Ultima IV Powder XRD

MPU-4 Thin Film Configuration

Dr. Jianfeng Zhu

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Do NOT Remove from XRD lab!

1. Turn On the Diffractometer

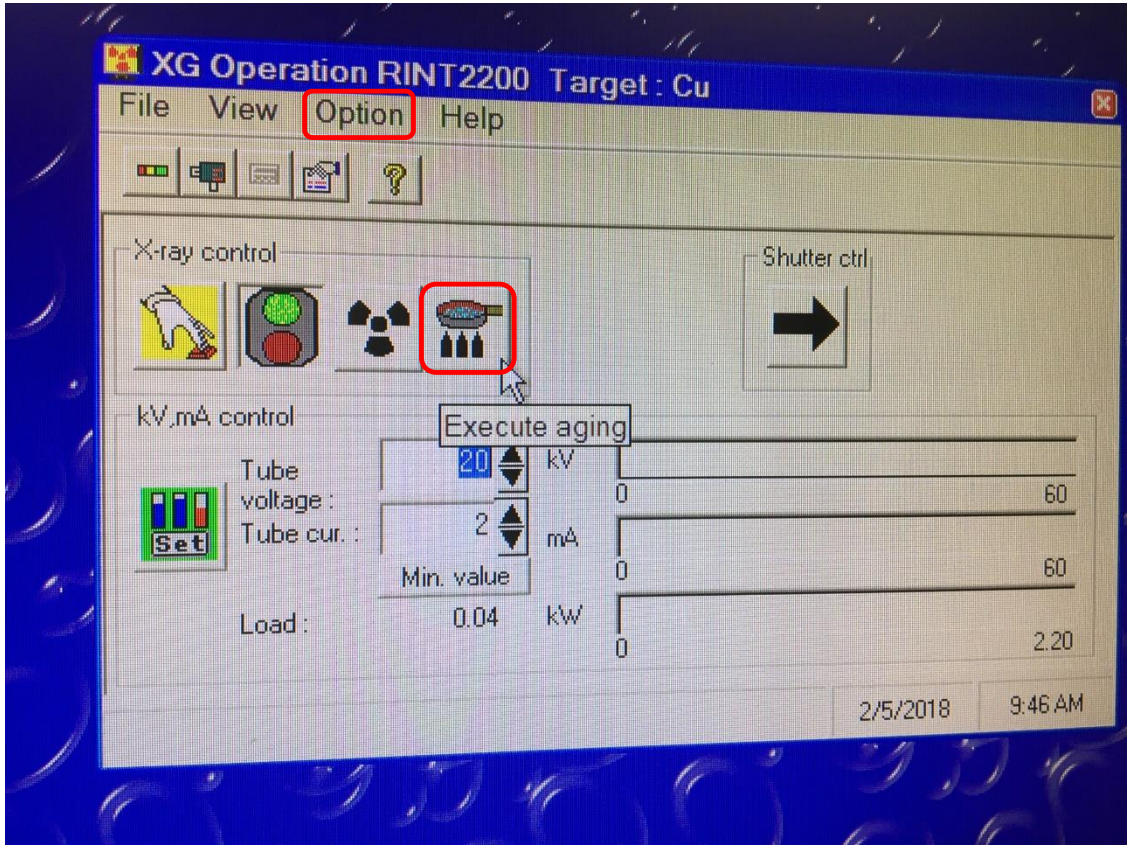
1) Turn on the Haskris (cooling water for X-Ray tube).
   Flow rate: ~4 L/min;
   Temperature: 65 – 69 K (the compressor will turn on when reaching 69 K).

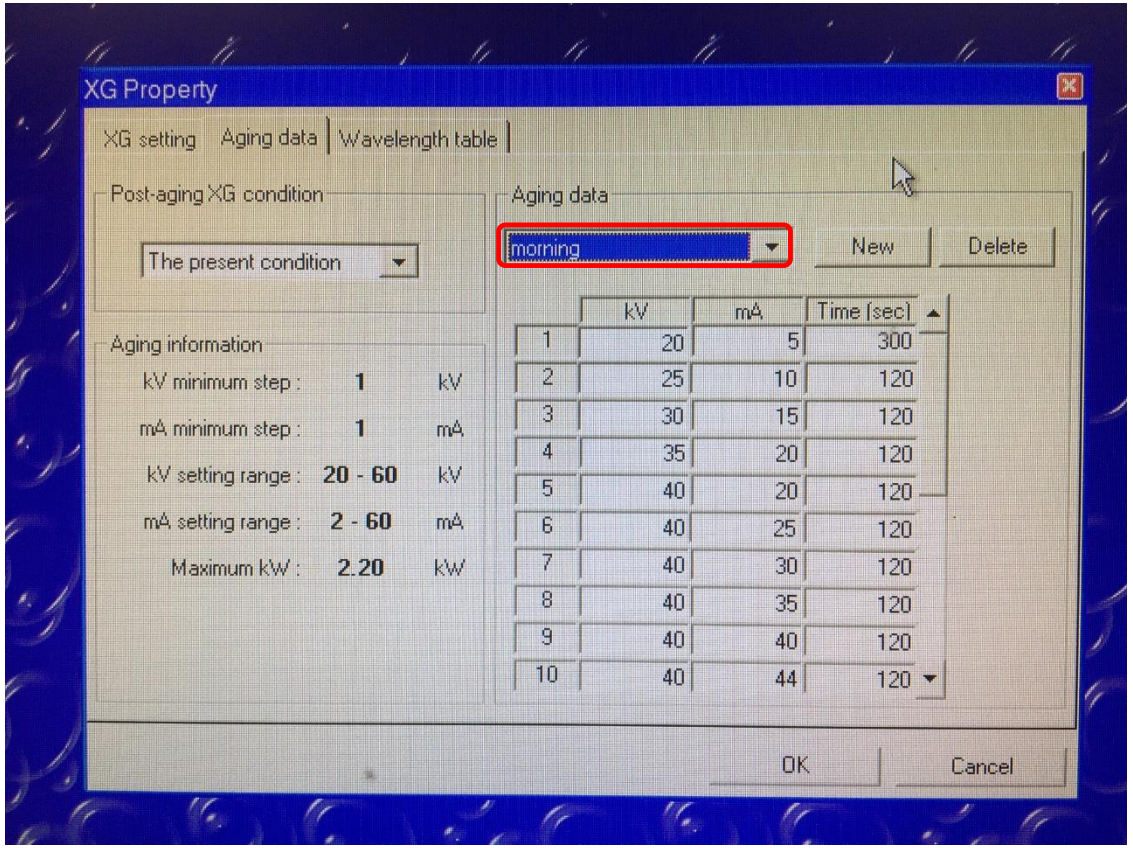
2) Turn on the power of Ultima IV (90° clockwise turn). The OPERATE LED will be flashing and remain green after ~20 seconds.

