Standard Operating Procedure
Rigaku SmartLab XRD

I. Powder X-ray Diffraction (PXRD)

Yale West Campus
Materials Characterization Core
ywcmatsci.yale.edu

ESC II, Room A119C
810 West Campus Drive
West Haven, CT 06516

Version 1.3, August 2020
> **FOLLOW** the SOP strictly to keep the instrument in good condition. **No** explorations allowed on software unless permitted by lab manager

> **NEVER** use your own USB drive on the XPS computer. Data can be either uploaded to Yale Box, or copied to the Jump Drive provided by the Core.

> **NEVER** surf the web on the XPS computer to minimize the risk of the computer being hacked

> Users should **acknowledge** MCC in their publications. Please check the following link for details:

  http://ywcmatsci.yale.edu/publications

> The core reserves the right to use the data for core promotion
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Rigaku SmartLab XRD Standard Operating Procedure

1 Introduction

a) Instrument features:
   > Full automated alignment under computer control
   > A high-efficiency 2D detector (Hypix 3000) with high-count rate
   > Cross Beam Optics (CBO) permits easy switching between focusing (BB) and parallel beam (PB) geometries without reconfiguration
   > In-plane diffraction arm for in-plane measurements without reconfiguration
   > High temperature (~1500 C) measurements in air, vacuum and helium
   > Air-Tight Sample Container for contamination free measurement

2) Location
   Materials Characterization Core
   Room A119C
   810 West Campus Drive
   West Haven, CT 06516

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The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

Notice: Please follow strictly the SOP to keep the facility under good condition. No explorations on program allowed unless approved by core manager.
2 System Status Check

1) Please check to make sure the instrument sliding door is closed and there is no beeping sound once getting into the XRD room.

2) If there is a beeping sound from the instrument and meanwhile the OPERATE light is flashing as show below. Hit the white OFF button on the front panel above and contact manager immediately.

3) Meanwhile, please check the floor at the back corner of the machine and make sure no water leaking on the floor. If no water leaking and the manager can’t be reached, before leaving, put a machine down not on the machine sliding door.

4) If there is water leaking, it must be taken care before leaving. Please go to the service corridor A117 outside the Core in the next door and shut down the XRD chiller at the end of the corridor. The key is hang on the shelf right across the manager’s office.

5) Come back to the XRD room and clean the water leak with paper tower.

6) Leave a machine down note on the machine sliding door.
3 Sample Preparation

1) **Wear gloves** and clean the sample holder with provided IPO.
2) Powder samples should be milled to below 5 µm to minimize measurement deviations due to particle size effects.
3) Two types of powder sample holders, the **zero-background** holder and **glass** holder are provided as shown below. An amorphous hump signal could appear in the spectra on glass holder, which can be avoided if use the **zero-background** holder.

4) Put powder samples at the center of the circle on the zero-background holder or square on the glass holder as shown below; cover the powder with a **weighing paper** and press and rotate sample with a glass slide to flat the sample surface.

4 XRD Computer Login

1) Sign in on the logbook.
2) Login FOM system: click on **Click here to login with NetID** to unlock the screen lock.
3) Check system status:
   a) Open the SmartLab Guiddance software if it was closed (login: administrator, password: rigaku).
   b) Check the highlighted areas on the bottom left of the Hardware Status window as shown below:
      > Make sure that the “CBO selection slit” is on BB (Bragg-Brentano focusing) mode,
      > No number display on Tube voltage and Tube current if the X-ray is at shutdown status.

   c) If the Tube voltage and Tube current read 40 kV and 44 mA, skip Step d) and e) below.

   d) If the system is at shutdown status, click button in the Package/Macro Measurement window below:
e) In the popup **Startup** window below, choose **Use everyday** if the machine was used within 24 hours and click **Execute** button. It takes 15 minutes for the system to reach the operation power of **40 kV** and **44 mA**.

![Startup Window](image)

5 User Package Definition

1) On the **Package** window at the top right of software window, find **Preinstalled** > **General Measurement** > **General (Bragg-Brentano Focusing)** package as shown below:

![Package Window](image)
2) Double Click on General (Bragg-Brentano Focusing) to open the package in the Package/Macro Measurement window below which composes of three parts as shown below:

![Package/Macro Measurement window](image)

3) Click on Save As button on above window to save the package as user defined package which will appear in the User defined tab on the top right side of the software window. On the bottom of User defined tab click to create a user folder and move saved package file into the folder by clicking .

6 Sample Loading

1) Press the Door Lock button on the instrument cabinet door, wait till the button flashes, and then grab the handle (highlighted below) to slide the door open.

![Door Lock button](image)
2) Push the glass or zero background holder all the way into the **height reference sample plate** as shown below. Align the holder to the center of the **plate** as highlighted by a dashed yellow line.

