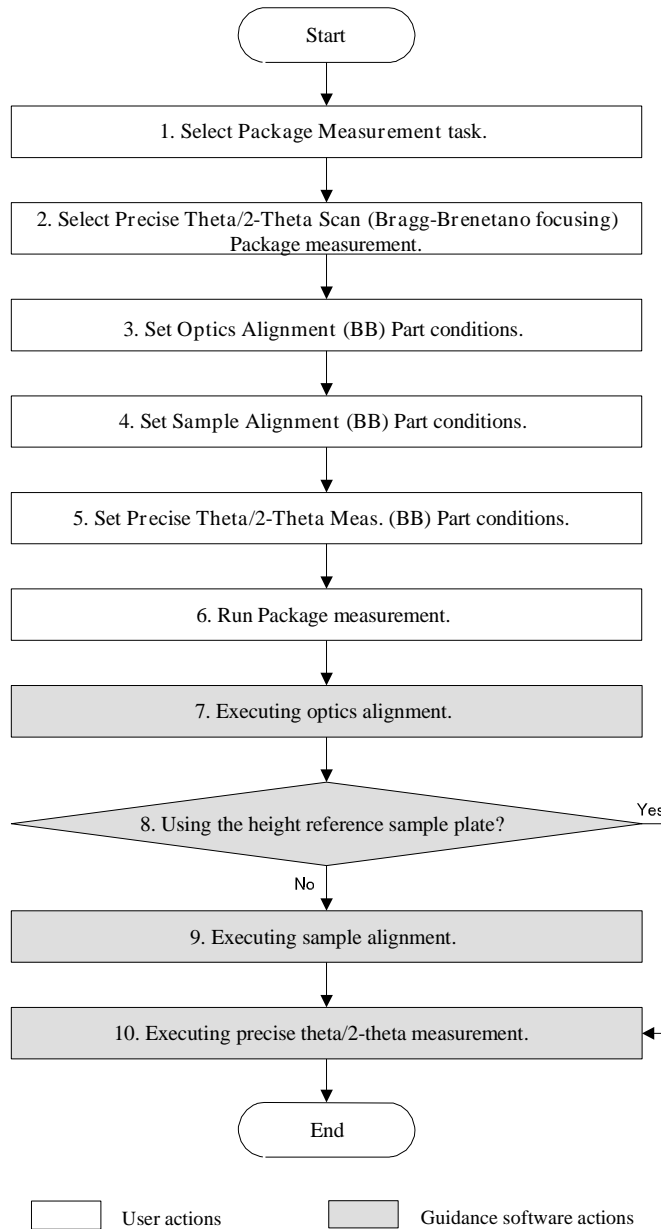


Fig. 1.2.1 Precise Theta/2-Theta Scan (Bragg-Brentano focusing) Package measurement procedural flow

1. Package measurement flow

1. Click Package Measurement in the Tasks menu.

2. Click Precise Theta/2-Theta Scan (Bragg-Brentano focusing).

3. Set the Optics Alignment (BB) Part conditions.

4. Set the Sample Alignment (BB) Part conditions.

5. Set the Precise Theta/2-Theta Meas. (BB) Part conditions.

6. When the Run button is clicked, the optics alignment is started.

1.3 General (Bragg-Brentano focusing) Package measurement

Figure 1.3.1 shows the procedural flow for a General (Bragg-Brentano focusing) Package measurement.

i Tip: If the measurement is performed with the height reference sample plate, the measurement can begin immediately after completion of the optics alignment and sample positioning on the height reference sample plate, eliminating the need for sample alignment. Sample alignment must be performed if you use the wafer sample plate.

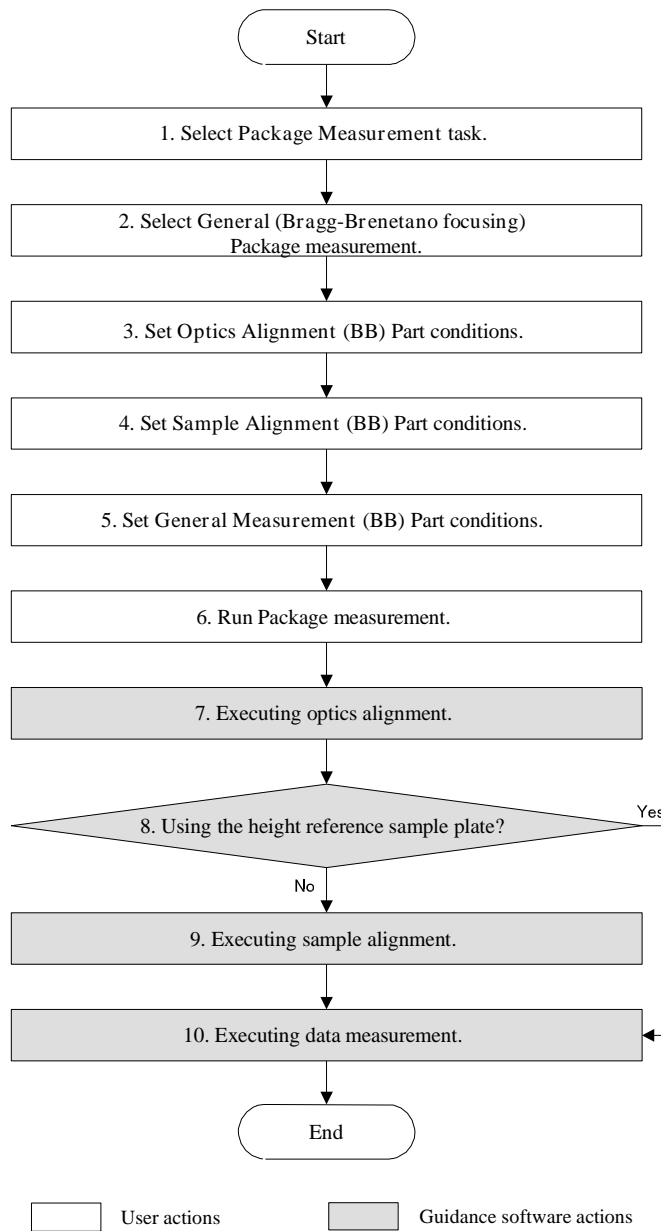
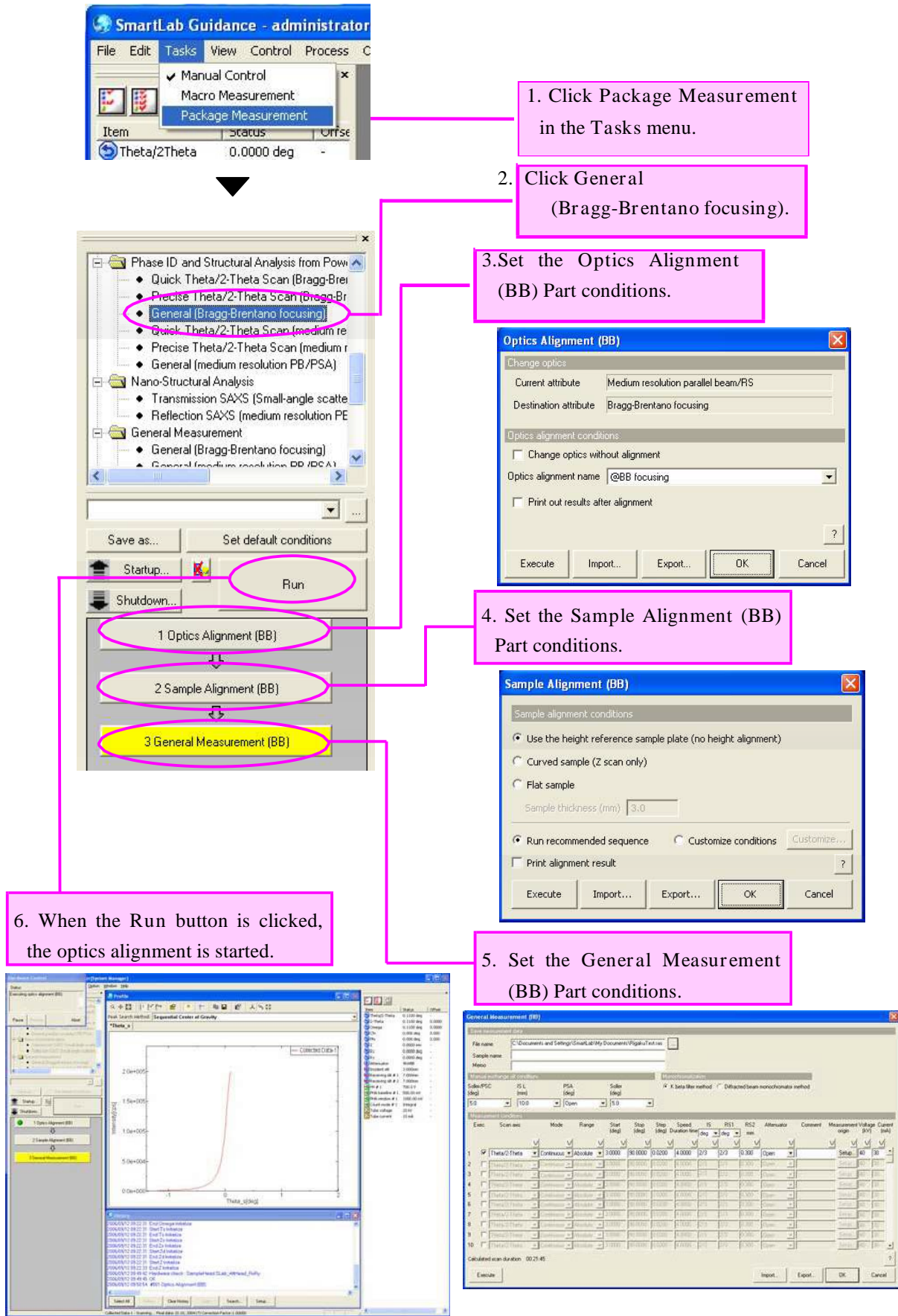


Fig. 1.3.1 General (Bragg-Brentano focusing) Package measurement procedural flow

1. Package measurement flow



2. Measurement procedures

1. Startup

Before measurement, set the output of the x-ray generator (XG) as described below.

- (1) Click the Startup button on the flow bar to open the Startup dialog box.



- (2) Uncheck the Timer box.



Fig. 2.1.1 Startup dialog box

- (3) Select an appropriate setting in the Generator usage box, based on the frequency of XG usage.
 (4) Select Set in the XG set box.
 (5) Enter the following values in the Voltage (kV) and Current (mA) boxes.

When using 3-kW sealed tube	40 kV, 30 mA
When using 9-kW rotating anode	45 kV, 200 mA

- (6) Click the Execute button.
 (7) The aging operation begins. The XG output will be set to the values entered in step (5) within 30 to 60 minutes.



CAUTION: If Not used for more than 3 weeks is selected in the Generator usage box, the aging operation will take approximately five hours.



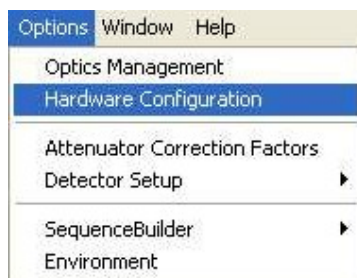
Tip: For other functions available from the Startup dialog box, refer to Chapter 21 of the SmartLab Guidance Reference Manual (ME13365A).

2. Hardware setup

Before measurement, the equipment configuration must be set up for a Quick Theta/2-Theta Scan (Bragg-Brentano focusing), Precise Theta/2-Theta Scan (Bragg-Brentano focusing), and General (Bragg-Brentano focusing) Package measurements.

The hardware setup method is described below.

- (1) Select the Hardware Configuration command under the Options menu to open the Hardware Configuration dialog box.



- (2) The Hardware Configuration dialog box shows the current hardware configuration.

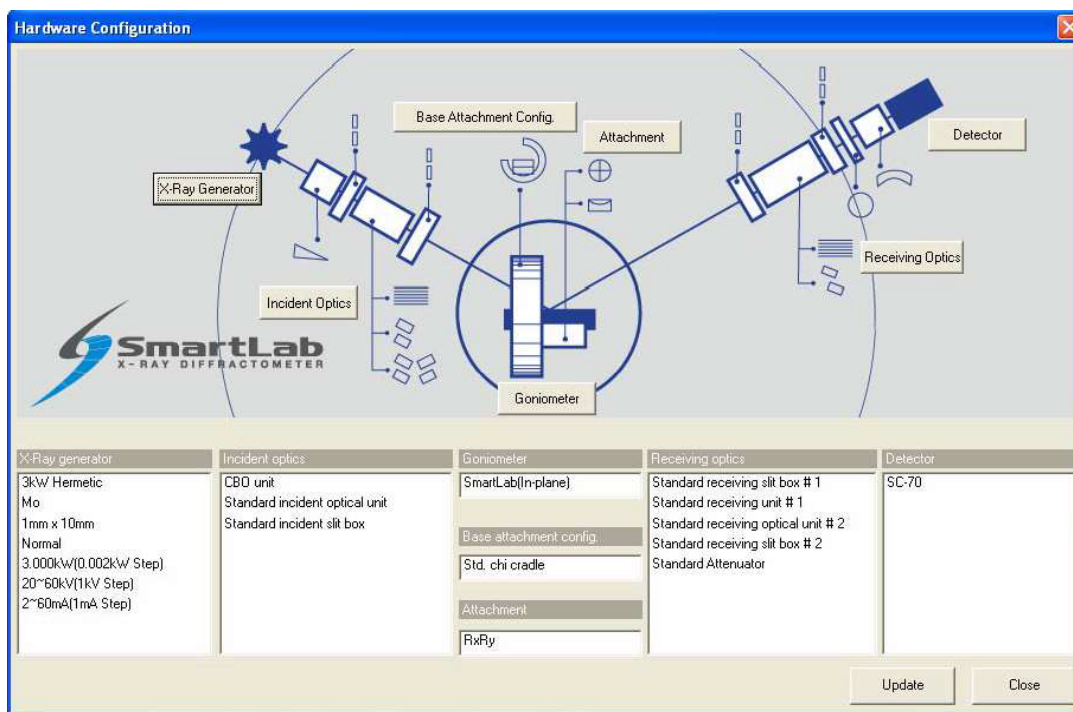


Fig. 2.2.1 Hardware Configuration dialog box

- (3) Table 2.2.1 shows the hardware configuration that enables the Quick Theta/2-Theta Scan (Bragg-Brentano focusing), Precise Theta/2-Theta Scan (Bragg-Brentano focusing), and General (Bragg-Brentano focusing) Package measurements. If the configuration units shown in the Hardware Configuration dialog box differ from those indicated in Table 2.2.1, install the units specified in Table 2.2.1 at the designated locations, referring to the Horizontal Sample Mount X-Ray Diffractometer for Thin Film Analysis Instruction Manual (ME11550A).

Table 2.2.1 Hardware configuration for the Quick Theta/2-Theta Scan (Bragg-Brentano focusing), Precise Theta/2-Theta Scan (Bragg-Brentano focusing), and General (Bragg-Brentano focusing) Package measurements

Hardware configuration	Configuration units
X-ray generator	Cu target
Incident optics	CBO unit Standard incident optical unit Standard incident slit box
Goniometer	SmartLab or SmartLab(in-plane)
Base attachment configuration	Standard chi cradle
Attachment	Any
Receiving optics	Standard receiving slit box # 1 Standard receiving optical unit # 1 Standard receiving optical unit # 2 Standard receiving slit box # 2 Standard attenuator
Detector	Scintillation counter SC-70

For example, if the target is set to Mo in the X-Ray Generator dialog box, you must change the target setting to Cu, since the Quick Theta/2-Theta Scan (Bragg-Brentano focusing), Precise Theta/2-Theta Scan (Bragg-Brentano focusing), and General (Bragg-Brentano focusing) Package measurements will not be made unless the Cu target is used. For sealed tubes, you must replace the Mo tube with the Cu tube.