3) Switch the X-Ray enable key ON (90° clockwise turn).

2. Aging the X-Ray Tube

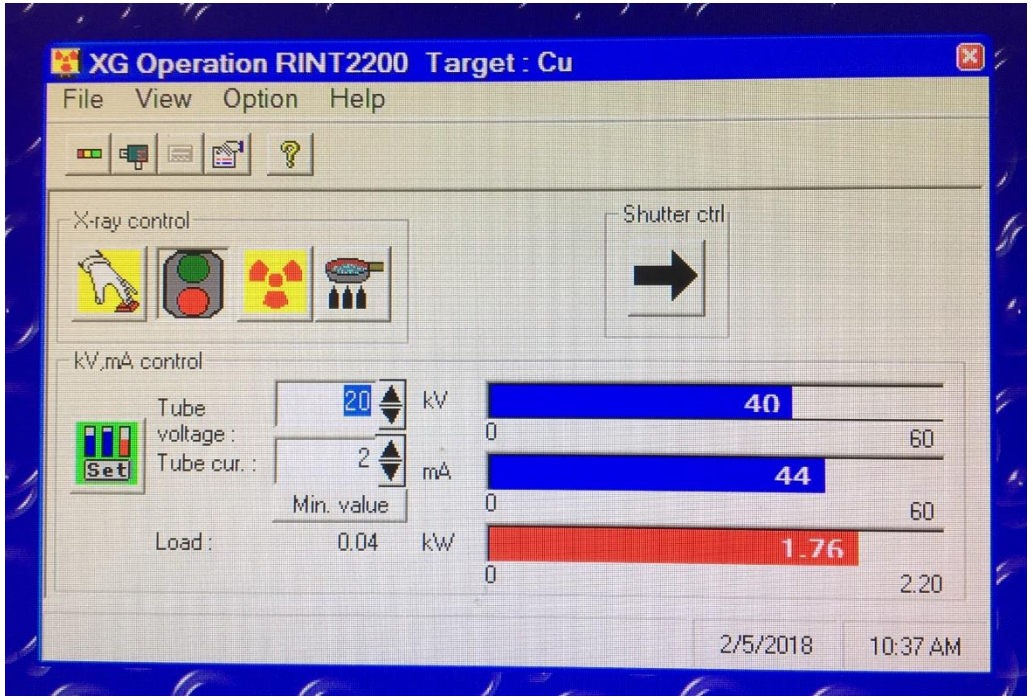
1) Login on the PC (pw: bworld).
2) Double-click XG Operation Icon (desktop).
3) Option → Control (Control Mode).
4) Option → Property → choose ‘morning’ → OK.
5) Click ‘Execute aging’ button to start aging.
6) The instrument will be ready in about 1 hour.



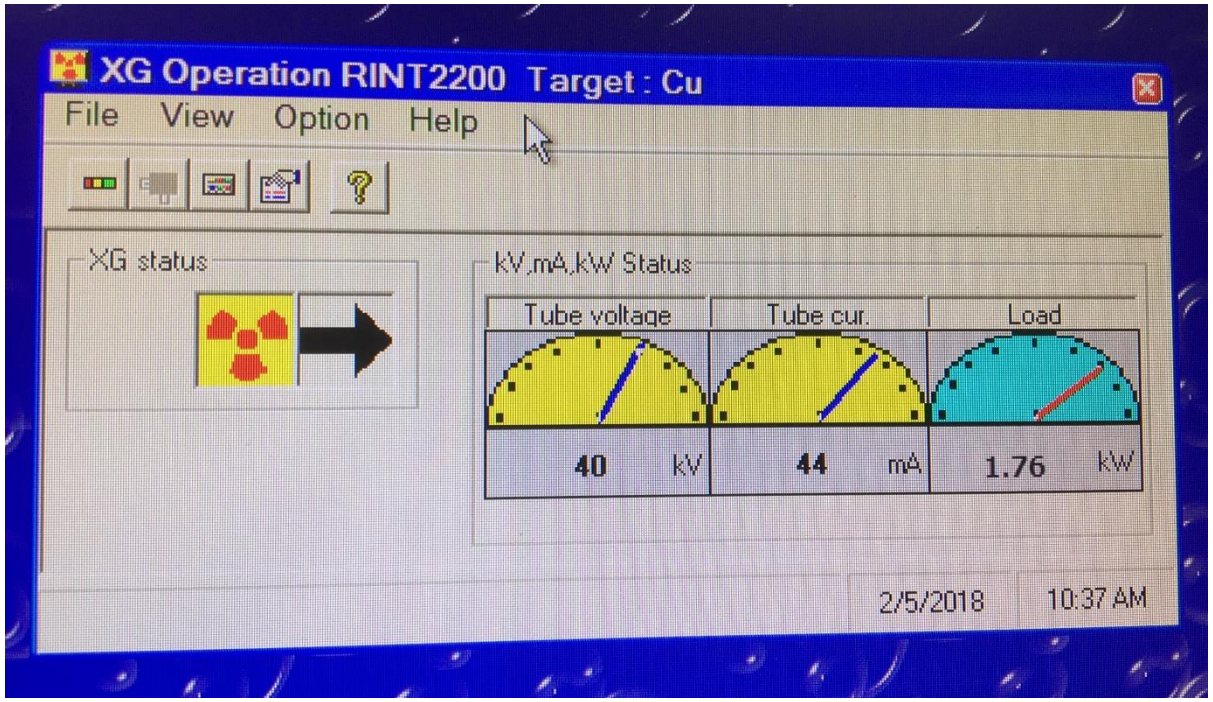


2. Aging the X-Ray Tube – Status

When aging finished:

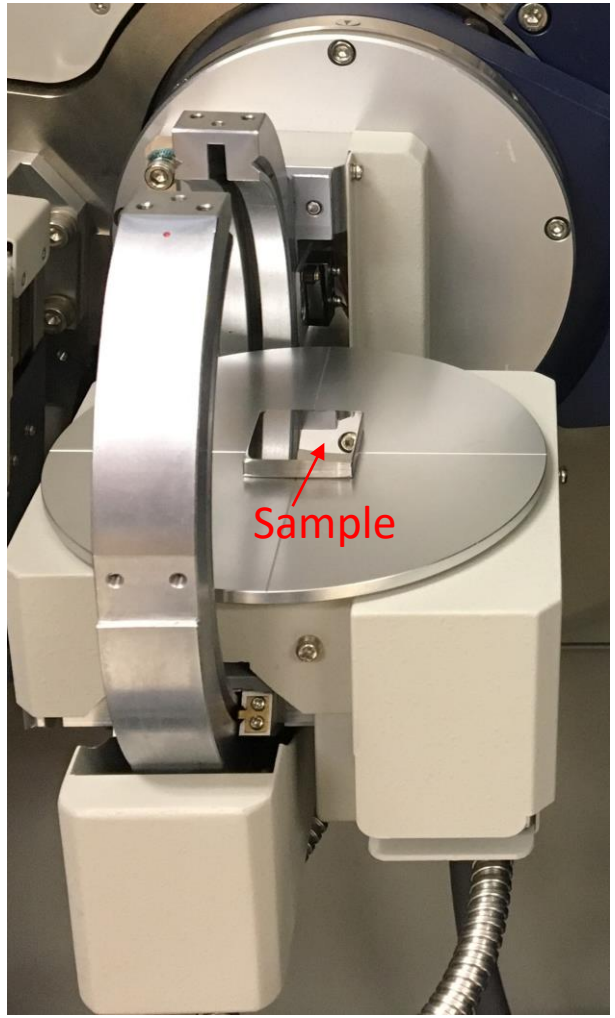


Option → Monitor (Monitor Mode):



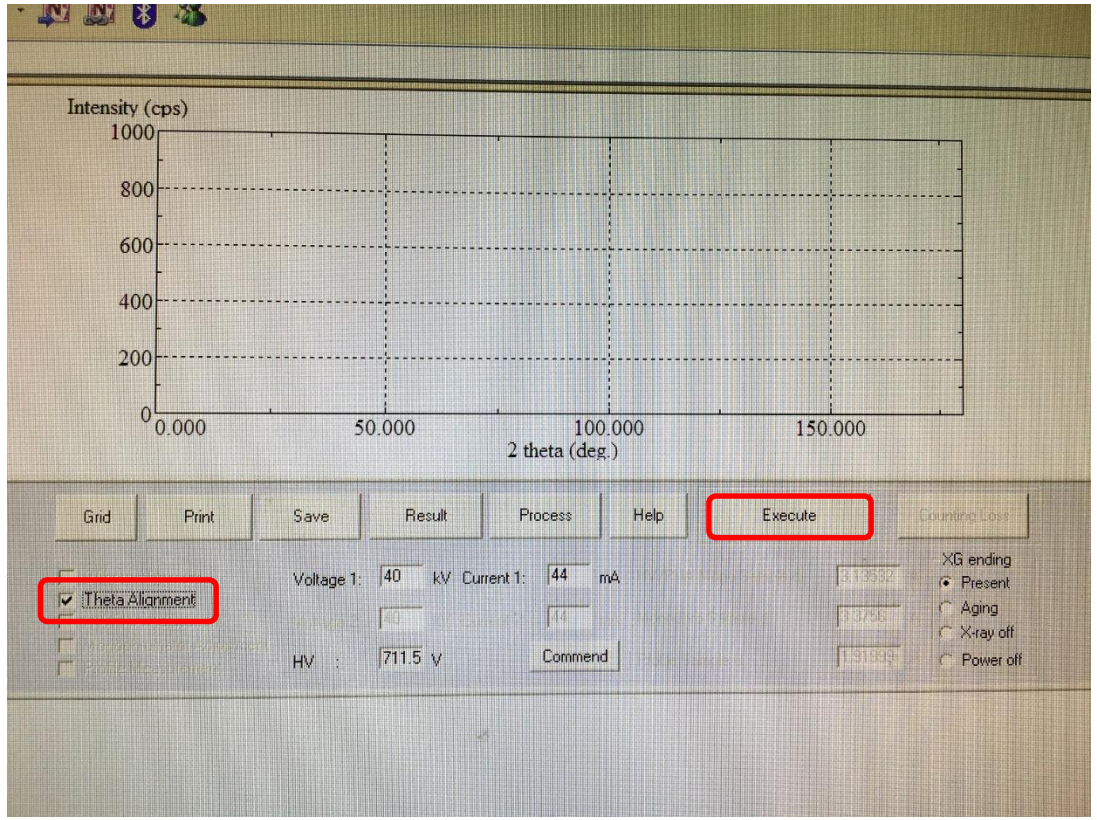
3. Prepare and Load Samples

1) Prepare the samples using Sample Plates or directly load your samples if they have special shapes (samples with rough face may affect the quality of the XRD pattern).
2) Press the DOOR LOCK button (flashing and beeping), then slide open the door.
3) Load the samples onto the Center of the MPU-4 Sample Holder. For samples with a thickness between 0-4mm , use the thicker Sample Holder; for 4-8mm samples, use the thin Sample Holder.
4) Slide close the door and Press the DOOR LOCK button again.

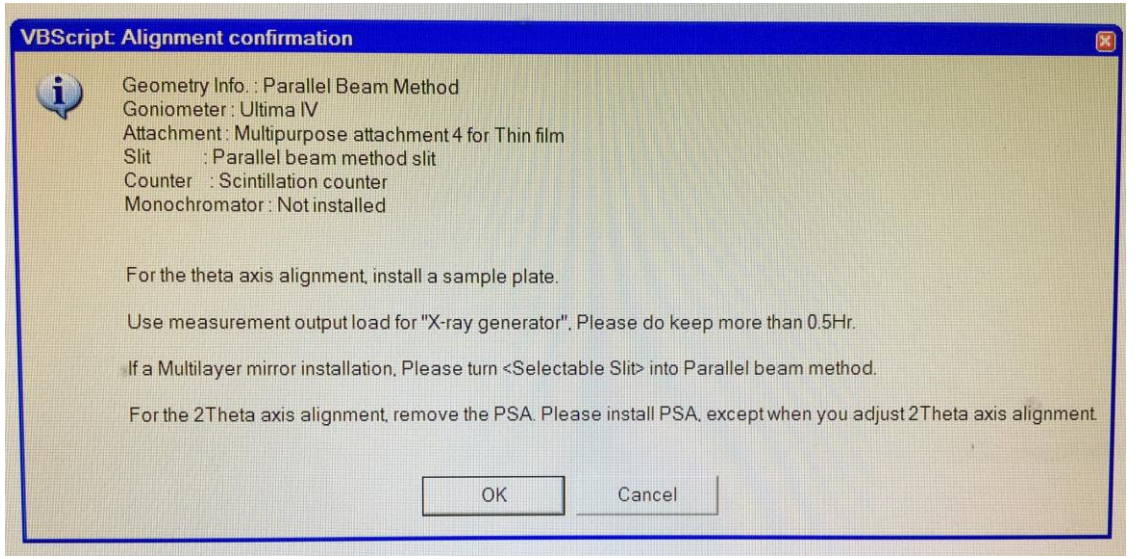


4. Automatic Alignment (1)

1) Double-click Automatic Alignment to start the software.
2) Tick the Theta Alignment and click Execute to start.

