Rigaku

MiniFlex600
MiniFlex600-C

Getting Started Guide

This guide describes only basic instructions. Before starting to use the MiniFlex600/600-C benchtop X-ray diffractometer (hereafter called "MiniFlex"), be sure to read "Guidelines for safe use of the Benchtop X-ray Diffractometer" and "Benchtop X-ray Diffractometer Instruction Manual" thoroughly.

Starting up

【1】 Starting up MiniFlex600/600-C

1 Supply the cooling water.

MiniFlex600: Run the cooling water.

MiniFlex600-C: Make sure that the circuit protector on the rear side of MiniFlex is turned on.

2 Start up the MiniFlex control PC.

If the two-dimensional detector HyPix-400 MF is installed, start up the server PC of the detector as well.

3 Turn on the power breaker on the rear side of MiniFlex, and then press the power on button (green button) on the front side.

4 The Door Lock button will blink. Press the Door Lock button (yellow button). The door will be locked.

5 Make sure that the OPERATE lamp on the front side of MiniFlex turned yellow.

6 Start up SmartLab Studio II on the MiniFlex control PC.

Default login name: Administrator

Default password: rigaku0

Login name set by the customer:_____

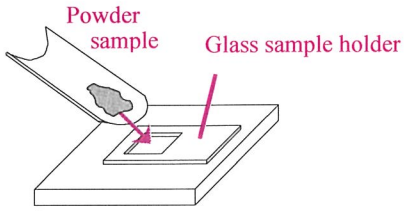
Password set by the customer:_____

Measurement

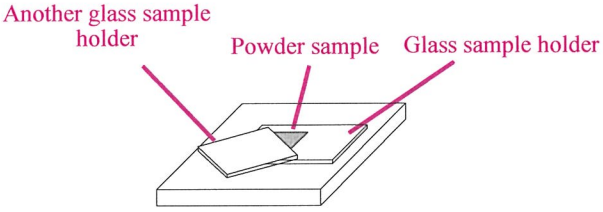
【2】 Preparing a sample

The following procedure explains sample preparation using a glass sample holder (included in the standard configuration) and a powder sample as an example.

1 Pour the powder sample into the sample filling section of the glass sample holder.



2 Flatten and compress the sample surface.

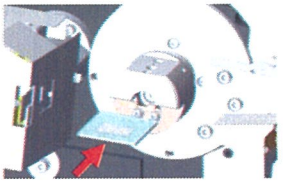


Distribute the sample evenly so that the sample surface is flattened and is the same height as the glass sample holder. Gently compress the sample by using the rear side of another glass sample holder or the like.

3 If the powder sample remains around the sample filling section, wipe it off by using a cleaning paper soaked in alcohol, etc.

4 Press the Door Lock button to unlock and open the sample chamber door on the front side.

5 Mount the glass sample holder onto the standard sample stage.



6 Close the sample chamber door of MiniFlex, and press the Door Lock button to lock the door.

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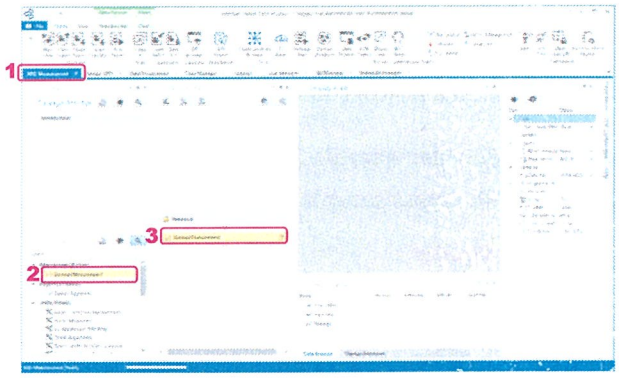
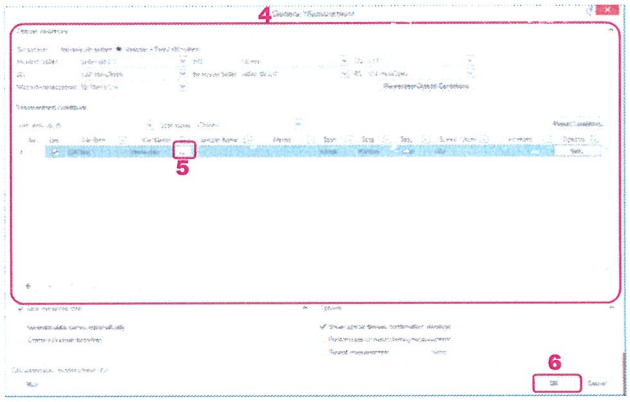
【3】 Setting measurement conditions

