

Standard Operating Procedure (SOP)

Important: If at any point you are unsure how to proceed, stop and contact the instrument managers. Do not attempt to troubleshoot instrument hardware or change settings that were not covered during your training.

1. Visually inspect the instrument and chiller for errors or unusual conditions. If an error or unusual condition is observed, do not proceed and contact the instrument managers.
NEVER touch the transformer switch located under the PC desk.
2. The instrument PC and SmartLab Studio II software should normally remain on at all times.
 - If the screen is off or the system is in sleep mode, turn on the monitor or move the mouse to wake the system.
 - **Do not power off the instrument PC or close the SmartLab Studio II software.**
 - If the PC or software is slow, unresponsive, or not operating normally, do not attempt to restart the system; contact the instrument managers for assistance.
3. In the **Startup/Shutdown** menu, under the **Startup** tab, press **Run**. This will start the X-ray tube warm-up process (~7 min).*, **
4. Once the warm-up is complete, check the **H/W Status** menu.
 - The X-ray tube power should be stabilized at **40 kV/40 mA**.
 - Also check the cooling water flow rate, which should be between **4.3 and 5.0 L/min**. If the flow rate is outside this range, do not proceed and contact the instrument managers.

The instrument is now ready for use!
5. Prepare your sample and place it in the diffractometer:
 - 5.1. Add finely pulverized powder to the frosted cavity at the center of the glass sample holder and gently press it using a microscope glass slide. The sample surface should be level with the surface of the glass sample holder; it should neither protrude above nor sit below the surrounding holder surface.
 - 5.2. Unlock the diffractometer door, if locked, using the **“DOOR LOCK ON”** tab in the main software menu. Check the LED indicator on the instrument door before opening it.
 - 5.3. Open the diffractometer door and mount the sample holder to the appropriate position. The smooth side of the holder should slide all the way in towards the two

* This warm-up process is required only for the first user of the day.

** If the SmartLab Studio II software has been shut down, the warm-up process will take approximately 17 minutes. Therefore, **do not shut down the software or power off the instrument PC unless absolutely necessary**. If you are unsure what to do, please contact the training personnel: Ioannis Vamvasakis (i.vamvasakis@uoc.gr), or Costas Stoumpos (cstoumpos@uoc.gr).

clip holders. Close the door gently until you hear it click into place. If the door does not close or lock normally, do not attempt to force it and contact the instrument managers.

You are now ready to set up the measurement!

6. In the **Activities** menu, select **General Measurement (BB)** for measurements in Bragg–Brentano configuration. This is the only available option in the current instrument configuration. If you need to perform a measurement in a different configuration, please contact the instrument managers for further advice.
7. Double-click **General Measurement (BB)**. It should appear in the **Flow Editor** menu.
8. Double-click it again to enter the measurement parameter menu. The default values are:

Range	Start (deg)	Stop (deg)	Step (deg)	Speed (deg/min)	Incident Slit	Receiving slit #1	Receiving slit #2	Comment	Options
Absolute	5	80	0.01	4					

Options shown in green are editable according to the requirements of the experiment.***

9. Select a filename for your experiment and save the measurement in your group's subfolder in **RASX format**. Add the sample name, then check the **“Move to home position ...”** box, and click **OK**. In the main software menu, click **Run Flow**. The hardware-control window that appears is non-interactive and is provided for information only. Wait until the measurement is complete.

IMPORTANT: The measurement is complete only when the **“Flow sequence completed”** message appears. Click **OK**.

10. Once you have finished measuring, delete the **General Measurement (BB)** instruction from the **Flow Editor** menu (right-click → Delete) to make the instrument ready for the next user.
11. Remove your sample from the sample chamber and carefully:
 - Clean the sample holder with acetone or another appropriate solvent. Make sure that the holder is clean, dry, and free of any sample residue before returning it.
 - Check the instrument sample chamber for any spilled powder or sample residue and, if necessary, gently clean the sample mounting area using a paper towel or an appropriate brush. **Do not use solvents to clean components inside the sample chamber.**
12. If you are the last person using the instrument for the day, as indicated by the online instrument schedule, set the instrument to standby mode. Select the **Startup/Shutdown** menu and, from the **Shutdown** panel, select **Set to minimum** and press **Run**. This process takes approximately 30 seconds.

DO NOT close the SmartLab Studio II software or power off the instrument PC!!!

*** Do not start measurements below $2\theta = 1^\circ$. At these very low angles, the direct X-ray beam may reach the detector and potentially cause damage!