- (4) Click the X-Ray Generator button to open the X-Ray Generator dialog box.

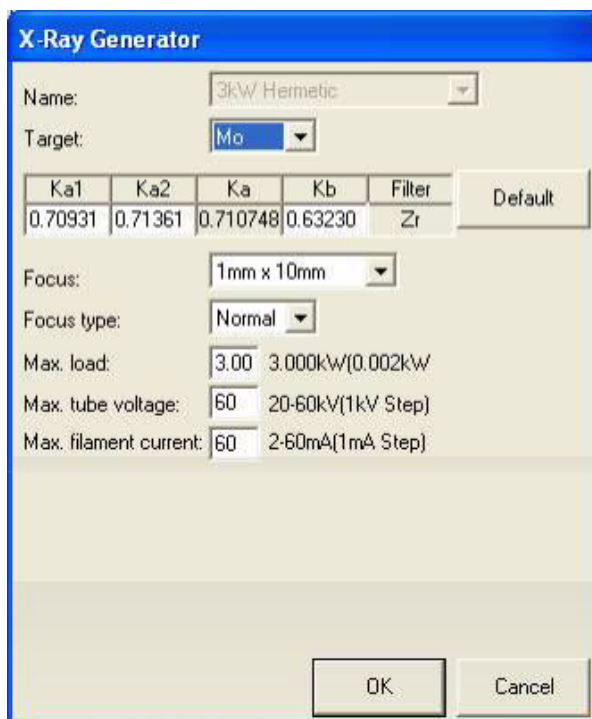


Fig. 2.2.2 X-Ray Generator dialog box

- (5) Select Cu in the Target box. Change other applicable parameters, such as wavelength.
- (6) Click the OK button to close the X-Ray Generator dialog box and register the changes made in the Hardware Configuration dialog box.
- (7) If necessary, change configuration units using the Incident Optics, Receiving Optics, and Detector buttons. Modify the conditions in the Hardware Configuration dialog box in the same way.
- (8) Click the Update button in the Hardware Configuration dialog box.



Tip: Mounted configuration units are automatically detected for Base Attachment Configuration and Attachment.

- (9) Confirm that each configuration unit displayed in the Hardware Configuration dialog box corresponds to the configuration unit indicated in Table 2.2.1, then click the Close button to close the dialog box.

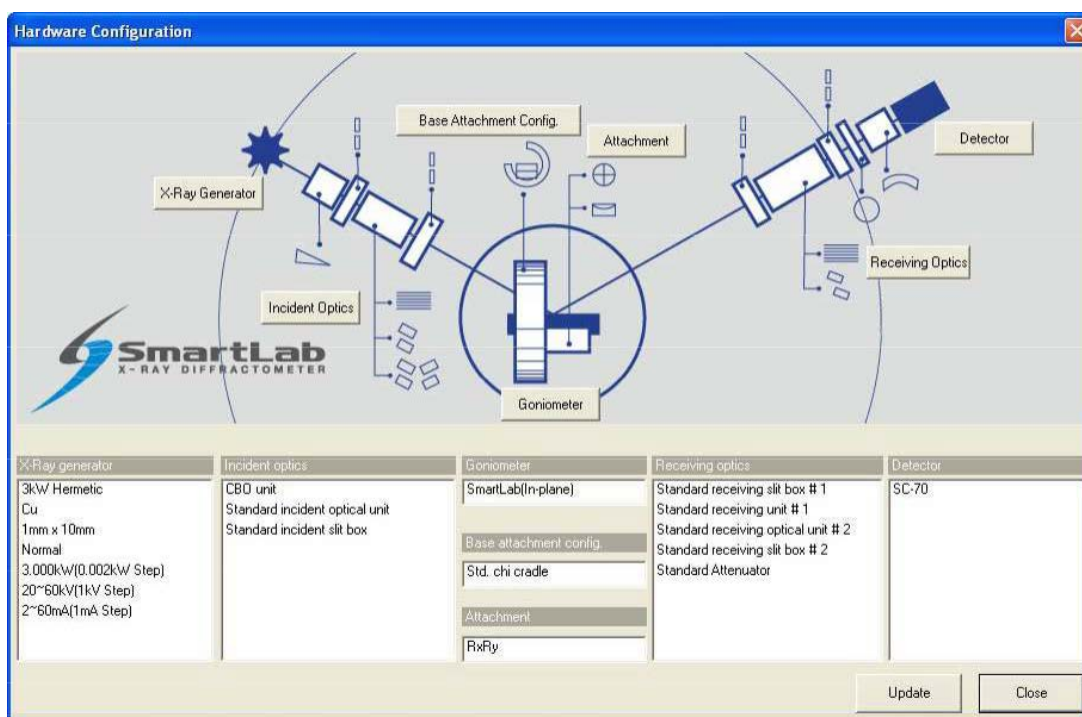


Fig. 2.2.3 Hardware Configuration dialog box

2.3 Setting Package measurement conditions

The Parts included in the Quick Theta/2-Theta Scan (Bragg-Brentano focusing), Precise Theta/2-Theta Scan (Bragg-Brentano focusing), and General (Bragg-Brentano focusing) Package measurements are shown in the table below.

Package measurement	Configuration Parts
Quick Theta/2-Theta Scan (Bragg-Brentano focusing)	<ul style="list-style-type: none"> • Optics Alignment (BB) • Sample Alignment (BB) • Quick Theta/2-Theta Meas. (BB)
Precise Theta/2-Theta Scan (Bragg-Brentano focusing)	<ul style="list-style-type: none"> • Optics Alignment (BB) • Sample Alignment (BB) • Precise Theta/2-Theta Meas. (BB)
General (Bragg-Brentano focusing)	<ul style="list-style-type: none"> • Optics Alignment (BB) • Sample Alignment (BB) • General measurement (BB)

To perform the Package measurements, you must set the conditions of each of the three Parts individually. Described below is how to set the conditions of each Part.



CAUTION: If another Package measurement is selected or another task such as the Manual Control task is chosen, discard the set Part conditions. To save the set conditions to a file, click the Export button in each dialog box or save Package measurement conditions as described in the “Tip” section in Subsection 2.3.3 (9).

2.3.1 Setting Optics Alignment (BB) Part conditions

- (1) Click the Optics Alignment (BB) button on the flow bar to open the Optics Alignment (BB) dialog box.

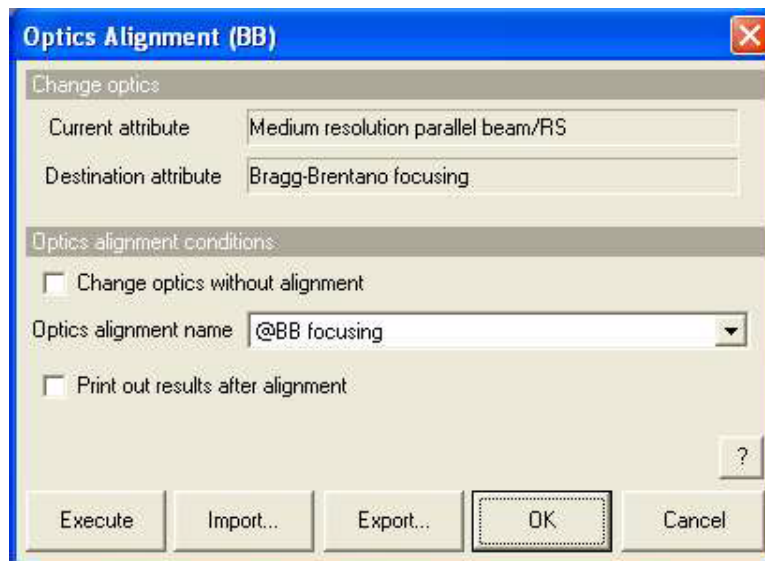




Fig. 2.3.1 Optics Alignment (BB) dialog box


- (2) Uncheck the Change optics without alignment box.

 Tip: Check the Change optics without alignment box to switch optics for the data measurement using the alignment results stored under the selected optics alignment name without performing optics alignment.

- (3) In the Optics alignment name box, select a location for storing optics alignment results.

 Tip: To store optics alignment results under a new optics alignment name, click the OK or Cancel button to close the Optics Alignment (BB) dialog box. Then, select the Optics Management command from the Options menu to open the Optics Management dialog box and add a new optics alignment name. After adding a new optics alignment name, return to step (1) in this subsection. For more information on creating an optics alignment name, refer to Chapter 17 of the SmartLab Guidance Reference Manual (ME13365A).

- (4) To print the optics alignment results, check the Print out results after alignment box.

 Tip: If both the Change optics without alignment and Print out results after alignment boxes are checked, the alignment results stored under the selected optics alignment name will be printed.

- (5) Click the OK button to close the dialog box.

2.3.2 Setting Sample Alignment (BB) Part conditions

- (1) Click the Sample Alignment (BB) button on the flow bar to open the Sample Alignment (BB) dialog box.

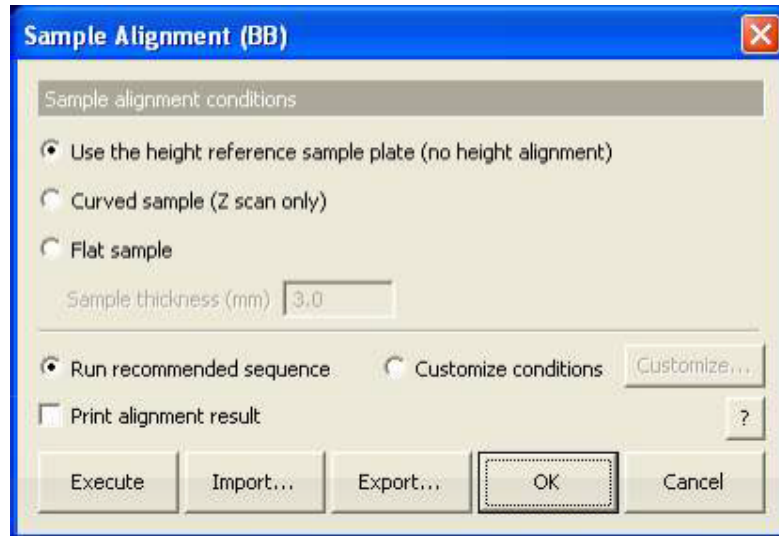




Fig. 2.3.2 Sample Alignment (BB) dialog box


- (2) To perform a data measurement using the height reference sample plate without performing sample alignment, select the Use the height reference sample plate (no height alignment) radio button.

 Tip: If one of the sample holders described in Section 2.8 is used or if the sample can be aligned visually to the reference position of the height reference sample plate, we recommend selecting the Use the height reference sample plate (no height alignment) radio button.


[2.8 Sample preparation](#)




- (3) To perform sample alignment, select the Curved sample (Z scan only) or Flat sample radio button and enter the thickness of the sample in the Sample thickness (mm) box.

 Tip: Use the wafer sample plate for sample alignment. Selecting the Curved sample (Z scan only) radio button sets the sample spacer and performs the vertical sample position (Z scan only) alignment.

- (4) To print the sample alignment results, check the Print alignment result box.

 Tip: If the Use the height reference sample plate (no height alignment) radio button is selected and the Print alignment result box is checked, only the alignment positions of the axes of the current optics will be printed.

- (5) Select the Run recommended sequence radio button.

 Tip: To confirm or set the scan conditions, select the Customize conditions radio button, then click the Customize button.

[2.4.1 Customizing Sample Alignment \(BB\) Part conditions](#)



- (6) Click the OK button to close the dialog box.

2.3.3 Setting Quick Theta/2-Theta Meas. (BB) Part conditions

- (1) Click the Quick Theta/2-Theta Meas. (BB) button on the flow bar to open the Quick Theta/2-Theta Meas. (BB) dialog box.

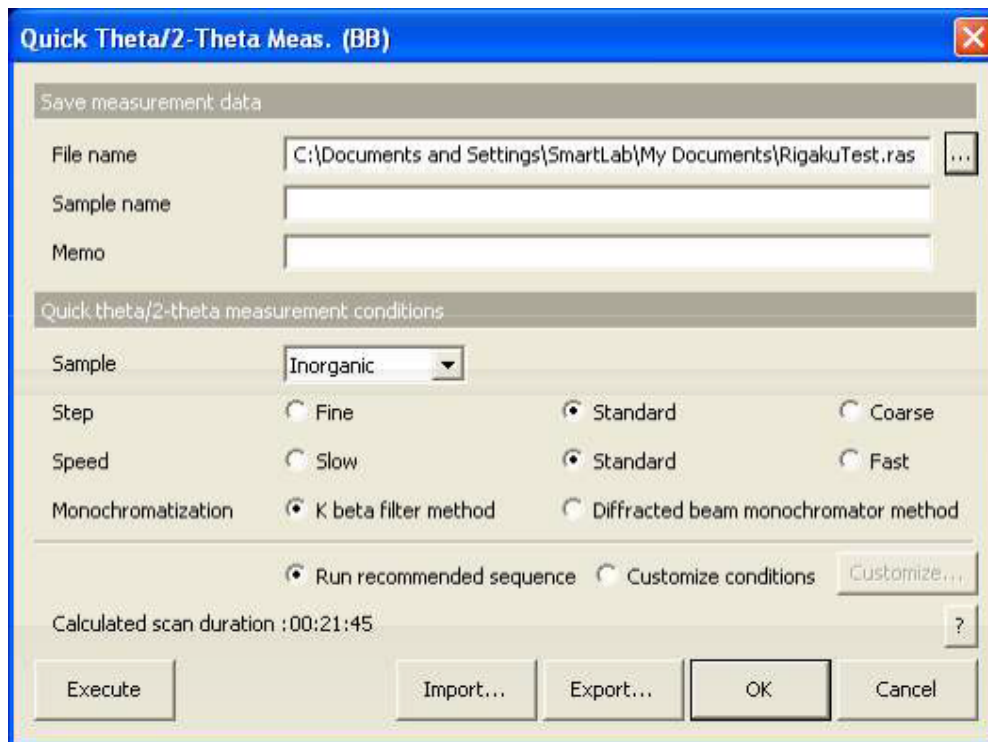


Fig. 2.3.3 Quick Theta/2-Theta Meas. (BB) dialog box

- (2) Set the folder to store the measurement data and the name of the measurement data file in the File name box in the Save measurement data section. After completing the quick theta/2-theta measurement, the measurement data will be saved with the specified file name.
 1. Click the [...] button to open the Save As dialog box.