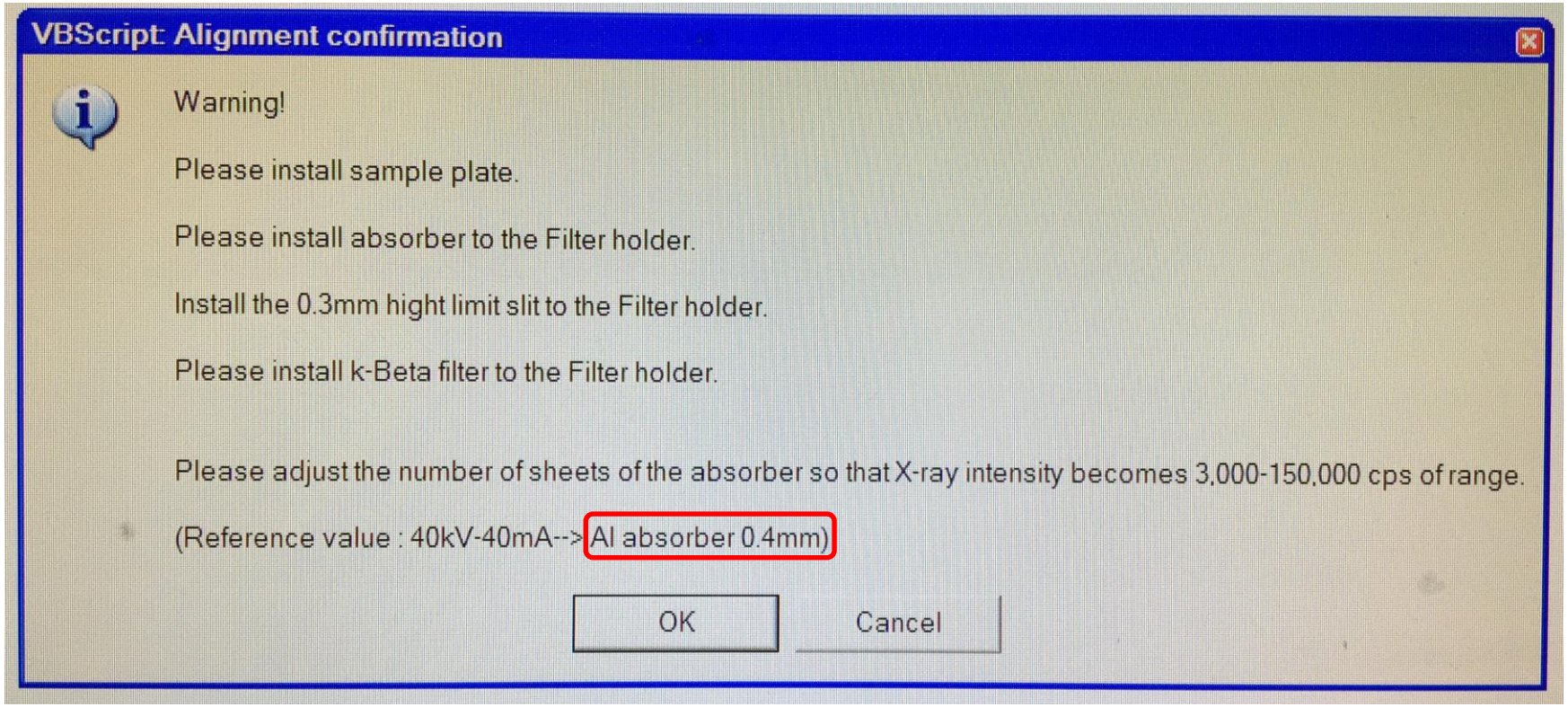


3) A pop-out window will ask you to install a sample, to make sure proper Slits is chosen, and to install PSA. Click OK to proceed.



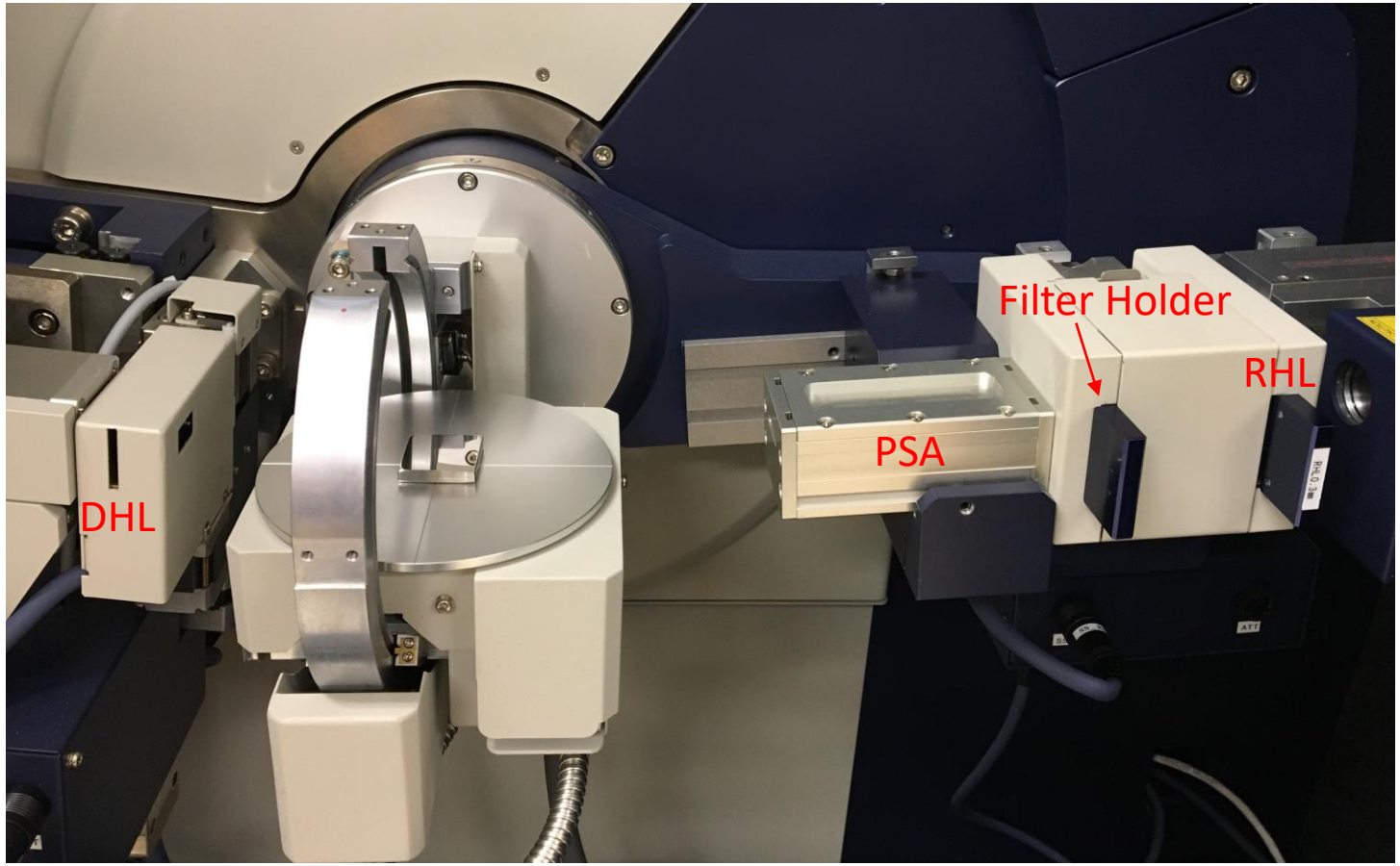
4. Automatic Alignment (2)

