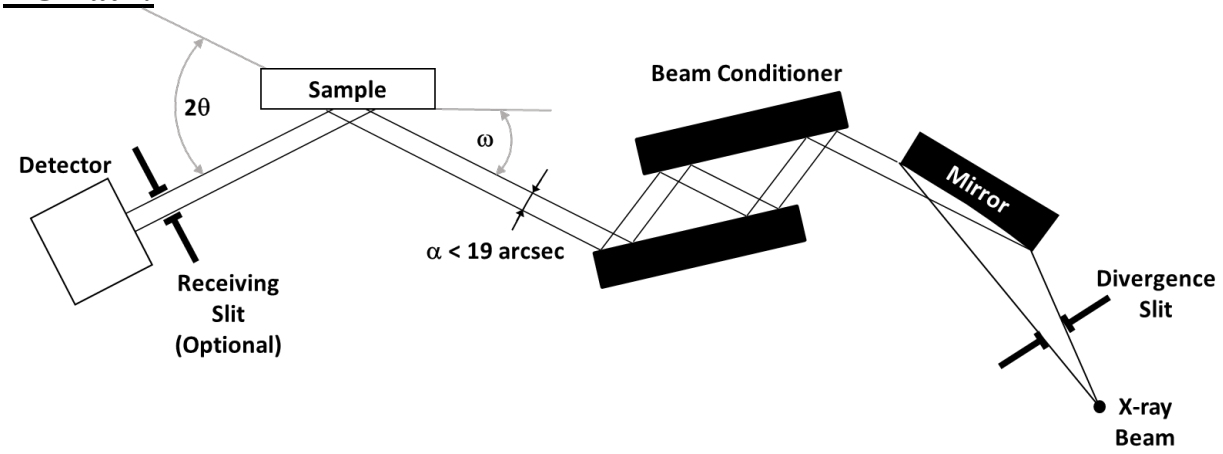
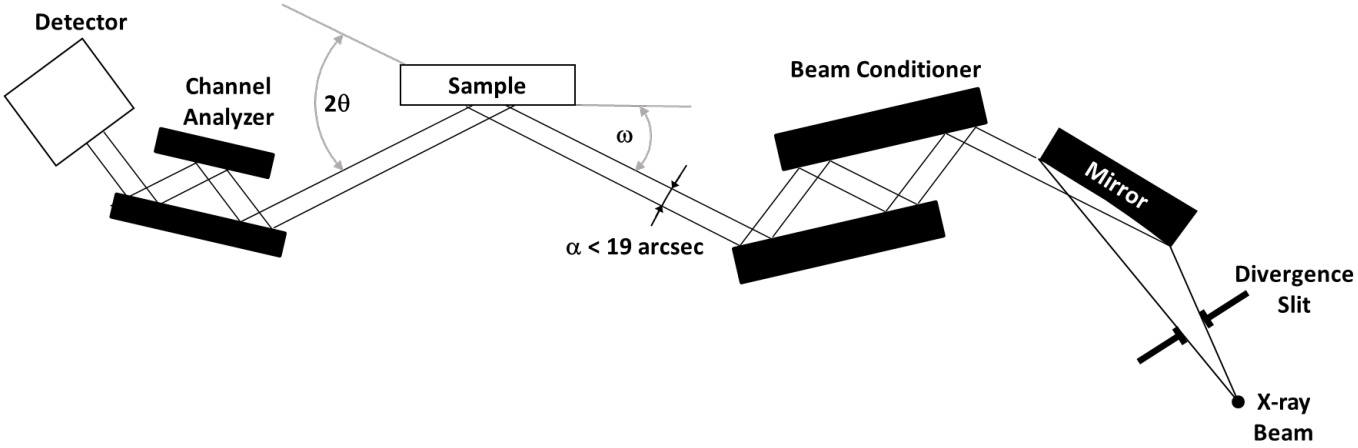
Rocking Curve and 2θ − ω scans using Hybrid Monochromator

RC Path:



TA Path:



I. Login

1. *Enable* instrument in **Badger**.
2. Start **Data Collector**.
3. Type your "User Name" and "Password".
4. Select *Instrument* → *Connect*.
5. Choose Configuration **Hybrid + RC/Triple**.
6. Click *OK*.

