2θ – ω scan using PDS and XCelerator in Bragg-Brentano geometry (X'Pert_2 only)

I. Login

1. Start **Data Collector**.
2. Type your *UserName* and *Password*.
3. Select *Instrument – Connect*.
4. Choose Configuration **PDS + XCelerator**.
5. Click *OK*.

II. Hardware Setup

1. X-ray Tube is in *Line Focus*.
2. Goniometer Resolution set to "Normal 0.001 deg"
3. Incident Beam Optics – **PDS (Programmable Divergence Slit).**

Note: if you have to change incident beam optics please first turn Automatic attenuator to "Activate" status and then unplug attenuator cable.

   a. Insert Cu 0.1mm attenuator.
   b. If sample is smaller than 25 mm, insert correct size *Mask.*

4. Diffracted Beam Optics – **XCelerator**.
   a. Insert Soller Slit.
   b. Insert Ni filter.

III. Data Collector Software

1. Select the *Incident Beam* optics tab.
   a. Double click any item. Incident beam optics window will appear.
   b. Go through all tabs and select proper optic components.
   c. *Divergence Slit* – "Fixed" mode and slit set to $1/8^{o}$.
   d. Set *Automatic Attenuator Usage* to "Do not switch" and "Activated".
2. Select the *Diffracted Beam* optics tab.
   a. Double click any item. Diffracted beam optics window will appear.
   b. Go through all tabs and select proper optic components.
   c. *Detector* – "Receiving Slit" mode and active length set to 2.17mm.
3. Select *Instrument Settings* tab.
   a. Double click any item in the tree view to prompt another window.
   b. Press *X-ray* tab. Set generator power to 45 kV and 40 mA.

IV. Diffractometer Zero Alignment

1. In *Instruments Settings* check Z position. If it is larger than 5mm move it back to at least 5mm.
2. Move all other motors to zero positions.
3. From Menu Select *Measure – Manual Scan*.
4. From the *Scan Axis* drop down menu select 2Theta.
5. Enter *Range* 2^{o}, *Step Size* 0.01^{o}, and *Time per Step* 0.1sec. Then press *Start*.
6. After scan is finished, right click on mouse and select *Move Mode*. Move Scan Axis to the center of the mass of the peak.
7. In *User Settings – Sample Offsets* set current 2Theta position to zero.
8. Note the direct beam intensity.

V. Sample Mounting

1. Mount sample using scotch tape. If the sample is large, supplied clips can be used to hold the sample.
2. If in the *Instrument Settings* tab X = 0.0 and Y = 0.0, beam is positioned at the center of a sample stage (aluminum disk).

VI. Moving Sample into the Beam Position

1. Using supplied Micrometer.
   a. Mount micrometer onto the MRD cradle. Close the doors.
   b. In the *Instruments Settings* window, double click any item in the tree view to prompt another window.
   c. Move Z until micrometer reads (1.00±0.02) mm. This is the correct sample height.
2. Using direct beam.
   a. Note the direct beam intensity. In *Instruments Settings* move Z to higher values until intensity starts to drop.
   b. In *Manual Scan* window from the *Scan Axis* drop down menu select Z. Enter *Range* 2mm, *Step Size* 0.01mm, and *Time per Step* 0.2sec. Then press *Start*.
   c. After scan is finished, right click on mouse and select *Move Mode*. Move *Scan Axis* to the intensity value corresponding to ½ of the direct beam intensity.
   d. Z is aligned. Close shutter. Remove Cu 0.1mm attenuator from the **PDS**.

Note: sample height alignment using direct beam will work correctly if sample size is larger than the beam size in axial direction.

VII. Measurement

1. Select *Incident Beam* optics tab.
2. The *Divergence Slit* can be used in "Fixed" and "Automatic" modes. If "Fixed" mode is set, select the divergence of the slit to desired value from $1/32^{o}$ to 4^{o}. If "Automatic" mode is set, select a proper irradiated length of the sample.
3. Set *Automatic Attenuator Usage* to "Do not switch" and "Deactivated".
4. Click on *Diffracted Beam* optics tab.
5. Select *Detector* tab. Set detector mode to "Scanning" and active detector angle set to 1.595^{o}.
6. Simplest way to execute scan is to do a *Manual Scan*. It is a relative scan i.e. executed around current goniometer position with the range specified in *Manual Scan* window.
7. To do *2Theta – Omega* scan first move 2Theta and Omega to middle positions of the scan range.
8. In *Manual Scan* window select *Scan Axis* "2Theta – Omega" and appropriate *Range*, *Step Size* and *Time per Step*.
9. Click *Start*. When scan is completed, save it through *File – Save As* menu. Manual Scan will be lost if it is not saved.

Note: In symmetrical scan mode Omega axis is always ½ of the 2Theta axis.

VIII. Logging out

1. Close the shutter.
2. Switch *Automatic Attenuator* to "Do Not Switch" and "Activated".
3. Move all angles to zero positions and Z to 5mm.
4. Lower the power of the x-ray tube to 40kV and 10mA.

5. Close *Data Collector*.