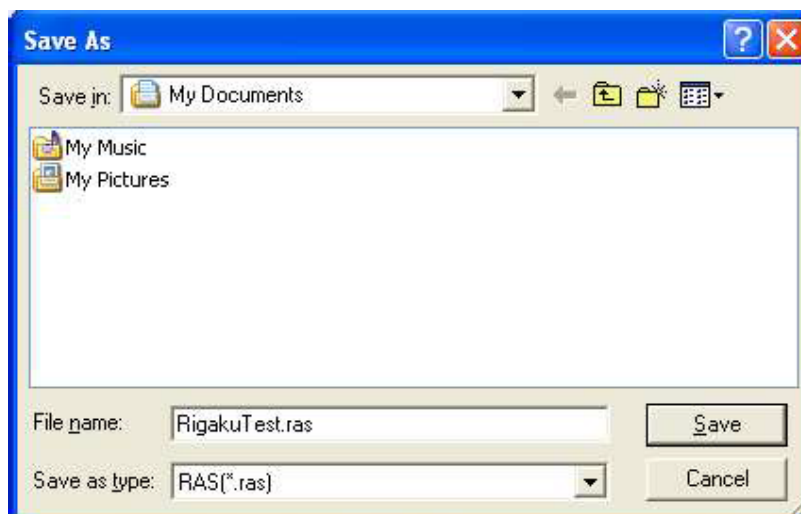


Fig. 2.3.4 Save As dialog box

2. Enter the settings in the Save in and File name boxes.
 3. Click the Save button.
- (3) Enter any required information in the Sample name and Memo boxes (optional).



CAUTION: The information entered here will be saved to the file. Any number of characters may be entered, but only the first 30 characters of the sample name and the first 84 characters of the memo will appear on the printed measurement data.

- (4) In the Sample box, select the type of sample to be measured from Inorganic, Organic, Mineral, or Metal. If the sample is a mixture of different material types or the material of the sample is unknown, select Unknown.
- (5) Select the Standard radio button for Step.



Tip: Selecting the Standard radio button sets the step width to 1/5 of the FWHM (0.1 °) of ordinary diffracted beams. If the step width is suspected to be narrower or wider than 1/5 the FWHM (0.1°) of ordinary diffracted beams, select Fine or Coarse.

- (6) Select the Standard radio button for Speed.



Tip: Selecting the Standard radio button will result in measurement duration of 15 to 30 minutes. The data thus obtained should cause no problems in the phase ID analysis of an ordinary sample. However, if you wish to improve the quality of the measurement data, select Slow. To shorten measurement duration, select Fast.

- (7) Select the K beta filter method for Monochromatization.



Tip: If the diffracted beam monochromator unit (option) is available, it is recommended to select the Diffracted beam monochromator method radio button. This setting removes almost all x-rays (K x-rays, continuous x-rays, fluorescent x-rays, etc.) other than those of the selected wavelength and provides measurement data with a low background level.

- (8) Select the Run recommended sequence radio button.




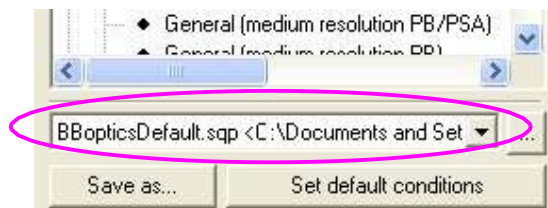
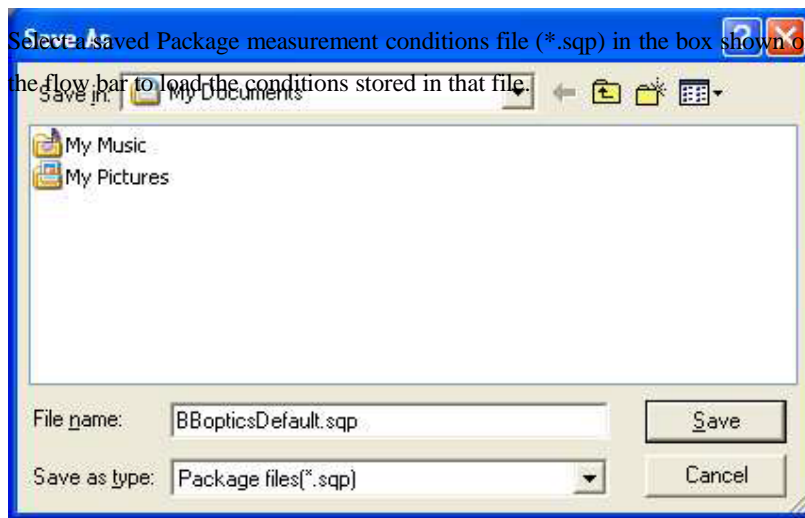
Tip: To confirm or set the scan conditions, select the Customize conditions radio button, then click the Customize button.

[2.4.2 Customizing Quick Theta/2-Theta Meas. \(BB\) Part conditions](#)



- (9) Click the OK button to close the dialog box.

 Tip: To save all the Part conditions set in Subsections 2.3.1 through 2.3.3 to a single file, click the Save as button on the flow bar to open the Save As dialog box, then enter the settings in the Save in and File name boxes. Click the Save button.



2.3.4 Setting Precise Theta/2-Theta Meas. (BB) Part conditions

- (1) Click the Precise Theta/2-Theta Meas. (BB) button on the flow bar to open the Quick Theta/2Theta Meas. (BB) dialog box.

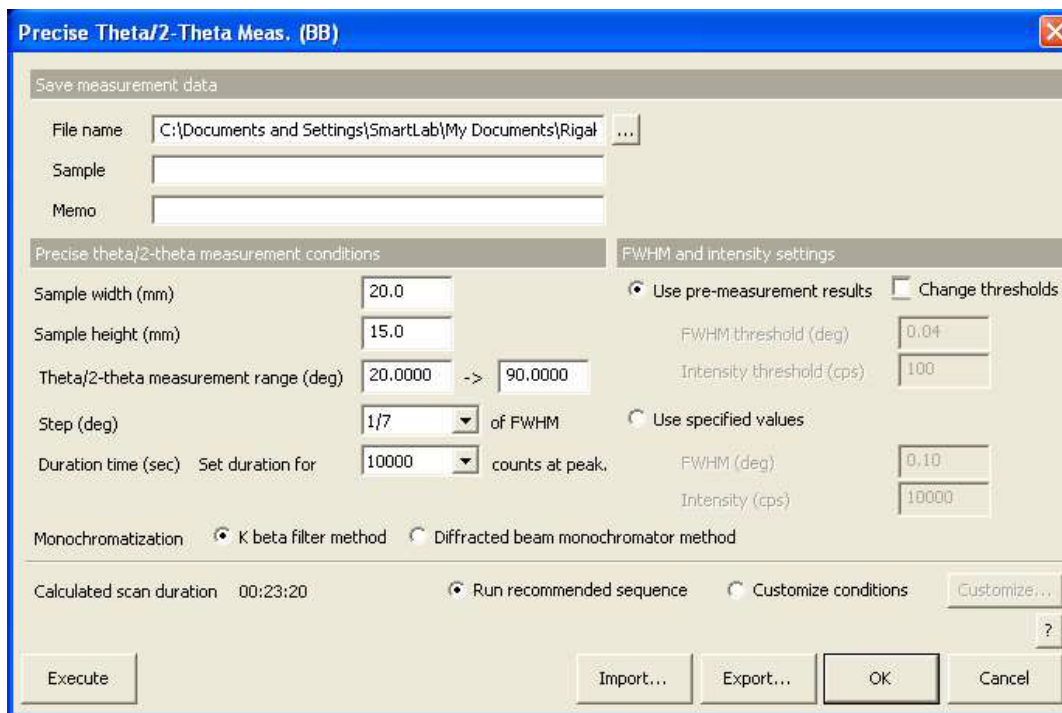


Fig. 2.3.5 Precise Theta/2-Theta Meas. (BB) dialog box

- (2) Set the folder to store the measurement data and the name of the measurement data file in the File name box in the Save measurement data section. After completing the precise theta/2-theta measurement, the measurement data will be saved with the specified file name.
 1. Click the [...] button to open the Save As dialog box.

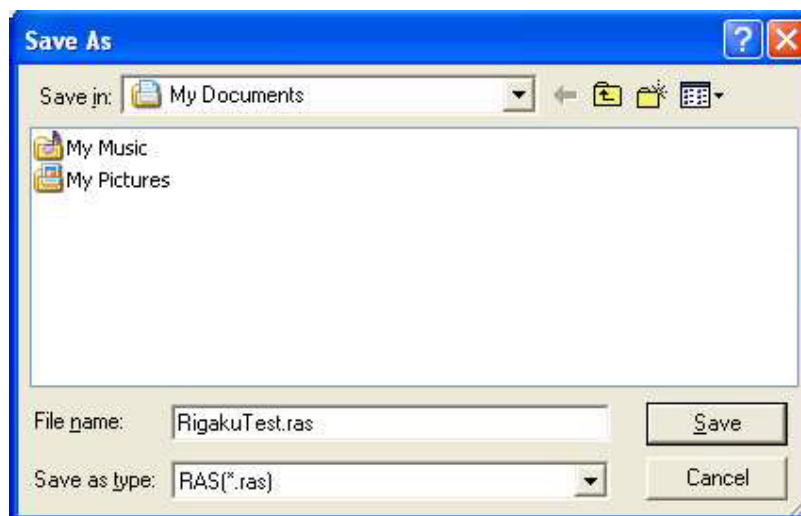


Fig. 2.3.6 Save As dialog box

2. Enter the settings in the Save in and File name boxes.
3. Click the Save button.
- (3) Enter any required information in the Sample name and Memo boxes (optional).

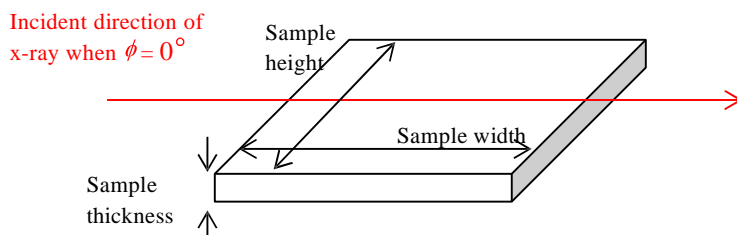


CAUTION: The information entered here will be saved to the file. Any number of characters may be entered, but only the first 30 characters of the sample name and the first 84 characters of the memo will appear on the printed measurement data.

- (4) Enter the sample width and sample height in the Sample width (mm) and Sample height (mm) boxes.



Tip: The sample width and sample height refer to the dimensions (unit: mm) of the sample in the directions shown below.



- (5) Enter the scan range in the Theta/2-Theta measurement boxes. Enter the start angle in the left box, and the stop angle in the right box.
- (6) Select 1/7 in the Step (deg) box.
- (7) Select 10000 in the Duration time (sec) box.



Tip: The duration time will be calculated so that the intensity of the highest peak becomes the selected value (unit: counts).

- (8) Select the K beta filter method radio button for Monochromatization.




Tip: If the diffracted beam monochromator unit (option) is available, it is recommended to select the Diffracted beam monochromator method radio button. This setting removes almost all x-rays (K x-rays, continuous x-rays, fluorescent x-rays, etc.) other than those of the selected wavelength and provides measurement data with a low background level.

- (9) Select the Use pre-measurement results radio button for FWHM and intensity settings.




Tip: To set the threshold of FWHM or intensity, or to set the FWHM and intensity when pre-measurement has been already made, refer to "Precise Theta/2-Theta Measurement (BB) Part" Help Topic of the online help section of the SmartLab Guidance software.

(10) Select the Run recommended sequence radio button.


 Tip: First, pre-measurement is made. Second, data measurement is made under the condition determined based on the pre-measurement results.

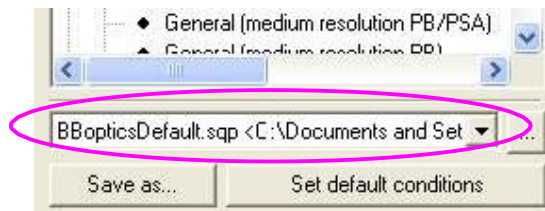
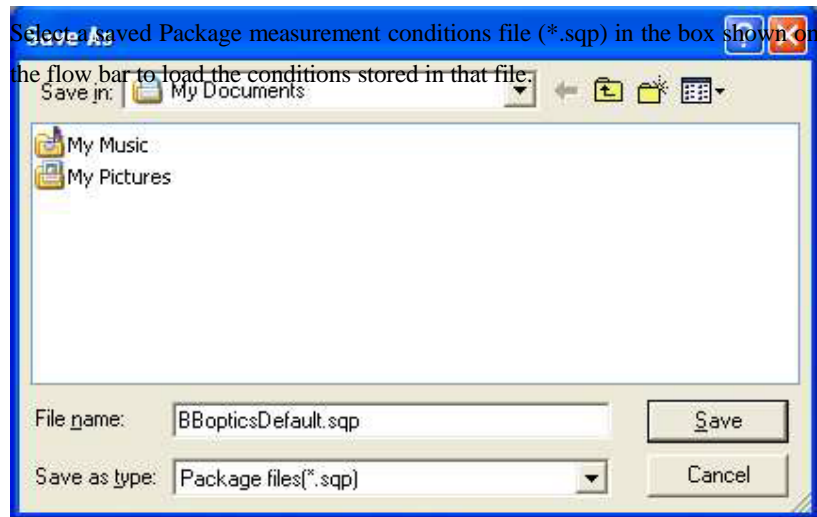
To confirm or set the scan conditions, select the Customize conditions radio button, then click the Customize button.

[2.4.3 Customizing Precise Theta/2-Theta Meas. \(BB\) Part conditions](#)



(11) Click the OK button to close the dialog box.

 Tip: To save all the Part conditions set in Subsections 2.3.1, 2.3.2, and 2.3.4 to a single file, click the Save as button on the flow bar to open the Save As dialog box, then enter the settings in the Save in and File name boxes. Click the Save button.



2.3.5 Setting General Measurement (BB) Part conditions

- (1) Click the General Measurement (BB) button on the flow bar to open the General Measurement (BB) dialog box.

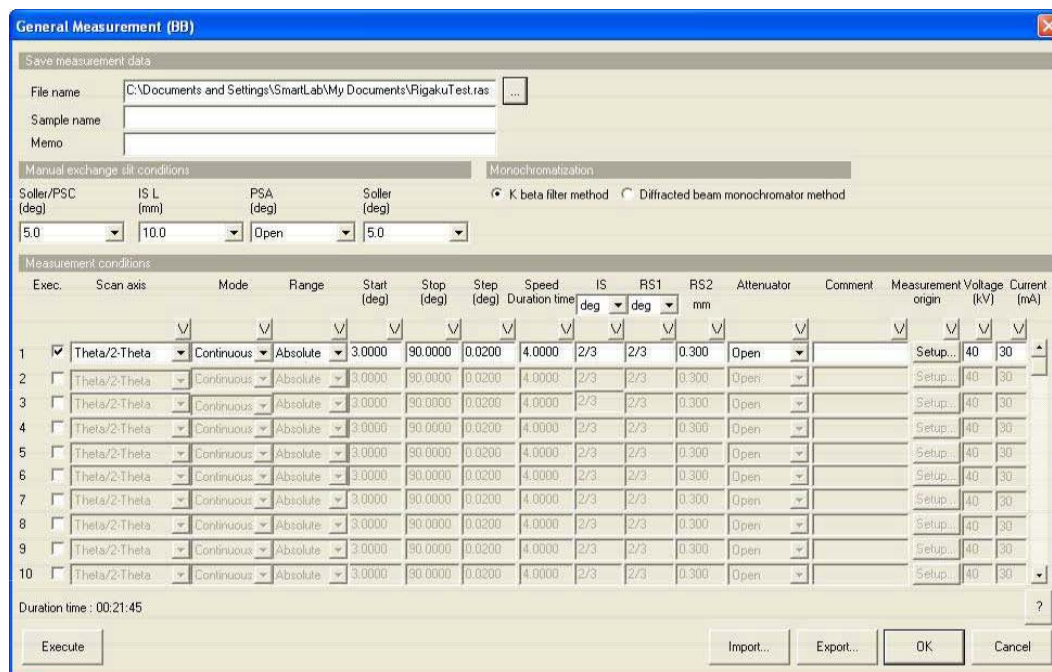


Fig. 2.3.7 General Measurement (BB) dialog box

- (2) Set the folder to store the measurement data and the name of the measurement data file in the File name box in the Save measurement data section. After completing the data measurement, the measurement data will be saved with the specified file name.
 1. Click the [...] button to open the Save As dialog box.