1 Select the [XRD Measurement] plugin tab in SmartLab Studio II.



2 Double-click the [General Measurement] Part on the [Part Activities] list to add it to the [Sequence].

3 Click the [General Measurement] Part, that was added to [Sequence] to open the [General Measurement] dialog box.



4 Set [Optical conditions] and [Measurement conditions].

An example of measurement conditions for qualitative analysis is provided for your reference.

Slit system: Variable + Fixed slit system
Incident Soller slit: Soller slit 2.5° or 5.0°
IHS: 10 mm
DS: 1.25°

Note: If main diffracted rays are present near 2θ=10°, select the 0.625° DS.

SS: 13.0 mm (Open)
Receiving Soller slit: Soller slit 2.5° or 5.0°
RS: 13.0 mm (Open)
Monochromatization: Kβ filter (x1.5)

Note: Insert the Kβ filter into the SS section.

Scan axis: 2θ/θ
Scan mode: 1D (Scan)

Start: 3°
Stop: 90°
Step: 0.01°
Speed: 10°/min

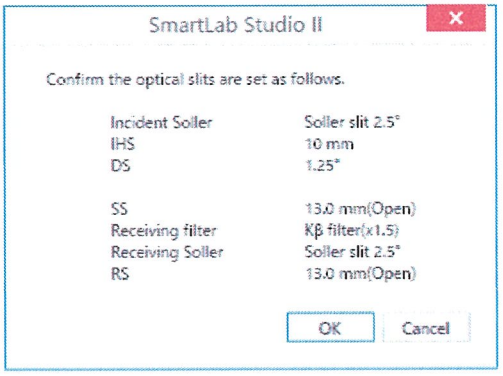
5 Click the button next to the [File Name] box, and specify any file path and file name.

6 Click the [OK] button to complete the settings.

【4】 Starting a measurement

1 Click the [Home] tab – the [Run Flow] button on the ribbon to start the measurement.

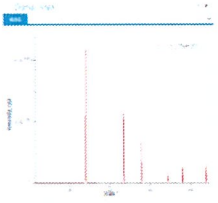
2 The message window appears. Install optical devices, according to the message. Then, click the [OK] button.



Message example

3 X-rays will be turned on, and the tube voltage and tube current will be set to the measurement values. Then the measurement will begin.

Note: The measurement results are displayed on the [Display Area] panel.



Measurement example

Analysis

Analysis is performed with the standard Data Manager plugin and Powder XRD plugin. This section describes the basic data processing for the determination of the peak position and integrated intensity of the measured data (【5-1】) and crystal structure determination (【5-2】).

【5-1】 Data Manager plugin

The Data Manager plugin allows one- and two-dimensional measured data display, simple data processing, and peak search.

1 Select the [Data Manager] plugin tab in SmartLab Studio II.

2 Click the [Home] tab – [Load Data] on the ribbon.

3 Select the measured data (e.g., measured data obtained in " 【4】 Starting a measurement"), and then click the [Open] button.

The data is loaded, and its profile is displayed.

4 Run the peak search.

(1) Click the [Chart] tab on the ribbon. If the [Chart] tab is not displayed, click the [Viewer] panel to display it.

(2) Click [Peak Search] to display the [Peak Search] dialog box.

(3) Set the peak search conditions, and then click the [Run] button.

5 Click the [Peak List] tab to display the peak search results (peak position, intensity, etc.).

6 Save the [Peak Search] results.

(1) Click the [Home] tab – [Save Solution As] on the ribbon.