4) A second pop-out window will ask you to install sample, 0.3mm RHL, and absorber. Do NOT click OK!!!
5) Get two 0.2mm Al absorber and put them into the absorber holder. Open the door and load the absorber into the Filter Holder (refer to picture on next page).



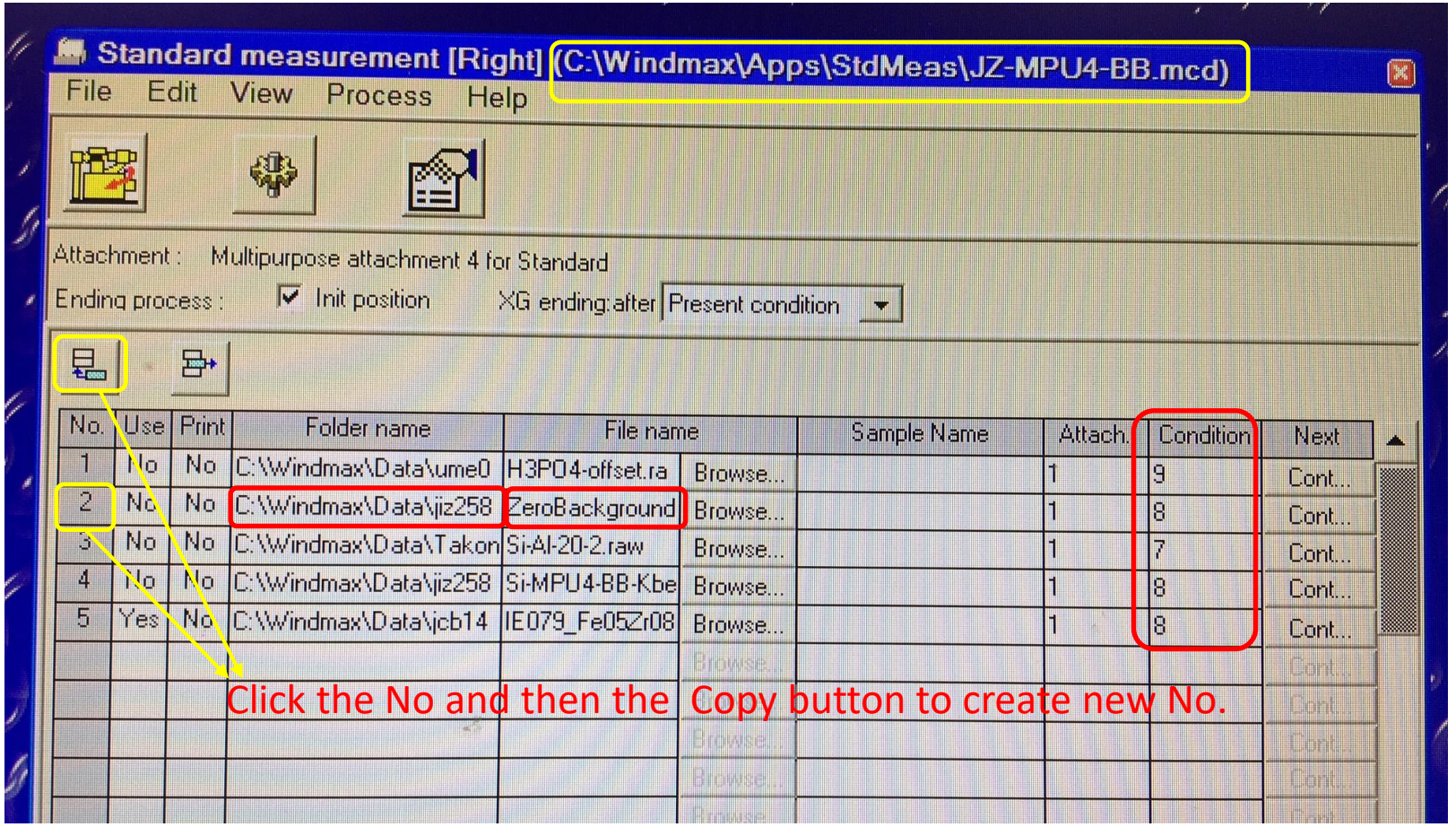
4. Automatic Alignment (3)

6) Remove the 10mm DHL slit. Close and Lock the door.
7) Click Ok to start the Automatic Alignment. It will take about 10 – 20 mins, depending on samples.
8) After completion, click Save to load the alignment results. Exit the software.
9) Important: remove the Al absorber and RHL, but keep the Cu K_β filter, and insert the 10mm DHL slit.