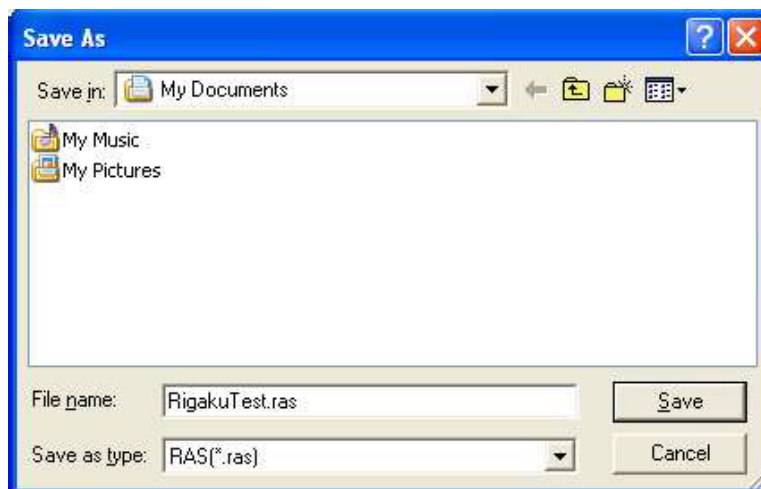


Fig. 2.3.8 Save As dialog box

2. Enter the settings in the Save in and File name boxes.
3. Click the Save button.

- (3) Enter any required information in the Sample name and Memo boxes (optional).



CAUTION: The information entered here will be saved to the file. Any number of characters may be entered, but only the first 30 characters of the sample name and the first 84 characters of the memo will appear on the printed measurement data.

- (4) Set the manual exchange slit conditions.

- (5) Set the measurement conditions.




Tip: To set the manual exchange slit conditions and measurement conditions, refer to “General Measurement (BB) Part” Help Topic of the online help section of the SmartLab Guidance software.

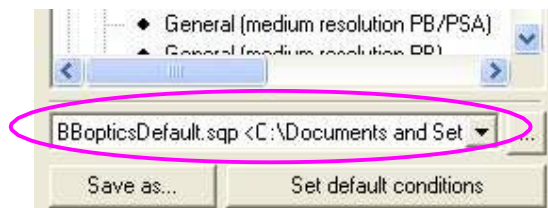
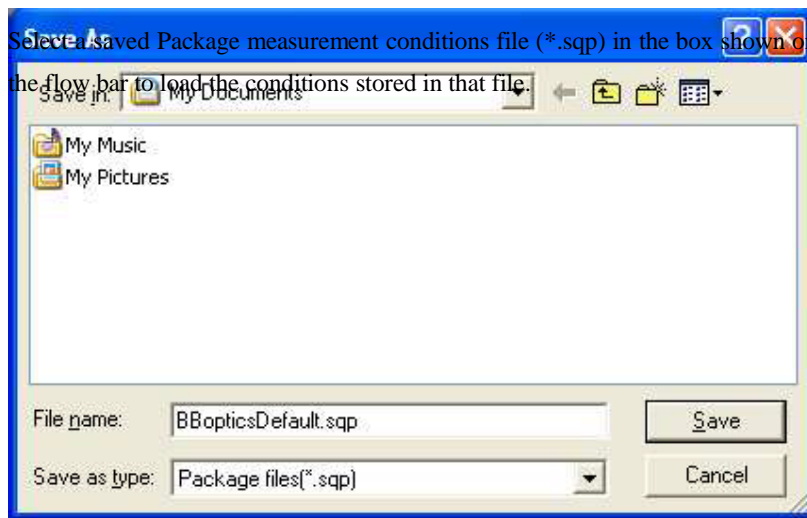
- (6) Select the K beta filter method radio button for Monochromatization.



Tip: If the diffracted beam monochromator unit (option) is available, it is recommended to select the Diffracted beam monochromator method radio button. This setting removes almost all x-rays (K x-rays, continuous x-rays, fluorescent x-rays, etc.) other than those of the selected wavelength and provides measurement data with a low background level.


- (7) Click the OK button to close the dialog box.

 Tip: To save all the Part conditions set in Subsections 2.3.1, 2.3.2, and 2.3.5 to a single file, click the Save as button on the flow bar to open the Save As dialog box, then enter the settings in the Save in and File name boxes. Click the Save button.



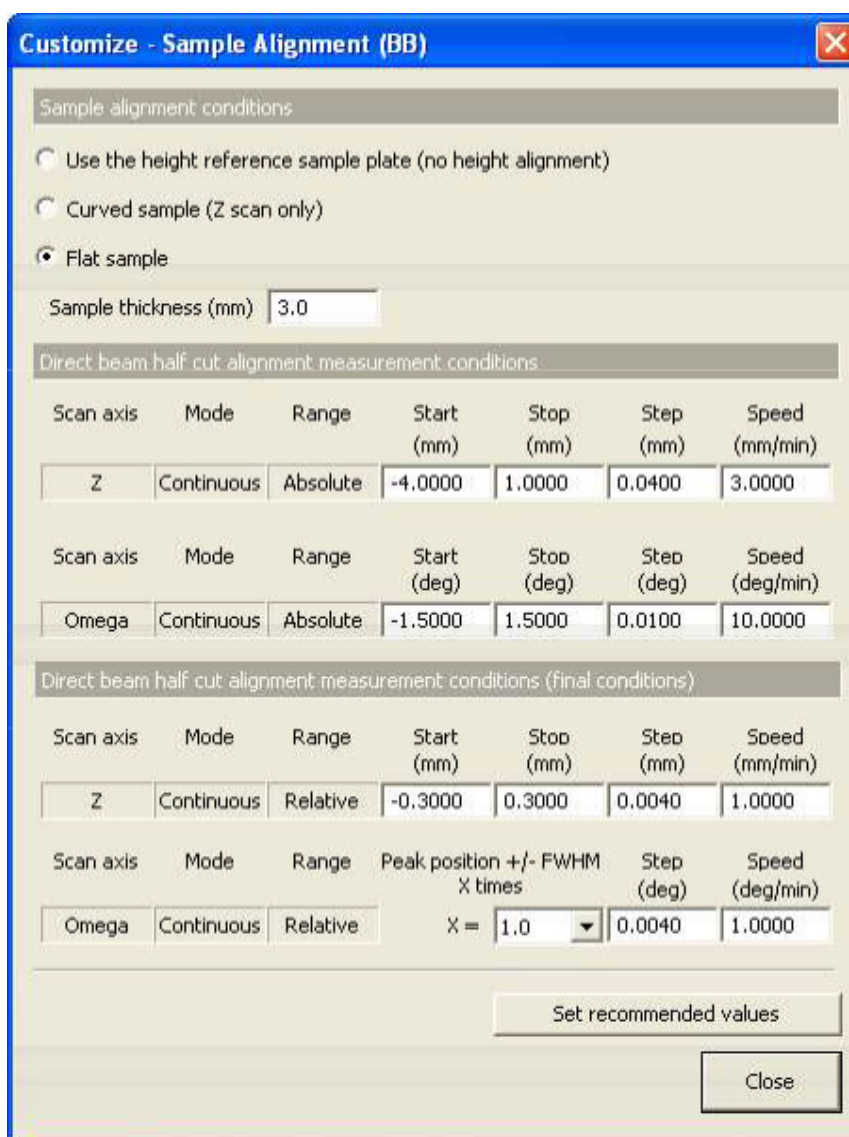
2.4 Customizing Part conditions

The user is free to set scan conditions and slit conditions of the Sample Alignment (BB), Precise Theta/2-Theta Meas. (BB), and Quick Theta/2-Theta Meas. (BB) Parts. Use the Customize dialog box to set scan conditions and slit conditions.

 Tip: To confirm the conditions set for Run recommended sequence, click the Set recommended values button in the Customize dialog box.

2.4.1 Customizing Sample Alignment (BB) Part conditions

- (1) Select the Customize conditions radio button in the Sample Alignment (BB) dialog box.
- (2) Click the Customize button to open the Customize dialog box.



Customize - Sample Alignment (BB)

Sample alignment conditions

Use the height reference sample plate (no height alignment)
 Curved sample (Z scan only)
 Flat sample

Sample thickness (mm)

Direct beam half cut alignment measurement conditions

Scan axis	Mode	Range	Start (mm)	Stop (mm)	Step (mm)	Speed (mm/min)
Z	Continuous	Absolute	-4.0000	1.0000	0.0400	3.0000

Scan axis	Mode	Range	Start (deg)	Stop (deg)	Step (deg)	Speed (deg/min)
Omega	Continuous	Absolute	-1.5000	1.5000	0.0100	10.0000

Direct beam half cut alignment measurement conditions (final conditions)

Scan axis	Mode	Range	Start (mm)	Stop (mm)	Step (mm)	Speed (mm/min)
Z	Continuous	Relative	-0.3000	0.3000	0.0040	1.0000

Scan axis	Mode	Range	Peak position +/- FWHM X times	Step (deg)	Speed (deg/min)
Omega	Continuous	Relative	X = 1.0	0.0040	1.0000

Fig. 2.4.1 Customize dialog box

The conditions set in the Sample Alignment (BB) dialog box are indicated in the Sample alignment conditions section.

- (3) If necessary, set conditions in the Direct beam half cut alignment measurement conditions and Direct beam half cut alignment measurement conditions (final conditions) sections.



CAUTION: Clicking the Set recommended values button changes settings in the Direct beam half cut alignment measurement conditions and Direct beam half cut alignment measurement conditions (final conditions) sections to the values recommended based on the value entered in the Sample thickness (mm) box. For more information, refer to “Sample Alignment (BB) Part” Help Topic of the online help section of the SmartLab Guidance software.

- (4) After setting the conditions, click the Close button to close the Customize dialog box.

2.4.2 Customizing Quick Theta/2-Theta Meas. (BB) Part conditions

- (1) Select the Customize conditions radio button in the Quick Theta/2-Theta Meas. (BB) dialog box.
- (2) Click the Customize button to open the Customize dialog box.

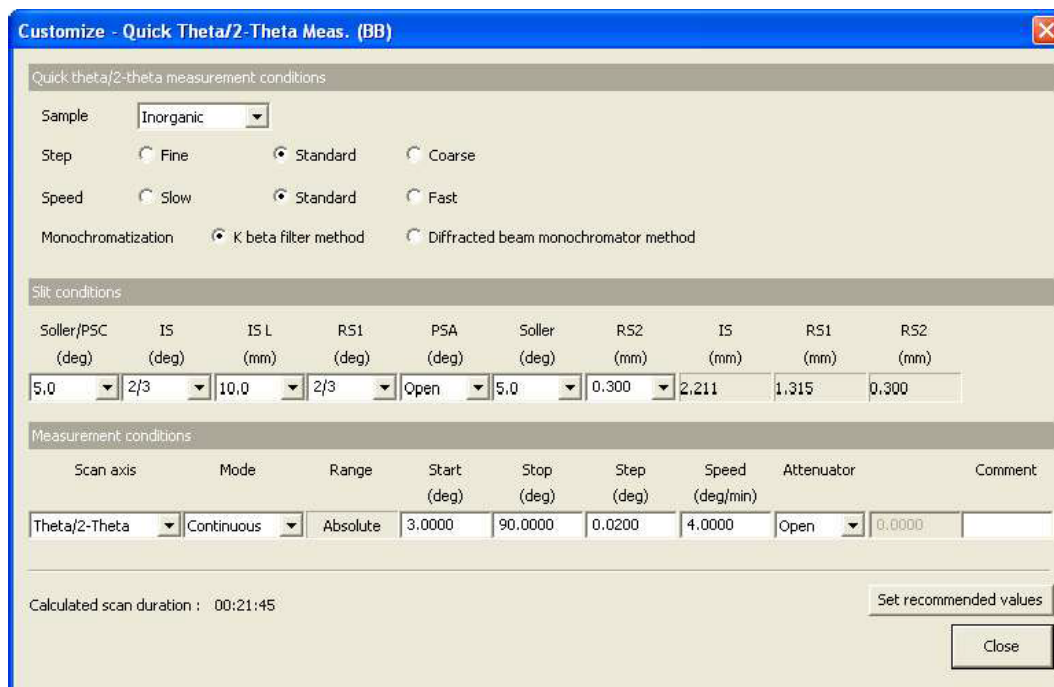


Fig. 2.4.2 Customize dialog box

The conditions set in the Quick Theta/2-Theta Meas. (BB) dialog box are indicated in the Quick theta/2-theta measurement conditions section.

- (3) If necessary, set conditions in the Slit conditions and Measurement conditions sections.



CAUTION: Clicking the Set recommended values button changes settings in the Slit conditions and Measurement conditions sections to the values recommended based on the values specified in the Quick theta/2-theta measurement conditions section. For more information, refer to “Quick Theta/2-Theta Measurement (BB) Part” Help Topic of the online help section of the SmartLab Guidance software.

- (4) After setting the conditions, click the Close button to close the Customize dialog box.

2.4.3 Customizing Precise Theta/2-Theta Meas. (BB) Part conditions

- (1) Select the Customize conditions radio button in the Precise Theta/2-Theta Meas. (BB) dialog box.
- (2) Click the Customize button to open the Customize dialog box.

Customize - Precise Theta/2-Theta Meas. (BB)

Precise theta/2-theta measurement conditions

Sample width (mm) 20.0

Sample height (mm) 15.0

Theta/2-theta measurement range (deg) 20.0000 -> 90.0000

Step (deg) 1/7 of FWHM

Duration time (sec) Set duration for 10000 counts at peak.

FWHM and intensity settings

Use pre-measurement results Change thresholds

FWHM threshold (deg) 0.04

Intensity threshold (cps) 100

Use specified values

FWHM (deg) 0.10

Intensity (cps) 10000

Monochromatization K beta filter method Diffracted beam monochromator method

Slit conditions

Soller/PSC (deg)	IS (deg)	ISL (mm)	RS1 (deg)	PSA (deg)	Soller (deg)	RS2 (mm)	IS (mm)	RS1 (mm)	RS2 (mm)
5.0	0.663	10.0	0.663	Open	5.0	0.3	2.199	1.308	0.300

Pre-measurement conditions

Scan axis	Mode	Range	Start (deg)	Stop (deg)	Step (deg)	Speed (deg/min)	Attenuator	Comment
Theta/2-Theta	Continuous	Absolute	20.0000	90.0000	0.0100	3.0000	Open	0.0000

Data measurement conditions

Scan axis	Mode	Range	Start (deg)	Stop (deg)	Step (deg)	Duration time (sec)	Attenuator	Comment
Theta/2-Theta	Step	Absolute	20.0000	90.0000	0.0140	1.0000	Open	0.0000

Calculated scan duration : 03:10:00

Set recommended values

Close

Fig. 2.4.3 Customize dialog box

The conditions set in the Precise Theta/2-Theta Meas. (BB) dialog box are indicated in the Precise theta/2-theta measurement conditions and FWHM and intensity settings sections.

- (3) If necessary, set conditions in the Slit conditions, Pre-measurement conditions, and Data measurement conditions sections.