**Note:** If no enough sample to cover the entire holder, try to **extend** the sample area along the **longer** side of holder (dashed yellow line)

![Height reference plate](image1)

4) Insert the right slit size **IS L** (length limiting incident slit) as shown in the left picture below. Choose typical sizes from **2, 5, or 10 mm**. The x-ray beam size is doubled when reaching sample surface as shown on the illustration below. For instance, if choosing 5 mm slit size, the exposed sample surface area/length will become ~ 10 mm across the X-ray beam.

![Slit L](image2)

5) Close the cabinet door and hit **Door Lock** button

7 **Optics Alignment**

**Only required** if **PB** (parallel beam) mode was shown in **Hardware Status** window (check **Step 2b**) in **Section 3**.
1) Insert the **BB** slit into the **CBO** adapter and click on **1 Optics Alignment (BB)** in the **Package/Macro Measurement** window below:

![Package/Macro Measurement window](image1.png)

2) Click **Execute** button on the popup **Optics Alignment (BB)** window below to start optics alignment:

![Optics Alignment (BB) window](image2.png)

3) Follow the instructions on the popup **SmartMessage** window below to replace required parts and click **OK** to continue. The **Optics Alignment (BB)** window will be active after finish in ~5 mins.
4) Click OK on the **Optics Alignment (BB)** window in Step 2) above after finish.

8 Sample Alignment

1) Open user specific package file in **User defined** tab created in **Section 4 User Package Definition**.

2) Click on ![2 Sample Alignment (BB)](image) as highlighted in the **Package/Macro Measurement** window below:

3) In the pop-up **Sample Alignment (BB)** window below:
   a) Choose **Curved sample (Z scan only)** for powder samples.
b) For powder samples, input **2.0** as **Sample thickness (mm)** if the sample surface is flush with holder surface;  
c) For solid samples, input **2.0 + solid sample height above holder** as **Sample thickness (mm)** if the sample is higher than the holder.  
d) For multiple samples with similar thickness or if powders are flush with the holder, **no need** to repeat sample alignment.  
e) Check the box next to **Put a sample when the sample alignment starts** in above **Sample Alignment** window and click **Execute** button.  
f) Click **OK** button on the popup **SmartMessage** window below. The **Sample Alignment (BB)** window will be back active in ~ 2 minutes after finish.

4) Click **OK** on the **Sample Alignment (BB)** window in Step 3) above to quit the window.

9 Sample Measurement

1) Click on button as highlighted in the **Package/Macro Measurement** window below:
2) Perform following steps on the popup **General Measurement (BB)** window below:

a) Specify **File name** and folder as highlighted in the window below.

b) Check **K beta filter method**

c) Select **1D mode**

d) Click **Read current slits** button

e) Set measurement conditions:
   
   > **Exec**: click/check small box to activate line 1.
   > **Mode**: **Continuous**
   > **Range**: **Absolute**
   > **Start (deg)**: **10.0000**. **Never change to below 5**. The detector will be damaged.
   > **Stop (deg)**: **90.0000**, the upper limit.
   > **Step (deg)**: **0.0500**
   > **Speed Duration time (degree/min)**: **10.0000**, choose lower speed to smooth spectra.
   > **IS mm**: **1**, recommended size.
   > **RS1 mm**: **20.0000**, recommended size.
   > **RS2 mm**: **20.1**, recommended size.
   > **Voltage (kV)**: **40**, maximal voltage. **Never change to above 40 to damage the X-ray tube**.
   > **Current (mA)**: **44**, maximal current. **Never change to above 44 to damage the X-ray tube**.

f) Select box near **Drive the 4 axes to the current zero positions after measurement completed**.

g) Click **Execute** button to start measurement

h) Follow the instruction on SmartMessage windows if appear and hit **OK**
3) XRD spectra will be automatically saved to specified user folder after scan.

4) Click the button highlighted in the window below to save modified measurement parameters into user defined package.
10 Checklist after Experiment

1) Back up your data to Yale Box cloud drive (box.yale.edu) or using Core USB drive. Do not use personal USB drive.

2) Remove sample from the stage. Never leave samples inside XRD.

3) Shutdown instrument if finish after 5pm at regular time or anytime in off-peak hours.

Click \(\text{Shutdown...}\) in the window below:

4) Make sure XG Off is chosen and click Execute button in the popup Shutdown window below and watch the Tube current start to drop from 44 mA before logoff FOM.

5) Never minimize or close SmartLab Guidance software.

6) Data backup: upload data to box.yale.edu by using Firefox web browser. DO NOT use personal USB drive.
7) Logoff **FOM** program: click the 📖 icon on the taskbar below to activate the FOM program and click **Logoff** button in the FOM window. If any issues occurred during scan, check **“Something wrong”** and type message in the **Comments** space. The message will be sent to lab manager.

8) **Sign off** on the logbook.
9) **Clean** the sample holders and glass slides with clean wipes **IPA**.
10) **Put** the sample holder and other tools back into the tool box.