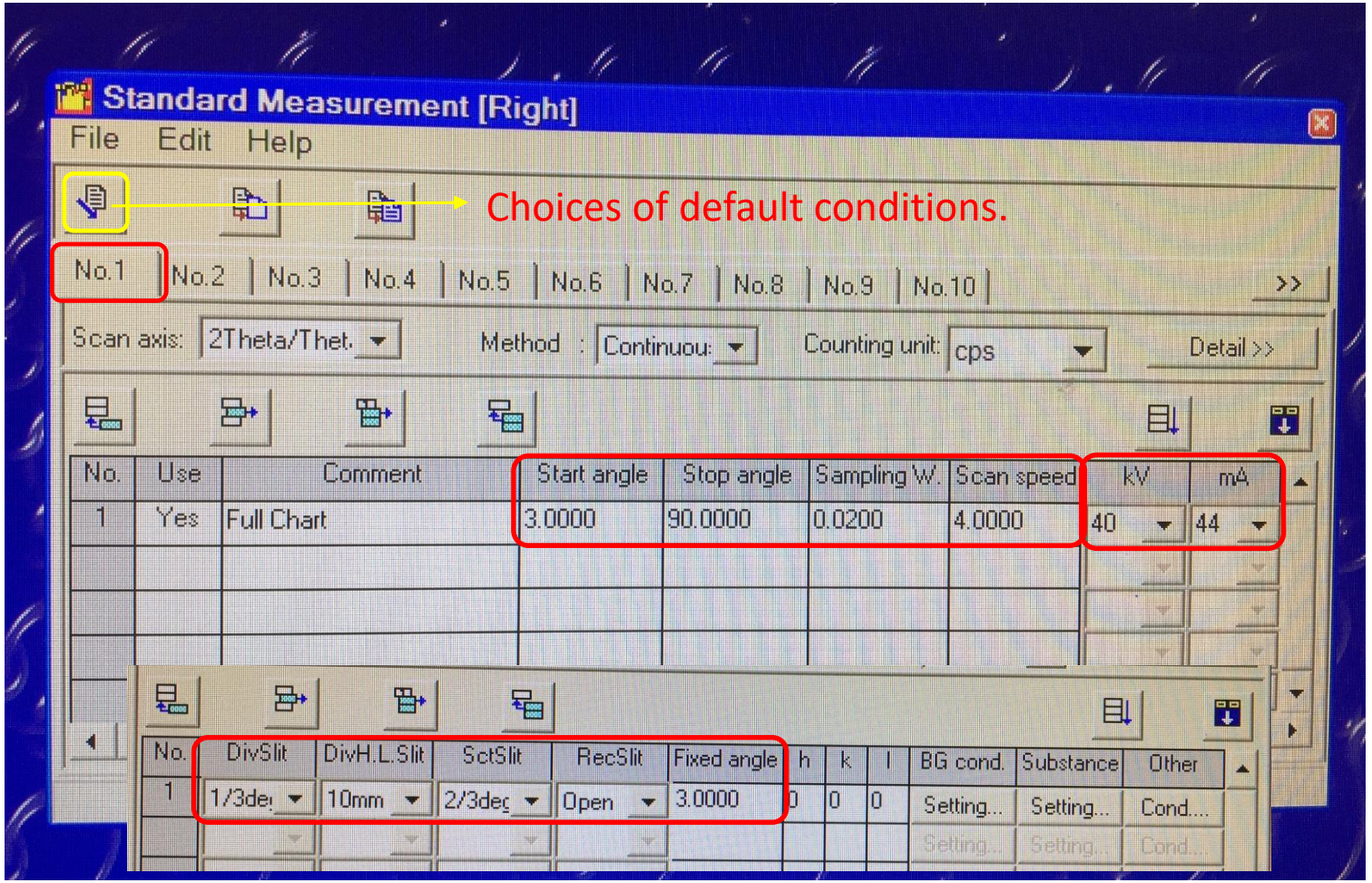
5. Set up the Experiments (1)

1) Double-click Standard Measurement.
2) File → Open to open the mcd file for your group.
3) Edit the Folder name and File name.
4) Double-click Condition # to open measure condition.



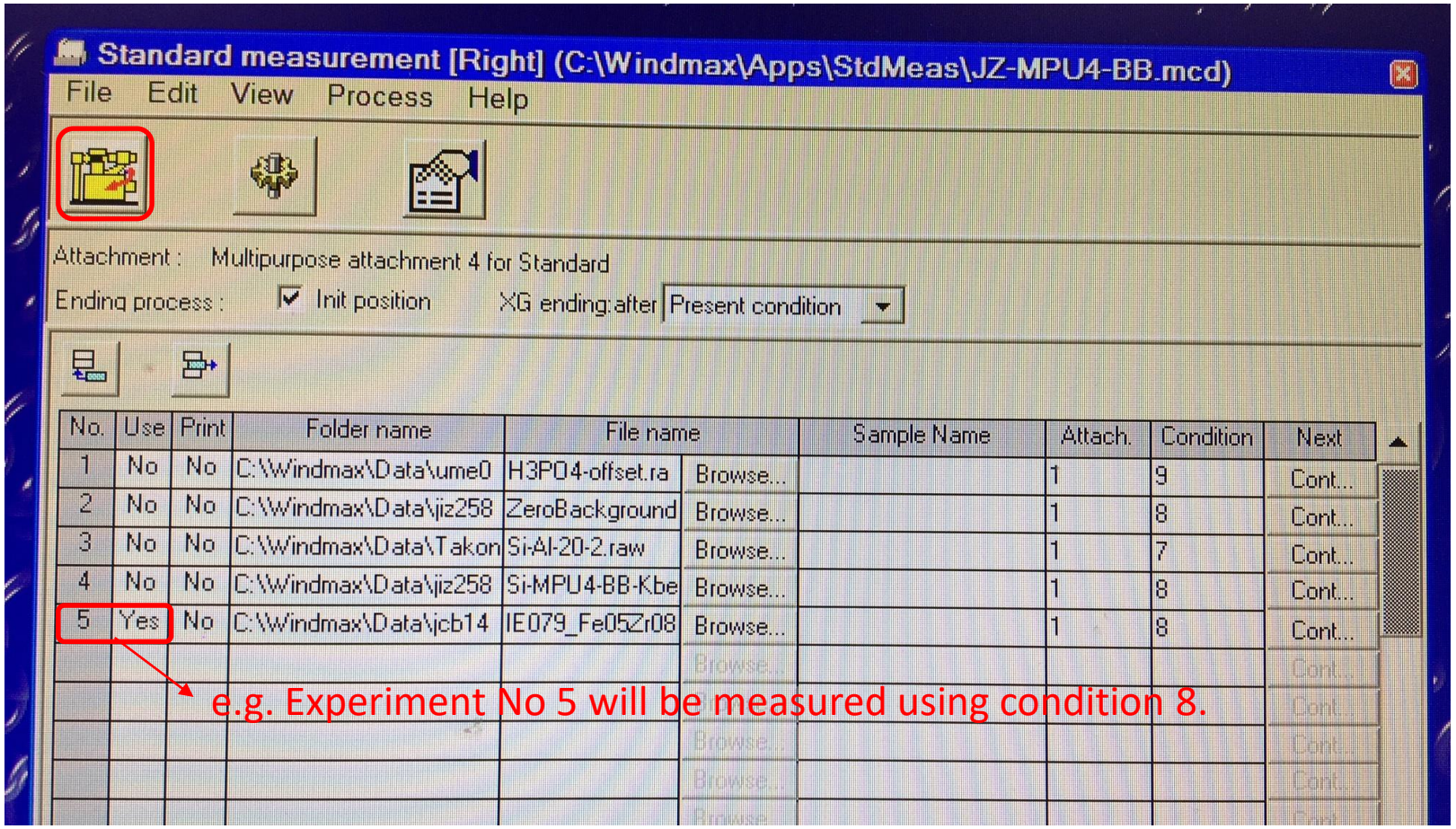
5. Set up the Experiments (2)

1) An example measuring condition shown below.
2) You may use the Default Condition button to create default measuring conditions for Inorganics or Organics.
3) Make sure power level at 40 kV and 44 mA. Slits and Grazing angle may need to be optimized.