CAUTION: Clicking the Set recommended values button changes settings in the Slit conditions and Pre-measurement conditions sections to the values recommended based on the values specified in the Precise theta/2-theta measurement conditions section. For more information, refer to “Precise Theta/2-Theta Measurement (BB) Part” Help Topic of the online help section of the SmartLab Guidance software.


When the Pre-measurement conditions box is checked, the data measurement conditions are determined from the pre-measurement results.



When the Pre-measurement conditions box is unchecked, pre-measurement is not made. Only data measurement is made under the conditions specified in the Data measurement conditions section.



- (4) After setting the conditions, click the Close button to close the Customize dialog box.


2.5 Executing a Package measurement

Described below is the procedure for executing the complete series of measurement operations from the optics alignment to the quick theta/2-theta measurement when the Quick Theta/2-Theta Scan (Bragg-Brentano focusing) Package measurement is selected. Measurement can be executed in the same way when the Precise Theta/2-Theta Scan (Bragg-Brentano focusing) or General (Bragg-Brentano focusing) Package measurement is selected.

 Tip: When executing optics alignment only, sample alignment only, quick theta/2-theta measurement only, precise theta/2-theta measurement only, or general measurement only, refer to [3. Executing a Part individually](#).

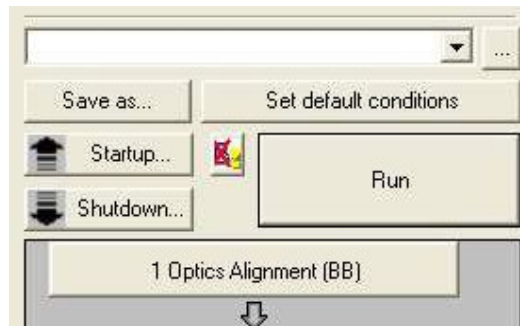
- (1) Click the  (Show confirmation messages) button to set it to  (Don't show confirmation messages).



 Tip: To confirm the optics alignment results or to show the message to verify that each Part has been completed, set it to  (Show confirmation messages).



The screenshot shows a control panel with several buttons: 'Save as...', 'Set default conditions', 'Startup...' (with an upward arrow icon), 'Shutdown...' (with a downward arrow icon), and a large 'Run' button. A small icon of the 'Show confirmation messages' button is highlighted in the 'Startup...' area.

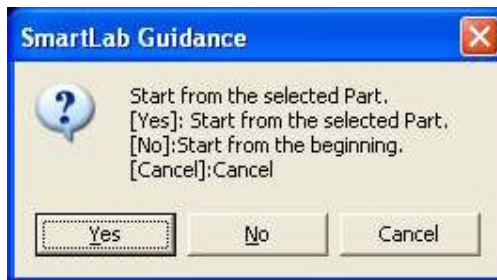
- (2) Click the Run button on the flow bar.




 Tip: After executing a Package measurement, the  mark appears on a button on the flow bar. The mark indicates that the Part corresponding to the button is in progress.




- (3) If the following message appears, click the No button.

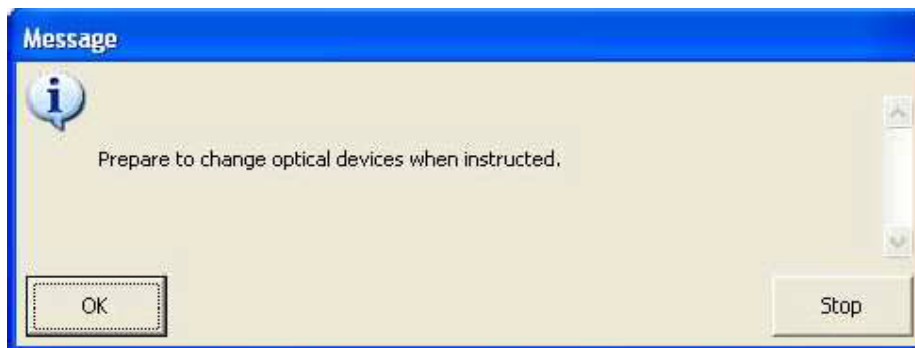


 CAUTION: If the current hardware configuration setting is different from that for the operation of the Quick Theta/2-Theta Scan (Bragg-Brentano focusing) Package measurement, the measurement will be aborted and the Hardware Configuration dialog box will open. If this happens, replace the configuration units (e.g., the attachment) as prompted by the message in the dialog box.

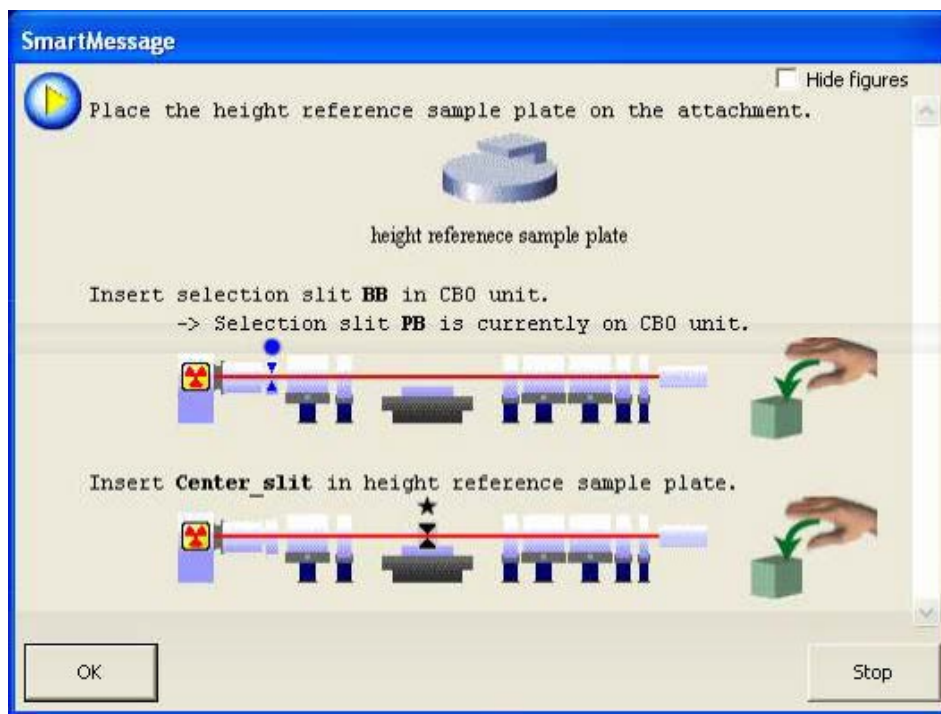
[2.6 Changing configuration units](#)




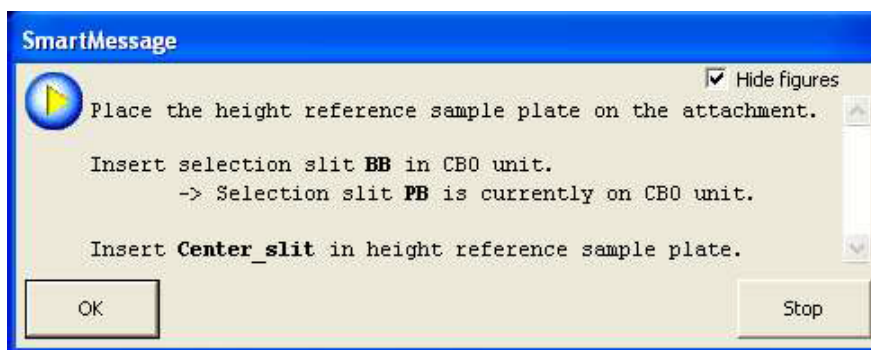
- (4) A message will prompt you to replace optical devices in the middle of the measurement. Confirm the message and click the OK button.





- (5) When the following message appears, place the height reference sample plate on the attachment and insert the center slit into the height reference sample plate. If another message appears at the same time prompting for replacement of the optical device(s) such as the selection slit, install the specified optical device(s) as indicated by the message.




 Tip: When the Hide figures box is checked, only the message is displayed without figures.



- (6) Click the OK button to execute optics alignment.
The optics alignment will be performed under the recommended conditions, and it will be completed in about 10 minutes.

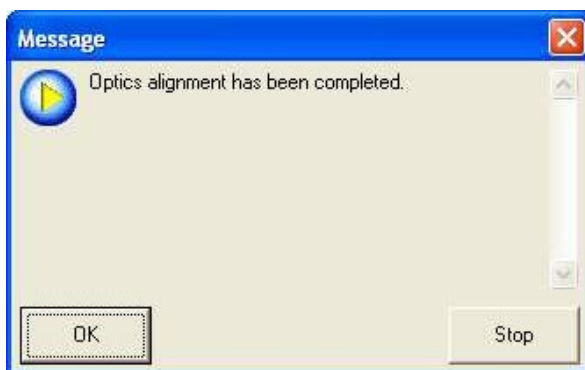
- (7) The specific procedure in this step depends on the setting  or  on the flow bar. Follow the directions given below.

When  (Don't show confirmation messages) is set:

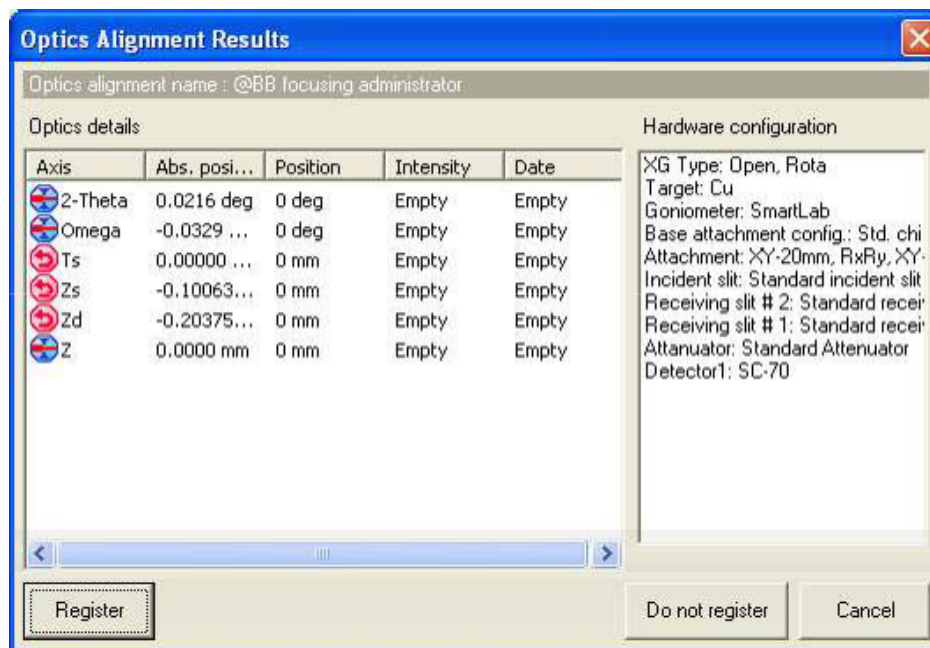
The optics alignment results will be registered in the optics management database.

When  (Show confirmation messages) is set:

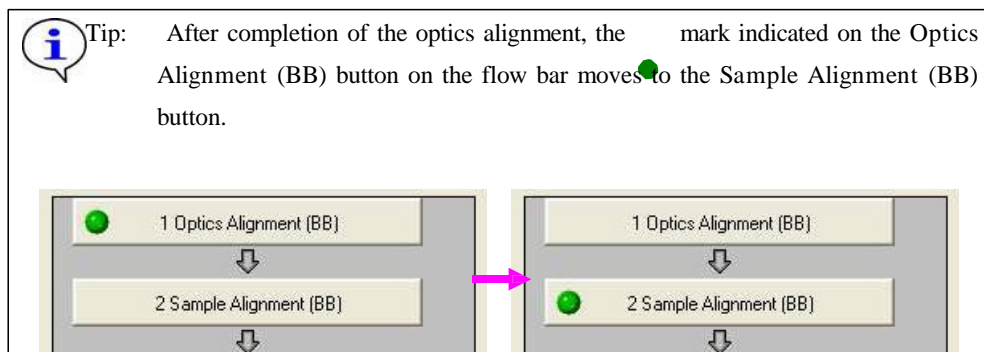
- The following message will appear to verify the optics alignment has been completed. Click the OK button.



- The Optics Alignment Results dialog box will appear. Confirm the results and click the Register button. The optics alignment results will be registered in the optics management database.



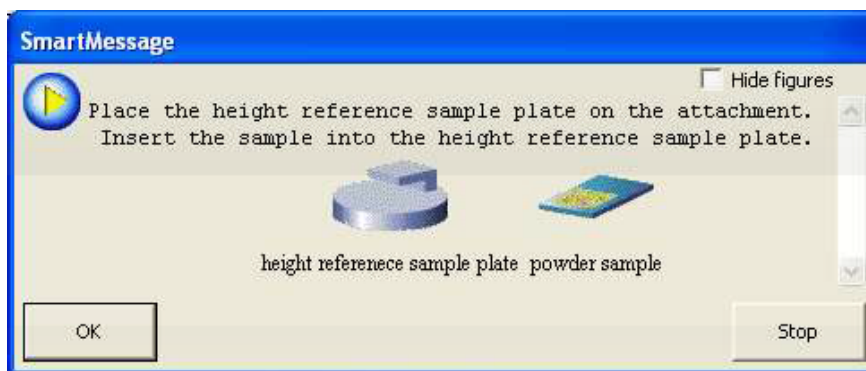
CAUTION: Clicking the Do not register button sets the zero offset value for each axis, but does not register the optics alignment results in the optics management database.



- (8) The specific procedure in this step depends on the item selected in the Sample alignment conditions section in the Sample Alignment (BB) dialog box. Follow the directions given below.

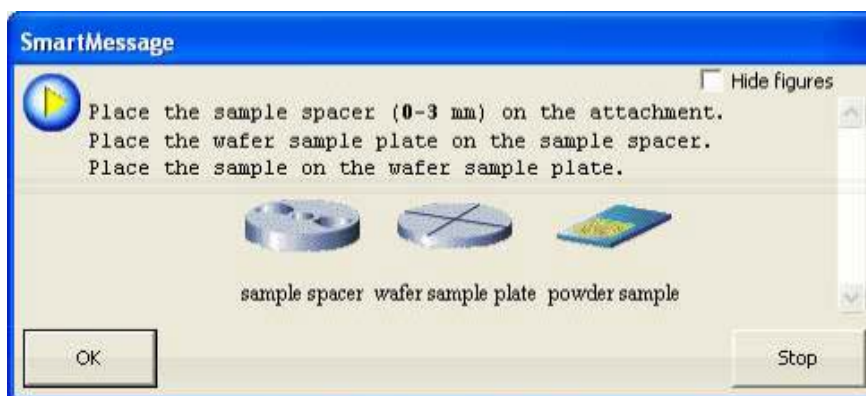
When Use height reference sample plate (no height alignment) has been selected:

The following message will appear. Based on this message, insert the sample into the height reference sample plate.




When Curved sample (Z scan only) or Flat sample has been selected:

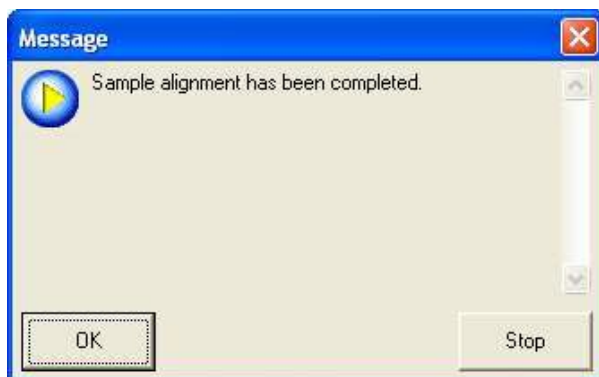
The following message will appear. Based on this message, place the sample spacer on the attachment, place the wafer sample plate on the sample spacer, and place the sample on the wafer sample plate.