(2) Enter the file name and click [Save].

【5-2】 Powder XRD plugin

In Powder XRD plugin, data is automatically processed during loading. After the data processing, analysis such as crystal structure determination can be performed.

1 Select the [Powder XRD] plugin tab in SmartLab Studio II.

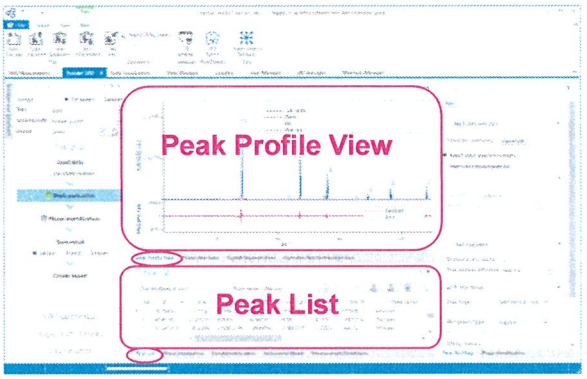
2 Perform loading and processing of measured data.

(1) Select [Basic] from [Task] on the flow bar.

(2) Click [Evaluation] to expand the evaluation flow.

(3) Click [Load Data] to display the [Load Measured Data] dialog box.

(4) Select measured data and click [Open]. Peak evaluation will be automatically performed. The profile will be displayed on the [Peak Profile View] panel as shown below, and the refined peak information (e.g., position, intensity, FWHM) will be displayed on the [Peak List] panel.



3 Determine crystal structure.

(1) Click [Phase identification] on the flow bar.

(2) Click the button on the right of the [Import] button on the [Phase Identification] panel.

(3) To use a database for assigning substances, select [d-I database]. To use your own CIFs for the assignment, select [File] and the corresponding CIFs in the displayed dialog box.

(4) If [d-I database] is selected, [d-I database]

dialog box will appear. Set the search conditions such as the crystal phase in the displayed dialog box, and then search for substances. After the search, select the check boxes of the corresponding cards and click the [Add] button.

(5) Click the [Close] button.

(6) Select the entry that has been newly added to the [Search results] list and click  (Add to the candidate phase list).

(7) Click the [Confirm] button in the [Candidate phase] list.

4 Save the analysis result.

(1) Select [Solution] in [Save result] on the flow bar, and then click [Save result].

(2) Enter the file name and click the [Save] button.

Shutting down

【6】 Shutting down MiniFlex600/600-C

1 Confirm that the X-rays are stopped.

If the X-rays are not stopped, stop it according to the procedure below.

(1) Click the [XRD Measurement] plugin tab in SmartLab Studio II, and then click the [Home] tab - [Startup/Shutdown]  on the ribbon. The [Startup/Shutdown] panel will appear.

(2) Select [XG Off] and click the [Run] button to stop the X-ray generation.

(3) When the X-ray generation stops, the orange X-ray warning lamp on top of the enclosure will turn off.

2 Shut down SmartLab Studio II.

3 Wait at least <u>three minutes</u> after X-ray generation stopped, and press the Power off button (a white button) on the front side of MiniFlex.

4 Turn off the power circuit breaker on the rear side of MiniFlex.

5 Shut down the MiniFlex control PC.

If the two-dimensional detector HyPix-400 MF is installed, press the power button of the detector server PC to shut down the server PC as well.

6 Stop the cooling water. (for MiniFlex600 only)

MiniFlex600:	Stop the cooling water.
MiniFlex600-C:	No action is required. (The built-in cooling unit automatically stops in three minutes after X-rays have been stopped)

Instruction Manual (Electronic manual)

If you need more details on the usage and other functions, please read the following manuals (electronic manuals).

SmartLab Studio II User Manual

To refer to this manual, click [File] - [Manual] on the ribbon in SmartLab Studio II.

MiniFlex600/600-C instruction manual, XRD Measurement plugin Parts Help

To refer to Help, open the SmartLab Studio II XRD Measurement plugin, and then click the [Home] tab - [Help Viewer]  on the ribbon.

Contact Information

Contact your sales representative.

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