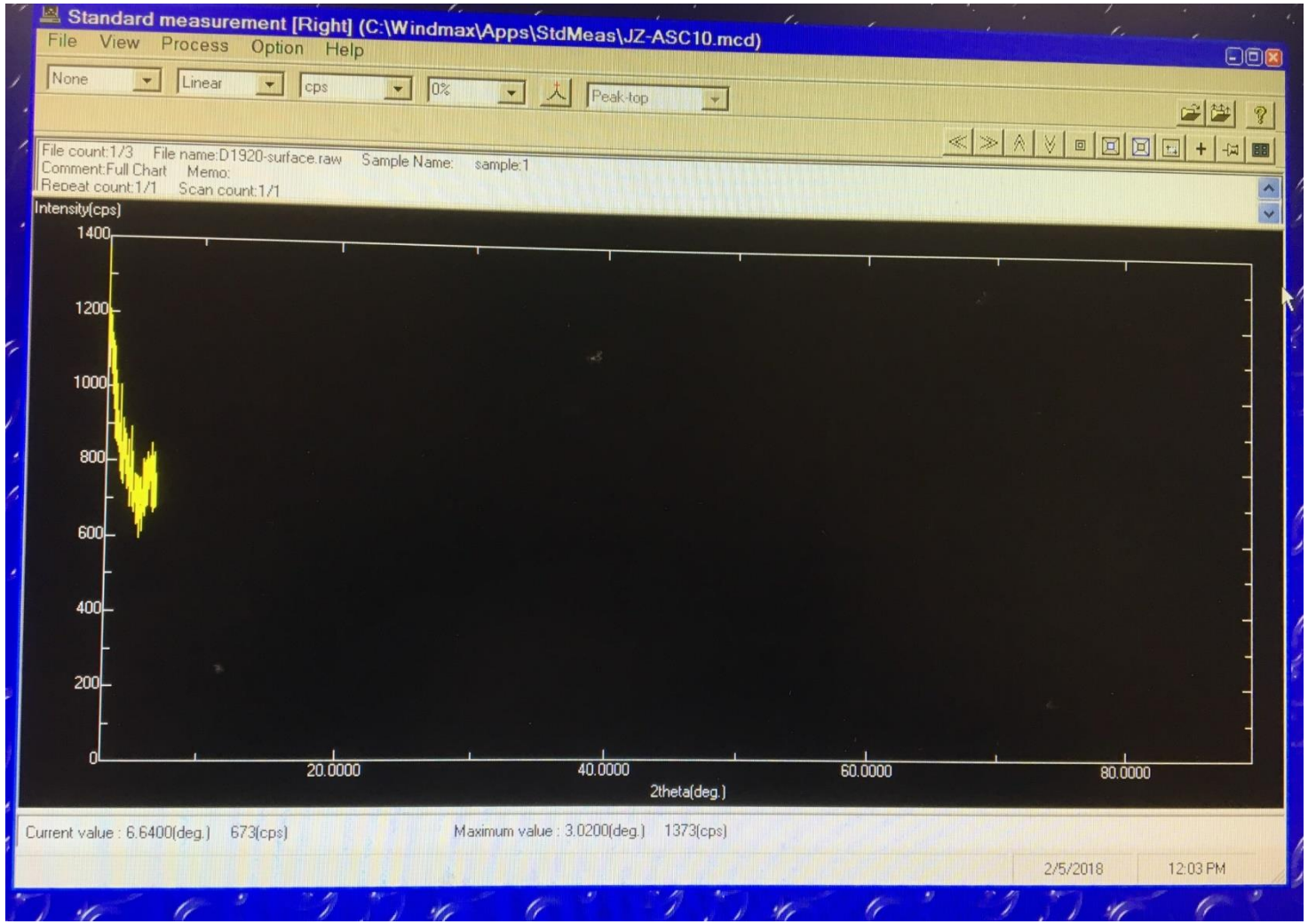
6. Start the Measurement

1) Click ‘No’ under ‘Use’ to change it to ‘Yes’.
2) Start the measurement by clicking the top left button.