- (9) Click the OK button to execute sample alignment.

When Curved sample (Z scan only) or Flat sample is selected in the Sample alignment conditions section in the Sample Alignment (BB) dialog box, direct beam half cut alignment will be performed under the specified conditions. The sample alignment will be completed in about 5 minutes.

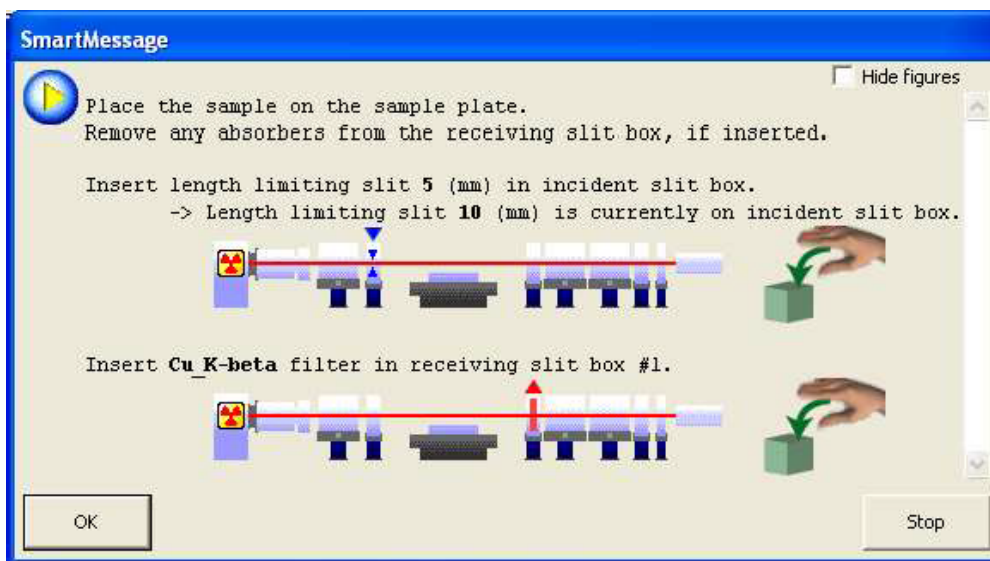
Only when  (Show confirmation messages) is set on the flow bar, the message will appear to verify the sample alignment has been completed. Click the OK button.



 Tip: After completion of the sample alignment, the  mark indicated on the Sample Alignment (BB) button on the flow bar moves to the Quick Theta/2-Theta Meas. (BB) button.




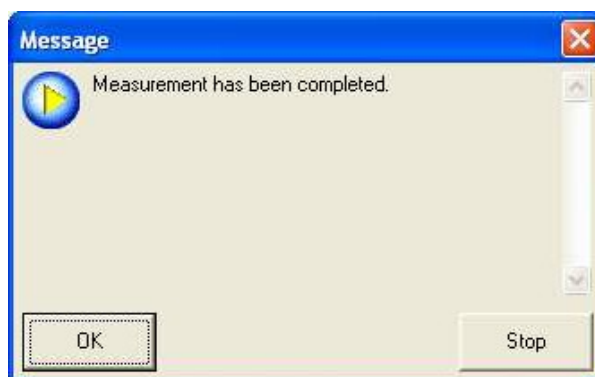
- (10) When K beta filter method has been selected for Monochromatization in the Quick Theta/2-Theta Meas. (BB) dialog box, the following message will appear. If instructed by this message, insert the $K\beta$ filter for Cu into the receiving slit box # 1. If another message appears at the same time to prompt for the replacement of the manual exchange slit such as the length limiting slit, install the specified manual exchange slit as prompted.



Tip: If Diffracted beam monochromator method has been selected in the Quick Theta/2-Theta Meas. (BB) dialog box as the monochromatization method, another message will appear at the same time. If instructed by the message, insert the diffracted beam monochromator unit and monochromator slit.

- (11) Click the OK button to execute a quick theta/2-theta measurement.
The measurement will be made under the specified conditions.
- (12) After completion of the measurement, the measurement data will be saved under the file name set in Subsection 2.3.3 (2).

Only when  (Show confirmation messages) is set on the flow bar, the message will appear to verify the quick theta/2-theta measurement has been completed. Click the OK button.



This is the end of the Quick Theta/2-Theta Scan (Bragg-Brentano focusing) Package measurement.

2.6 Changing configuration units

After a Package measurement (or Part) is executed, and the hardware configuration is not set for the Package measurement or the Part, the Hardware Configuration dialog box will open. A message appears below the dialog box prompting you to change the configuration units preventing the use of the Package measurement or the Part.

For example, if the target has been set to Mo in the X-Ray Generator dialog box, the displayed message will show “Target: Change Mo to Cu.”, since the target must be Cu for the Quick Theta/2-Theta Scan (Bragg-Brentano focusing), Precise Theta/2-Theta Scan (Bragg-Brentano focusing), and General (Bragg-Brentano focusing) Package measurements.



CAUTION: Execution of the Package measurement or Part aborts when the Hardware Configuration dialog box appears.

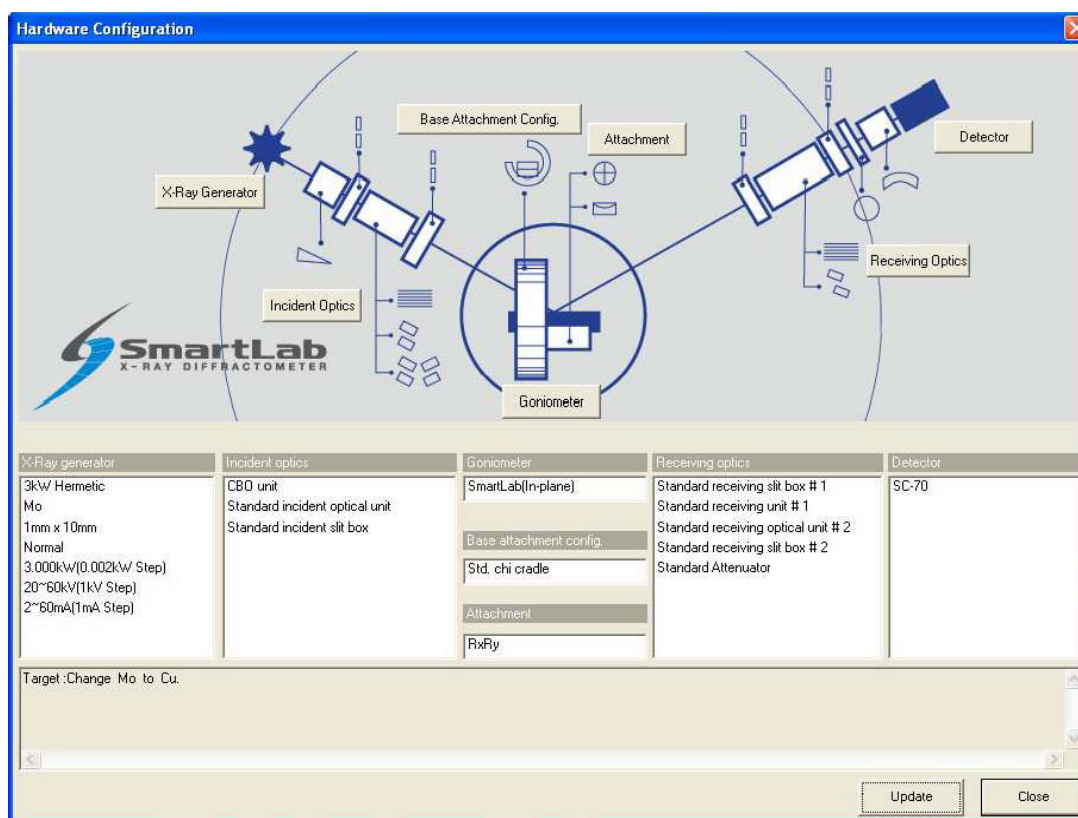


Fig. 2.6.1 Hardware Configuration dialog box

If the Hardware Configuration dialog box appears, replace the configuration units as prompted by the message in the dialog box and update the contents of the Hardware Configuration dialog box.

This procedure is described below.

- (1) Change the target in the x-ray generator from Mo to Cu. For sealed tubes, replace the Mo tube with the Cu tube.

- (2) Click the X-Ray Generator button to open the X-Ray Generator dialog box.

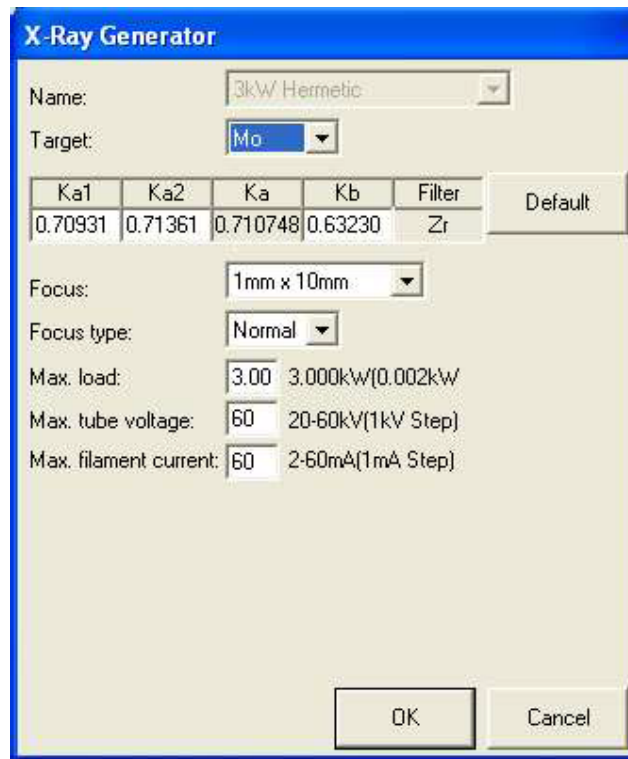


Fig. 2.6.2 X-Ray Generator dialog box

- (3) Select Cu in the Target box. Change any other parameters as necessary.
- (4) Click the OK button to close the dialog box.
- (5) If necessary, make changes for Incident Optics, Receiving Optics, and Detector in the same way.
- (6) Click the Update button in the Hardware Configuration dialog box.
- (7) Confirm that the Hardware Configuration dialog box displays the message “Hardware check OK”, then click the Close button to close the dialog box.



CAUTION: To execute the measurement once again, click the Run button on the Package Measurement flow bar or the Execute button in the applicable Part dialog box.

7. Shutdown

After completion of all measurements, turn off the x-ray generator as described below.

- (1) Click the Shutdown button on the flow bar to open the Shutdown dialog box.




- (2) Uncheck the Execute box.



Fig. 2.7.1 Shutdown dialog box

- (3) Select XG Off in the XG set box.
- (4) Click the Execute button.
- (5) The shutdown operation is executed. The x-ray generator will be turned off in about 10 minutes.

 Tip: For information on other functions available from the Shutdown dialog box, refer to Chapter 21 of the SmartLab Guidance Reference Manual (ME13365A).

2.8 Sample preparation

For measurements of a powder sample, either of the glass, back loading, or zero background sample holders may be used, based on the sample quantity and ease of sample placement.

For measurements of bulk samples, the back loading or block sample holders may be used based on sample shape, size, and weight.

All sample holders have a reference position for alignment of the sample surface. The reference position is more precisely finished than other holder surfaces. To ensure accurate measurement results, you must prepare the sample so that the sample surface aligns with the reference position. Diffraction angle deviations may occur if the sample is not loaded properly.

When using a sample holder loaded as indicated in Subsections 2.8.1 through 2.8.6, the sample surface is precisely aligned with the reference position on the height reference sample plate. In other cases, if it is possible to visually align the sample surface to the reference position on the height reference sample plate, we recommend using the height reference sample plate in place of the wafer sample plate.

When using the height reference sample plate, you can make measurements after optics alignment simply by mounting the sample, without performing sample alignment.

Figure 2.8.1 shows how to mount a sample holder on a sample plate.

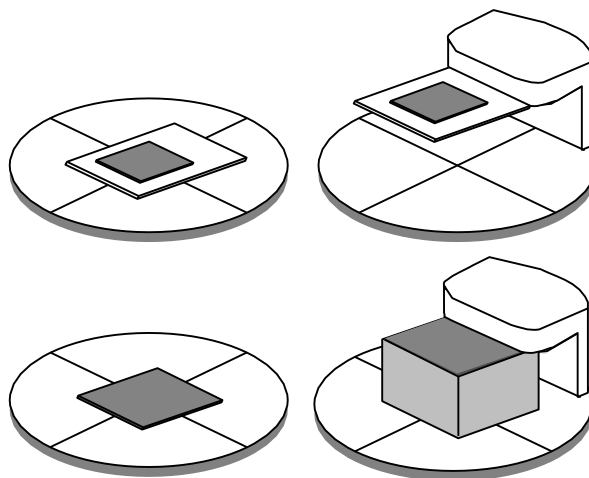
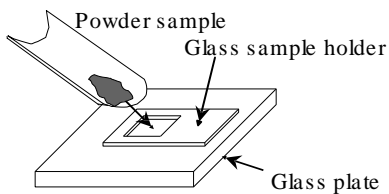


Fig. 2.8.1 Sample mounting examples

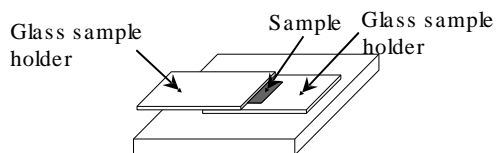
Described below is the method for preparing samples using various sample holders.

2.8.1 Glass sample holder (for adequate sample amounts)

- (1) Place the sample in the indentation on the glass sample holder.




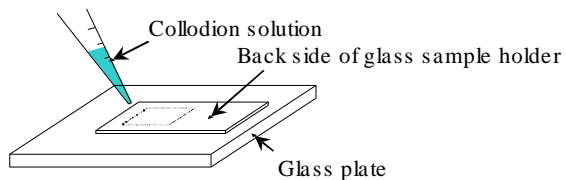
- (2) Using another glass sample holder, fill the indentation uniformly with the sample, making sure that the sample surface aligns with the reference position.




2.8.2 Glass sample holder (for small amounts of sample when the zero background sample holder is unavailable)

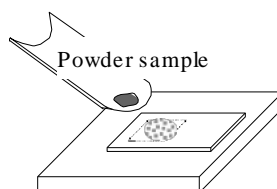
- (1) Apply a thin coat of a collodion solution diluted with isoamyl acetate on the back side of the glass sample holder.

 Tip: If you do not need to tilt the sample, there is no need to apply a coat of the above-mentioned solution.



- (2) Sprinkle the sample on the area coated with the collodion solution. Shake off excess sample powder.

 Tip: With this method, the glass may result in a visible halo.



2.8.3 Zero background sample holder (option)

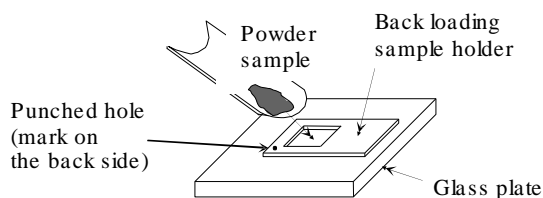
Place the sample in the indentation in the same way as for the glass sample holder. With very small amounts of sample, place the sample in the indentation on the back side of the zero background sample holder. When using the zero background sample holder, you will not see beams diffracted by the sample holder.



