High resolution rocking curve  
Step by Step / version 3.0 (09/12/16)

1) Log into the computer  
   Login: XpertPro MRD user  
   Pssw:  XpertPro

2) Double-Click on the Data collector software short cut icon

3) log into the program using your login and password  
   (if you do not have on contact Emmanuel Soignard; the account is generated during training)

4) Go to menu: Instrument / connect and select the detector needed for your experiment:

   Select Rocking Curve

   click NO to reset the sample offsets

   The computer is now connected to the diffractometer
Setting up the instrument parameters:

5) Check that the selected optical components are correct

Incident beam optics

Diffracted beam optics
6) User settings:
   Sample offset / clear all
   Fine calibration offset / clear all

7) Place all angles (2θ, offset, ω, Phi, Psi) and coordinates (X, Y) at zero place Z at 6 mm (if using the regular stage)

8) Set the X-ray power to 45 kV and 40mA
Sample alignment procedure:

**Manual Scan:** Measure / Manual Scan

9) calibrate 2θ procedure
   a) scan 2θ around 0°

b) set fine calibration offset
10) make sure the sample is in the beam and parallel to the beam
   c) set Z at 9.5 mm (if using a regular wafer thick sample on the regular holder)

   ![Instrument Settings](image)

   d) scan Z set half way point in intensity as the new Z

   Move to the half way height

   ![Prepare Manual Scan](image)

   e) scan ω set maximum as new ω

   Move to the maximum

   ![Prepare Manual Scan](image)

   f) repeat d) if ω was adjusted in step e)

   g) set sample offsets (user settings / sample offset) (Only set ω offset / Do not set Z offset)

   THE SURFACE OF THE SAMPLE IS NOW ALIGNED!!!
Aligning to the chosen diffraction peak:

11) Align your single crystal layer or substrate
   a) select the diffraction spot you will use to do
      your alignment (e.g. Si(111), Si(004), GaN(002), …)
      Select the unit cell and the h k l (space between each)
      the diffractometer will move the the correct
      2θ and ω position.

   b) scan ω move to maximum

      Move to the peak, if the peak is very sharp redo the scan with
      smaller range and step size.

   c) scan psi move to maximum

      Move to the peak.

   d) repeat b and c until neither is changing narrowing the range and step size as necessary.

   e) scan 2θ move to maximum

      2θ has a low resolution on the Rocking curve optics so only move 2θ if the position is off the peak.

      I the sample offset set:  \omega = \frac{1}{2} 2θ & \chi = 0

   f) scan ω with high resolution and small steps if you want to collect the rocking curve.

   g) save your spectrum. This is your rocking curve data.
12) Write down the 2θ position

13) In the Diffracted Beam Optics right click on Triple axis and select Activate

14) Clear the fine calibration offset

15) Set the Diffracted beam optics

16) Set 2θ and ω to 0 and set z at Z-2 mm.
17) Scan 2θ to set the detector offset

Move to the peak, it should be very close to zero

18) Set 2θ fine calibration offset

19) Instrument settings:

   a. set 2θ back to the noted position (Step 12)
   b. set ω at ½ of 2θ
   c. set Z at Z+2 mm (back to the aligned Z position)

   **We are now back in diffraction conditions**

20) Set the beam attenuator (Incident beam optics) to preset intensity

21) Scan 2θ

   Rescan 2θ with a narrower range if the peak is sharp. (range of 0.05° is not unusual)

22) In the sample offsets set ω = ½ of 2θ.

   **We are now all set to measure the ω/θ curve**
**Measuring the ω/2θ curve:**

**Option 1** move to the center of the range you would like to measure and do a manual scan. Then manually save the scan.

Instrument settings: set 2θ to the chosen position and \( \omega = \frac{1}{2} 2\theta \)

![Image of manual scan settings](image)

Typical time per step are between 0.1 and 1 s.

Typical step size varies with the thickness of the film, the thicker the film the smaller the step size.

Save date by clicking on the data window and going to file save as.

**Option 2** use an absolute scan program to do the measurement (measure /Program)

![Image of absolute scan settings](image)

The start and end angles depend on the diffraction peak position of your sample.

Typical time per step are between 0.1 and 1 s.

Typical step size varies with the thickness of the film, the thicker the film the smaller the step size.
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**Once done set the system to rest:**

1) Once done Place the stage to some convenient position for the next user

2) Set the X-ray current at 20 mA

3) Close the data collector.

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