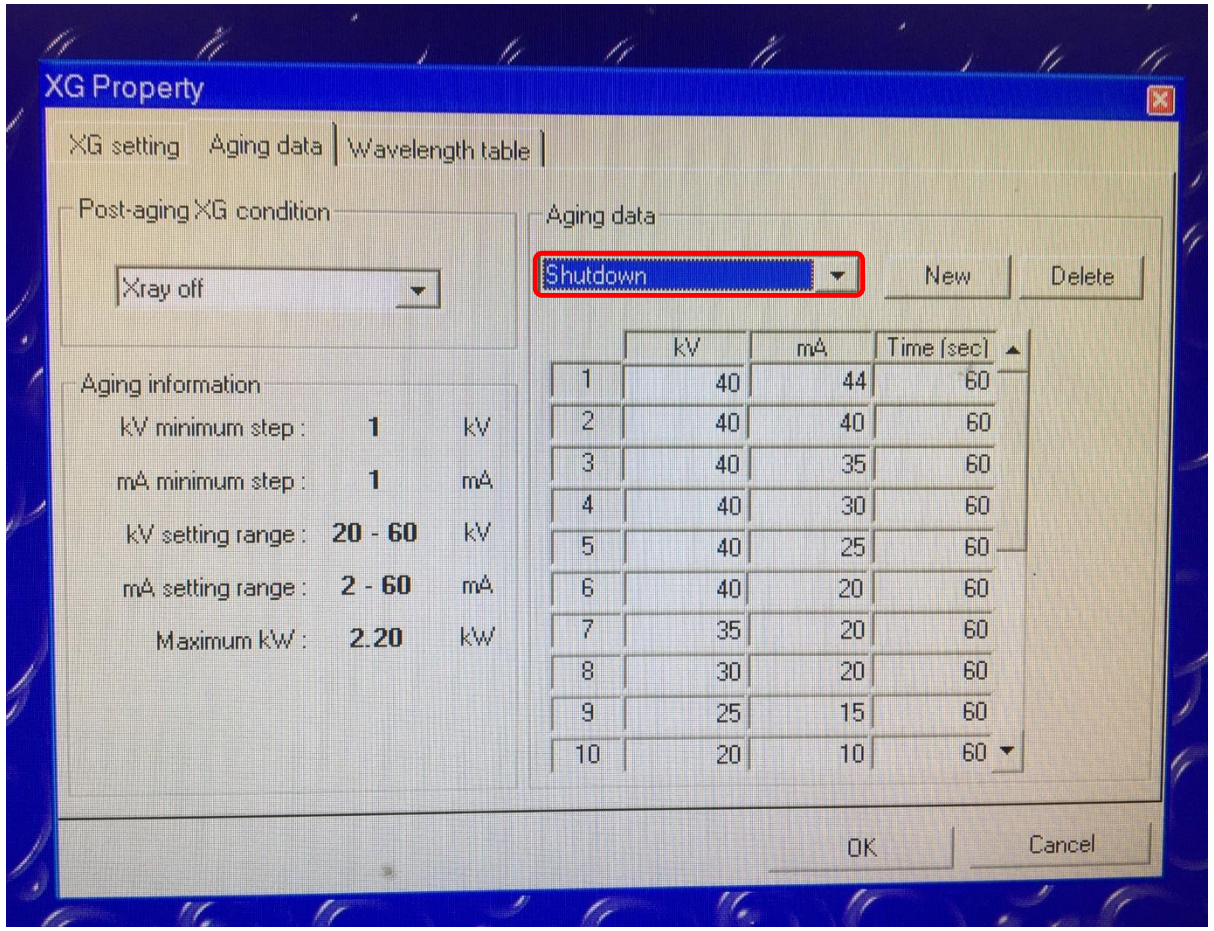
6. Start the Measurement – Status

1) The measuring diffraction pattern will be opened on a new window.
2) Do NOT attempt to open the cabinet door during the measurement.

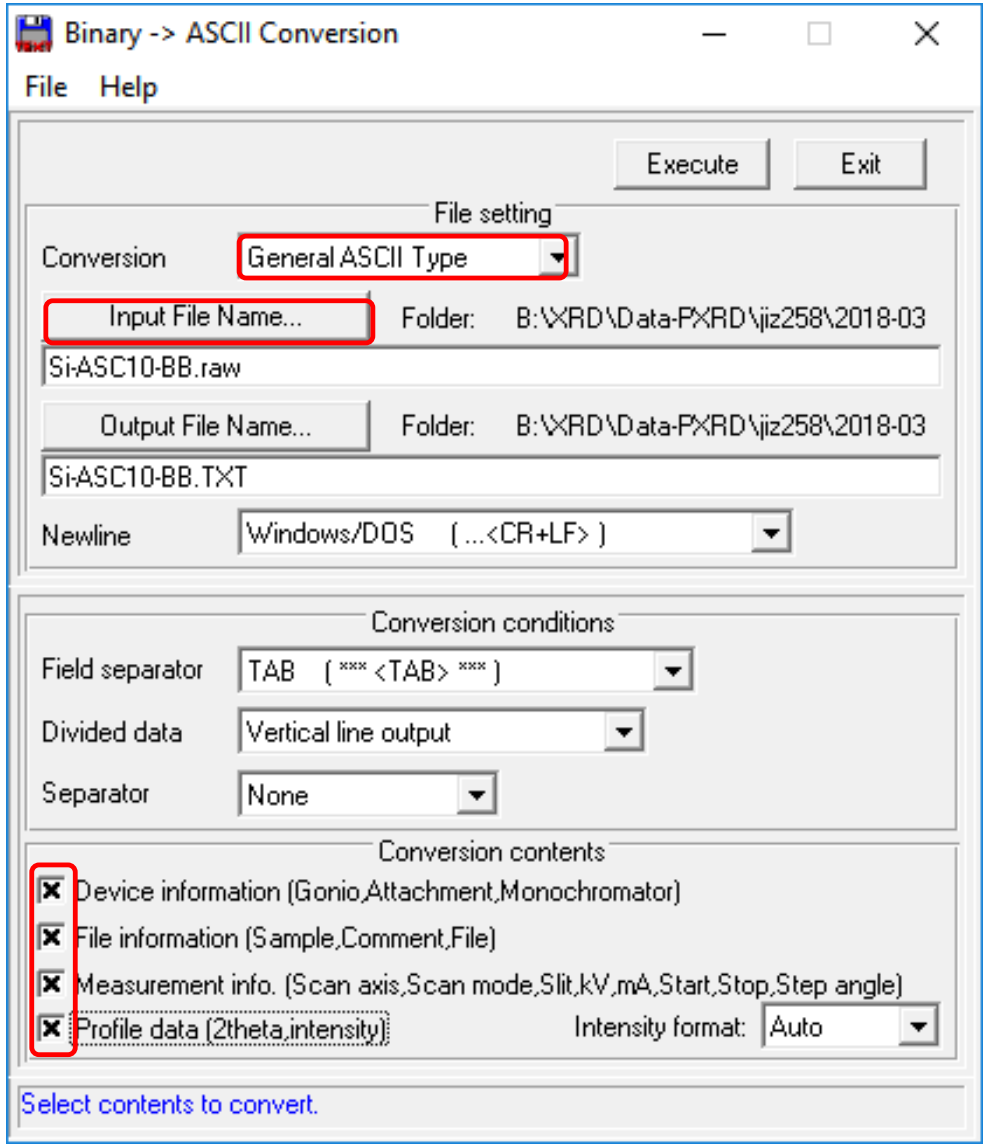


7. Turn Off the Diffractometer

1) Close all the windows, except the XG operation.
2) Go to the XG Operation window:
   - Option → Control
   - Option → Property → Choose ‘Shutdown’
   - Start aging. It will take ~10 mins.
   - Close the XG Operation window.
3) Turn the X-Ray enable key back to upright position.
4) Turn off the main power of the diffractometer.
5) Turn off the Haskris.



8. Convert RAW file to ASCII



1) Open Rigaku folder (on desktop)

   → Bianry–ASCII Conversion.

2) Choose General ASCII Type.
3) Open the Input RAW File(s).
4) Choose the contents to be

   included in the TXT file.

5) Click Execute to finish.