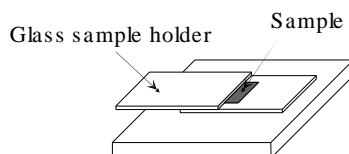
CAUTION: If the measuring optics does not use the diffracted beam monochromator or if a wavelength other than the Cu K beam is used for the measurement, you may note scattered or diffracted beams.

2.8.4 Back loading sample holder (for adequate sample amounts)

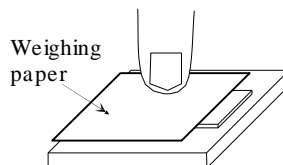
- (1) Place the sample holder on a flat glass plate so that the surface (reference position) of the sample holder contacts the glass plate. While pressing down on the sample holder with a hand to ensure firm contact, place the sample in the indentation (recessed section on the sample plate).



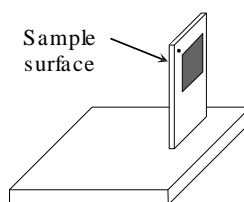
- (2) Using a glass sample holder, uniformly fill the indentation with the sample.



- (3) Place weighing paper on the sample and compact the sample using a fingertip, making sure that the sample holder does not move during this process.



- (4) If the sample remains in place and does not extend from the indentation when the sample holder is lifted, the sample is ready for measurement (the sample surface is the side placed against the glass plate.)

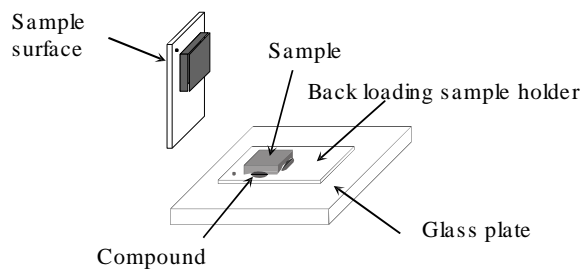


2.8.5 Back loading sample holder (for small samples)

Place the back loading sample holder on a glass plate, as described in Subsection 2.8.4, and secure the sample in place using clay or a similar modeling compound, as shown in the diagram below.



CAUTION: Since the modeling compound used to prepare the sample may either scatter or diffract x-rays, make sure the compound does not extend onto the sample surface.

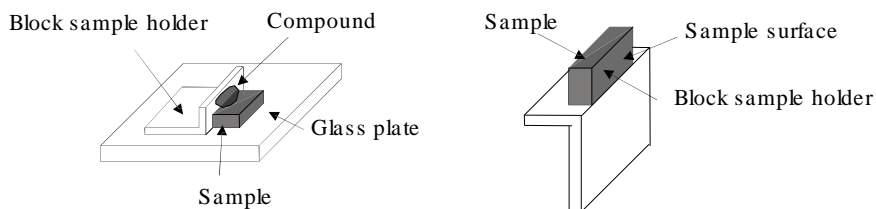


2.8.6 Block sample holder (option)

Secure the sample in place using clay or a similar modeling compound, as shown in the diagram below.



CAUTION: Since the modeling compound used to prepare the sample may either scatter or diffract x-rays, make sure the compound does not extend onto the sample surface.



3. Executing a Part individually

In this chapter, how to execute the following Parts individually is described.

- Optics Alignment (BB) Part
- Sample Alignment (BB) Part
- Quick Theta/2-Theta Meas. (BB) Part
- Precise Theta/2-Theta Meas. (BB) Part
- General Measurement (BB) Part

1. Executing the Optics Alignment (BB) Part

- (1) Click the Optics Alignment (BB) button on the flow bar to open the Optics Alignment (BB) dialog box.

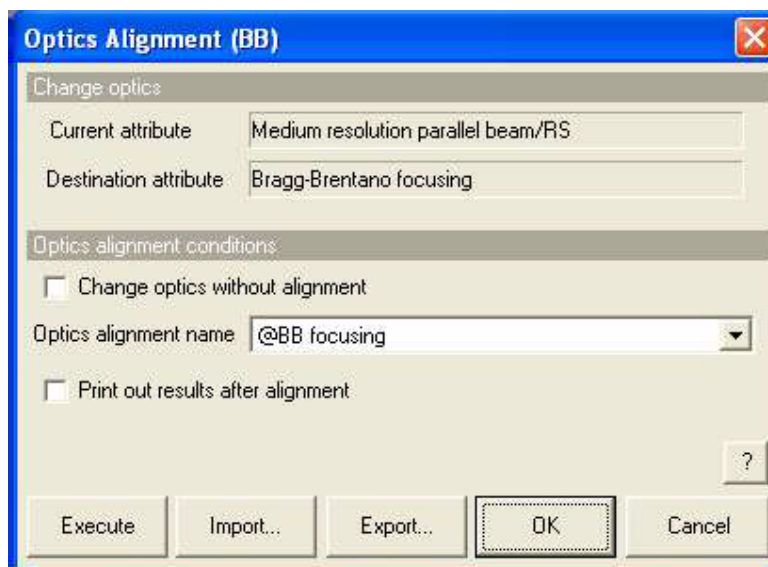


Fig. 3.1.1 Optics Alignment (BB) dialog box

- (2) Uncheck the Change optics without alignment box.

i Tip: Check the Change optics without alignment box to switch optics for the data measurement using the alignment results stored under the selected optics alignment name without performing optics alignment.

- (3) In the Optics alignment name box, select a location for storing optics alignment results.

i Tip: To store optics alignment results under a new optics alignment name, click the OK or Cancel button to close the Optics Alignment (BB) dialog box. Then, select the Optics Management command from the Option menu to open the Optics Management dialog box and add a new optics alignment name. After adding a new optics alignment name, return to step (1) in this section. For more information on creating an optics alignment name, refer to Chapter 17 of the SmartLab Guidance Reference Manual (ME13365A).

- (4) To print the optics alignment results, check the Print out results after alignment box.



Tip: If both the Change optics without alignment and Print out results after alignment boxes are checked, the alignment results stored under the selected optics alignment name will be printed.

- (5) Click the Execute button in the Optics Alignment (BB) dialog box.

- (6) Optics alignment is executed followed by steps (5) through (7) in Section 2.5.



CAUTION: If the current hardware configuration setting is different from that for the operation of the Optics Alignment (BB) Part, the optics alignment will be aborted and the Hardware Configuration dialog box will open. If this happens, replace the configuration units (e.g., the attachment) as prompted by the message in the dialog box.



[2.6 Changing configuration units](#)

2. Executing the Sample Alignment (BB) Part

- (1) Click the Sample Alignment (BB) button on the flow bar to open the Sample Alignment (BB) dialog box.

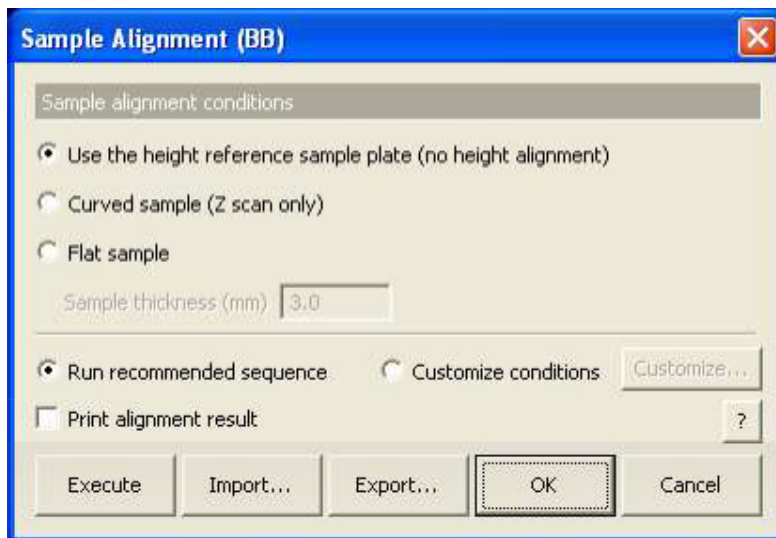




Fig. 3.2.1 Sample Alignment (BB) dialog box


- (2) To perform a data measurement using the height reference sample plate without performing sample alignment, select the Use the height reference sample plate (no height alignment) radio button.

 **Tip:** If one of the sample holders described in Section 2.8 is used or if the sample can be aligned visually to the reference position of the height reference sample plate, we recommend selecting the Use the height reference sample plate (no height alignment) radio button.


[2.8 Sample preparation](#)




- (3) To perform sample alignment, select the Curved sample (Z scan only) or Flat sample radio button and enter the thickness of the sample in the Sample thickness (mm) box.

 **Tip:** Use the wafer sample plate for sample alignment. Selecting the Curved sample (Z scan only) radio button sets the sample spacer and performs the vertical sample position (Z scan only) alignment.

- (4) To print the sample alignment results, check the Print alignment result box.


 **Tip:** If the Use the height reference sample plate (no height alignment) radio button is selected and the Print alignment result box is checked, only the alignment positions of the axes of the current optics will be printed.

- (5) Select the Run recommended sequence radio button.

 Tip: To confirm or set the scan conditions, select the Customize conditions radio button, then click the Customize button.

 [2.4.1 Customizing Sample Alignment \(BB\) Part conditions](#)

- (6) Click the Execute button in the Sample Alignment (BB) dialog box.
(7) Sample alignment is executed followed by steps (8) and (9) in Section 2.5.

 CAUTION: If the current hardware configuration setting is different from that for the operation of the Sample Alignment (BB) Part, the sample alignment will be aborted and the Hardware Configuration dialog box will open. If this happens, replace the configuration units (e.g., the attachment) as prompted by the message in the dialog box.

 [2.6 Changing configuration units](#)

3. Executing the Quick Theta/2-Theta Meas. (BB) Part

- (1) Click the Quick Theta/2-Theta Meas. (BB) button on the flow bar to open the Quick Theta/2-Theta Meas. (BB) dialog box.

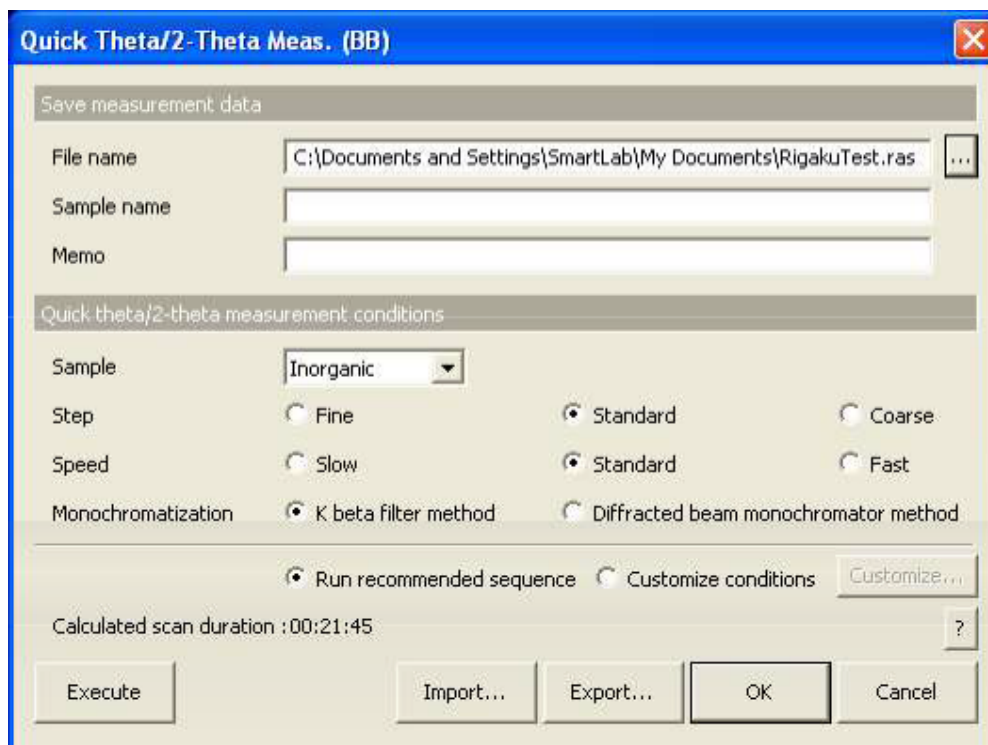


Fig. 3.3.1 Quick Theta/2-Theta Meas. (BB) dialog box

- (2) Set the folder to store the measurement data and the name of the measurement data file in the File name box in the Save measurement data section. After completing the quick theta/2-theta measurement, the measurement data will be saved with the specified file name.
 1. Click the [...] button to open the Save As dialog box.

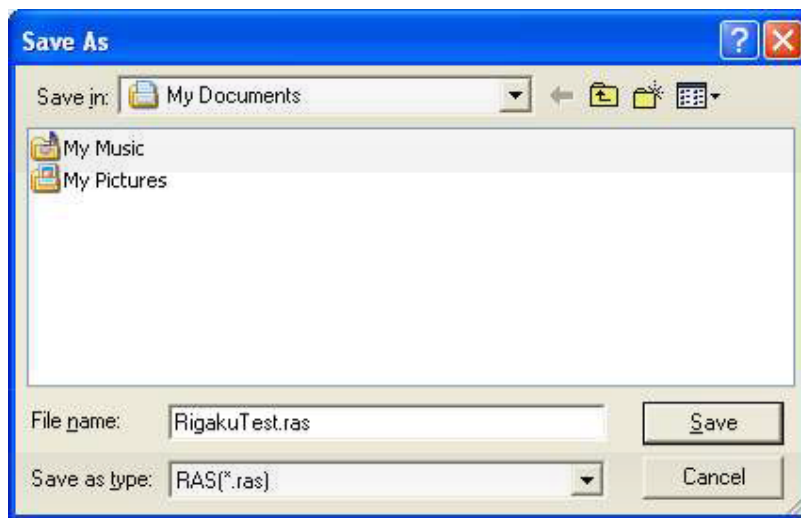


Fig. 3.3.2 Save As dialog box

2. Enter the settings in the Save in and File name boxes.
3. Click the Save button.
- (3) Enter any required information in the Sample name and Memo boxes (optional).



CAUTION: The information entered here will be saved to the file. Any number of characters may be entered, but only the first 30 characters of the sample name and the first 84 characters of the memo will appear on the printed measurement data.

- (4) In the Sample box, select the type of sample to be measured from Inorganic, Organic, Mineral, or Metal. If the sample is a mixture of different material types or the material of the sample is unknown, select Unknown.
- (5) Select the Standard radio button for Step.



Tip: Selecting the Standard radio button sets the step width to 1/5 of the FWHM (0.1 °) of ordinary diffracted beams. If the step width is suspected to be narrower or wider than 1/5 the FWHM (0.1°) of ordinary diffracted beams, select Fine or Coarse.

- (6) Select the Standard radio button for Speed.



Tip: Selecting the Standard radio button will result in measurement duration of 15 to 30 minutes. The data thus obtained should cause no problems in the phase ID analysis of an ordinary sample. However, if you wish to improve the quality of the measurement data, select Slow. To shorten measurement duration, select Fast.

- (7) Select the K beta filter method radio button for Monochromatization.



Tip: If the diffracted beam monochromator unit (option) is available, it is recommended to select the Diffracted beam monochromator method radio button. This setting removes almost all x-rays (K x-rays, continuous x-rays, fluorescent x-rays, etc.) other than those of the selected wavelength and provides measurement data with a low background level.

- (8) Select the Run recommended sequence radio button.



Tip: To confirm or set the scan conditions, select the Customize conditions radio button, then click the Customize button.

[2.4.2 Customizing Quick Theta/2-Theta Meas. \(BB\) Part conditions](#)



- (9) Click the Execute button in the Quick Theta/2-Theta Meas. (BB) dialog box.

- (10) Quick theta/2-theta measurement is executed followed by steps (10) through (12) in Section 2.5.



CAUTION: If the current hardware configuration setting is different from that for the operation of the Quick Theta/2-Theta Meas. (BB) Part, the quick theta/2-theta measurement will be aborted and the Hardware Configuration dialog box will open. If this happens, replace the configuration units (e.g., the attachment) as prompted by the message in the dialog box.

[2.6 Changing configuration units](#)



4. Executing the Precise Theta/2-Theta Meas. (BB) Part

- (1) Click the Precise Theta/2-Theta Meas. (BB) button on the flow bar to open the Precise Theta/2-Theta Meas. (BB) dialog box.

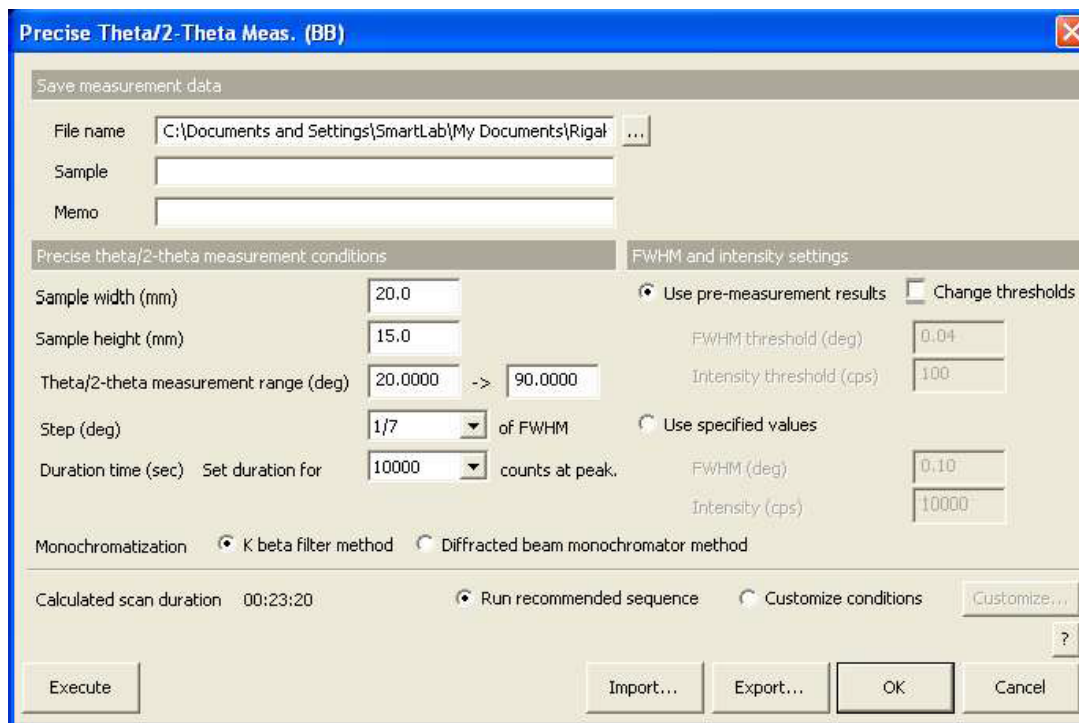


Fig. 3.4.1 Precise Theta/2-Theta Meas. (BB) dialog box

- (2) Set the folder to store the measurement data and the name of the measurement data file in the File name box in the Save measurement data section. After completing the precise theta/2-theta measurement, the measurement data will be saved with the specified file name.
 1. Click the [...] button to open the Save As dialog box.

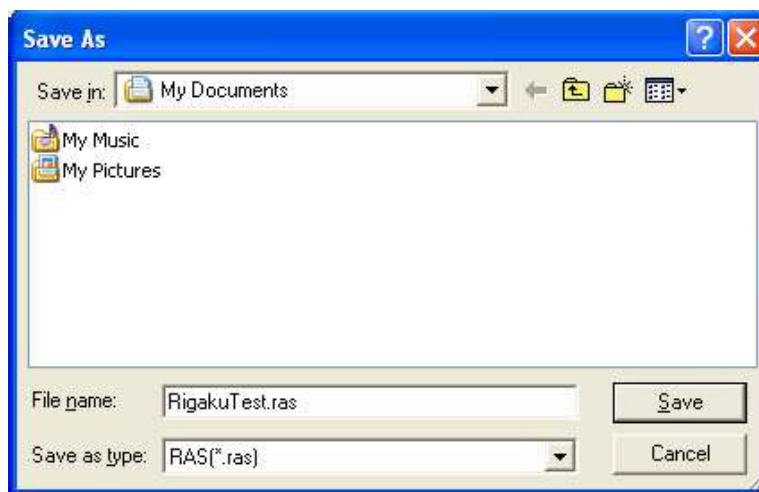


Fig. 3.4.2 Save As dialog box

2. Enter the settings in the Save in and File name boxes.
3. Click the Save button.

- (3) Enter any required information in the Sample name and Memo boxes (optional).



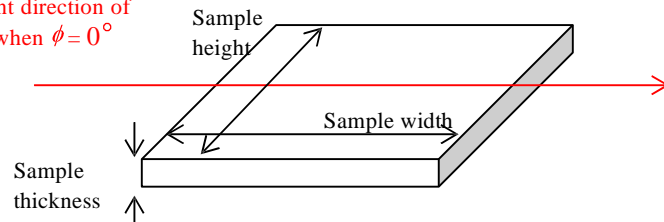
CAUTION: The information entered here will be saved to the file. Any number of characters may be entered, but only the first 30 characters of the sample name and the first 84 characters of the memo will appear on the printed measurement data.

- (4) Enter the sample width and sample height in the Sample width (mm) and Sample height (mm) boxes.



Tip: The sample width and sample height refer to the dimensions (unit: mm) of the sample in the directions shown below.

Incident direction of
x-ray when $\phi = 0^\circ$



- (5) Enter the scan range in the Theta/2-theta measurement boxes. Enter the start angle in the left box, and the stop angle in the right box.
- (6) Select 1/7 in the Step (deg) box.
- (7) Select 10000 in the Duration time (sec) box.



Tip: The duration time will be calculated so that the intensity of the highest peak becomes the selected value (unit: counts).

- (8) Select the K beta filter method radio button for Monochromatization.




Tip: If the diffracted beam monochromator unit (option) is available, it is recommended to select the Diffracted beam monochromator method radio button. This setting removes almost all x-rays (K x-rays, continuous x-rays, fluorescent x-rays, etc.) other than those of the selected wavelength and provides measurement data with a low background level.

- (9) Select the Use pre-measurement results radio button for FWHM and intensity settings.




Tip: To set the threshold of FWHM or intensity, or to set the FWHM and intensity when pre-measurement has been already made, refer to "Precise Theta/2-Theta Measurement (BB) Part" Help Topic of the online help section of the SmartLab Guidance software.


- (10) Select the Run recommended sequence radio button.


 Tip: First, pre-measurement is made. Second, data measurement is made under the condition determined based on the pre-measurement results.

To confirm or set the scan conditions, select the Customize conditions radio button, then click the Customize button.

 [2.4.3 Customizing Precise Theta/2-Theta Meas. \(BB\) Part conditions](#)

- (11) Click the Execute button in the Precise Theta/2-Theta Meas. (BB) dialog box.
- (12) Precise theta/2-theta measurement is executed followed by similar steps (10) through (12) in Section 2.5.

 CAUTION: If the current hardware configuration setting is different from that for the operation of the Precise Theta/2-Theta Meas. (BB) Part, the precise theta/2-theta measurement will be aborted and the Hardware Configuration dialog box will open. If this happens, replace the configuration units (e.g., the attachment) as prompted by the message in the dialog box.

 [2.6 Changing configuration units](#)

5. Executing the General Measurement (BB) Part

- (1) Click the General Measurement (BB) button on the flow bar to open the General Measurement (BB) dialog box.

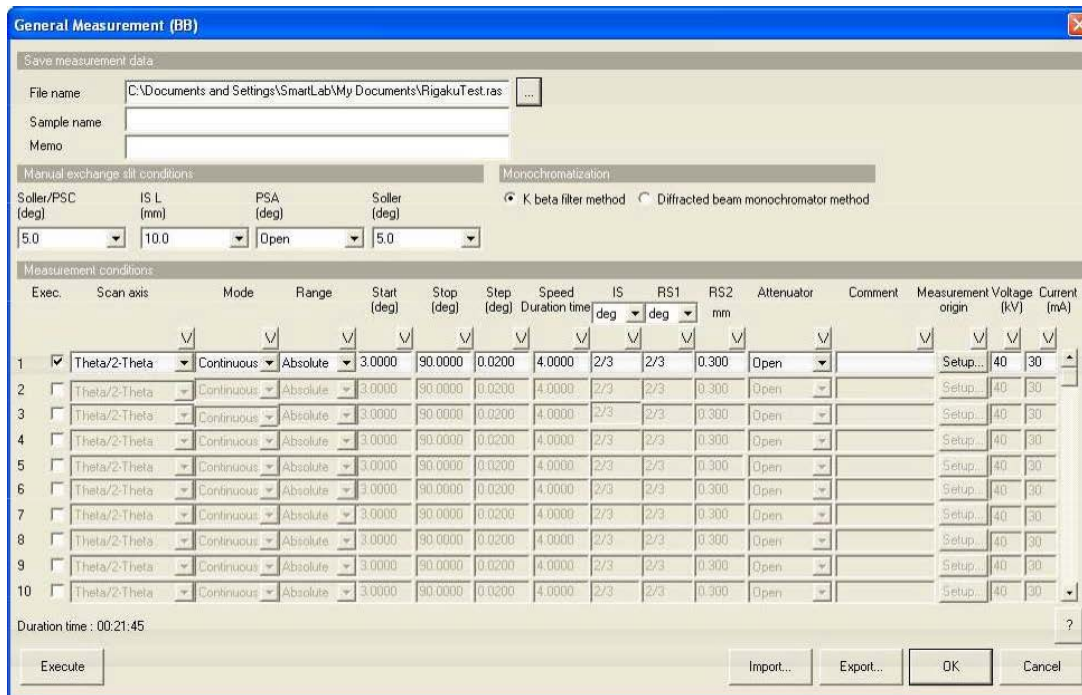


Fig. 3.5.1 General measurement (BB) dialog box

- (2) Set the folder to store the measurement data and the name of the measurement data file in the File name box in the Save measurement data section. After completing the data measurement, the measurement data will be saved with the specified file name.

1. Click the [...] button to open the Save As dialog box.

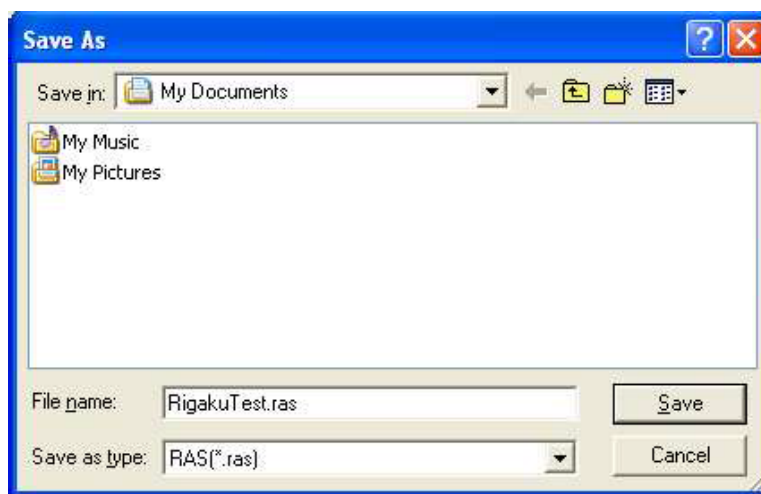


Fig. 3.5.2 Save As dialog box

2. Enter the settings in the Save in and File name boxes.
3. Click the Save button.

- (3) Enter any required information in the Sample name and Memo boxes (optional).



CAUTION: The information entered here will be saved to the file. Any number of characters may be entered, but only the first 30 characters of the sample name and the first 84 characters of the memo will appear on the printed measurement data.

- (4) Set the manual exchange slit conditions.

- (5) Set the measurement conditions.



Tip: To set the manual exchange slit conditions and measurement conditions, refer to “General Measurement (BB) Part” Help Topic of the online help section of the SmartLab Guidance software.

- (6) Select the K beta filter method radio button for Monochromatization.



Tip: If the diffracted beam monochromator unit (option) is available, it is recommended to select the Diffracted beam monochromator method radio button. This setting removes almost all x-rays (K x-rays, continuous x-rays, fluorescent x-rays, etc.) other than those of the selected wavelength and provides measurement data with a low background level.

- (7) Click the Execute button in the General Measurement (BB) dialog box.

- (8) Data measurement is executed followed by similar steps (10) through (12) in Section 2.5.



CAUTION: If the current hardware configuration setting is different from that for the operation of the General Measurement (BB) Part, the data measurement will be aborted and the Hardware Configuration dialog box will open. If this happens, replace the configuration units (e.g., the attachment) as prompted by the message in the dialog box.

[2.6 Changing configuration units](#)



4. Troubleshooting

Problem	Response
Previously saved conditions cannot be imported through the Optics Alignment (BB) dialog box.	<ul style="list-style-type: none"> ● Confirm that the user name displayed on the title bar of the main window is correct. ● Add the name of the optics alignment again in the Optics Management dialog box.
Previously saved conditions cannot be imported through the Sample Alignment (BB) dialog box.	<ul style="list-style-type: none"> ● Confirm that the user name displayed on the title bar of the main window is correct.
Previously saved conditions cannot be imported through the Quick Theta/2-Theta Meas. (BB) dialog box.	<ul style="list-style-type: none"> ● Confirm that the user name displayed on the title bar of the main window is correct.
Previously saved conditions cannot be imported through the Precise Theta/2-Theta Meas. (BB) dialog box.	<ul style="list-style-type: none"> ● Confirm that the user name displayed on the title bar of the main window is correct.
Previously saved conditions cannot be imported through the General Measurement (BB) dialog box.	<ul style="list-style-type: none"> ● Confirm that the user name displayed on the title bar of the main window is correct.
Package measurement cannot be executed.	<ul style="list-style-type: none"> ● Check to see if the XG output is as specified. ● Check to see if the door of the radiation enclosure is closed.
When optics alignment is executed, the goniometer will move slightly and the operation will immediately stop.	<ul style="list-style-type: none"> ● Confirm that the Change optics without alignment box in the Optics Alignment (BB) dialog box is unchecked.
Clicking the OK button in a message box will result in the same message box reappearing.	<ul style="list-style-type: none"> ● Confirm that the specified slit and other devices are installed correctly. Also, make sure that the necessary devices have not been removed.
Sufficient intensity cannot be obtained for quick theta/2-theta measurement.	<ul style="list-style-type: none"> ● <u>Check to see if an absorber is inserted in the receiving slit box.</u> ● Confirm that the XG output has reached the specified levels. ● Measure the Si powder sample (Si powder reference sample) using the Run recommended sequence mode and check the intensity.