II. Hardware Setup

1. X-ray Tube is in "Line Focus".
2. Goniometer Resolution set to "High 0.0001 deg".
3. Incident Beam Optics – **Hybrid Monochromator**
   Note: *if you have to change incident beam optics please first turn Automatic attenuator to "Activate" status and then unplug attenuator cable.*
   a. Insert 1/32° **Divergence Slit** into **Hybrid Monochromator** optics.
   b. If sample's vertical dimension is smaller than 25 mm, insert correct size **Mask**.
4. Diffracted Beam Optics – **RC/TA (Rocking Curve/Triple Axis)**.

a. Depending on application start with **RC** or **TA** beam path by selecting it.

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:
      - *PreFIX Module* – select **Hybrid Monochromator**.
      - *Divergence Slit* – select 1/32° **Divergence Slit**.
      - *Anti-Scatter Slit* – select **None**.
      - *Mask* – select appropriate **Mask**.
      - *Beam Attenuator* – **Progr. Beam Attenuator**. For initial alignment set *Usage* = "Do not switch" and *Status* = "Activated". Make sure the *Description* = "Hybrid". If not, click *Select* and select Hybrid attenuator.
      - *Filter* – select **None**.
2. Select the **Diffracted Beam Optics** tab
   a. Double click any item. **Diffracted Beam Optics** window will appear.
   b. For Open Detector **RC** configuration select following optic components:
      - *PreFIX Module* – select **RC**.
      - *Anti-Scatter Slit* – select **None**.
      - *Receiving Slit* – select **None** or the size of the slit which is being used.
      - *Filter* – select **None**.
      - *Monochromator* – select **None**.
   c. For Triple Axis **TA** configuration select following optic components:
      - *PreFIX Module* – select **TA**.
      - *Anti-Scatter Slit* – select **None**.
      - *Receiving Slit* – select **None** or the size of the slit which is being used.
      - *Filter* – select **None**.
      - *Monochromator* – select **Triple Axis Monochromator**.
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. Sample Mounting

1. Mount sample using scotch tape. In most cases longer sample dimension should be vertical. If the sample is large, supplied clips can be used instead of scotch tape.
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).

V. Diffractometer Zero Alignment

1. Diffractometer Zero Alignment using **TA** optics.
   a. In **Instruments Settings** move to **Z** = 0.0 – 5.0 mm.
   Note: *sample should not interfere with the direct beam.*
   b. Move all other motors to zero positions.
   c. From Menu select *Measure* → *Manual Scan*.
   d. From the *Scan Axis* drop down menu select **2Theta**.
   e. Enter *Range* = 0.5°, *Step Size* = 0.002°, and *Time per Step* = 0.2sec. Then press *Start*.
   f. After scan is finished, move **2Theta** axis to a peak position using one of the two ways:

- Peak Mode. Right click on mouse and select *Peak Mode*. New window will appear showing the **2Theta** position of the peak. Click *Move To*. Close the window.
- Move Mode. Right click on mouse and select *Move Mode*. Move **2Theta** to the center of the mass of the peak.

g. Select *User Settings* → *Sample Offsets* and set current **2Theta** position to zero.

2. Diffractometer Zero Alignment using **RC** optics.
   a. Insert 1/16° **Divergence Slit** into **RC** optics.
   b. Perform steps described in "Diffractometer Zero Alignment using TA optics".

VI. Moving Sample into the Beam Position Using Direct Beam.

a. Note the direct beam intensity.
b. In **Instruments Settings** move **Z** to higher values until intensity starts to drop.
c. **Z** alignment can be performed using either optimization program or manually:
   - Using optimization program.
     a) Select *Measure* → *Program*. New window with user written programs will appear.
     b) From the *Measurement Type* select *Optimize Program*.
     c) Find proper program that says "Opt Z_Hybrid" and select it.
     d) Click *OK* and start the scan.
   - Manually.
     a) *Select Measure* → *Manual Scan*.
     b) In *Manual* **Scan** window from the *Scan Axis* drop down menu select **Z**.
     c) Enter *Range* = 2mm, *Step Size* = 0.01mm, and *Time per Step* = 0.2sec. Press *Start*.
     d) *After* scan is finished, right click on mouse and select *Move Mode*.
     e) Move **Z** to the intensity value corresponding to ½ of the direct beam intensity.

VII. Aligning diffractometer on the known diffraction peak. Si(001) example.

1. Switch **Diffracted Beam Optics** from **TA** path into **RC** path.
2. Select **Instrument Settings** tab
3. Double click any item in the tree view to prompt another window.
4. Click **Positions** tab.
5. In *Unit Cells* select *Si_001.*
6. In *h k l* field enter "0 0 4".
7. Click *OK*. Diffractometer moves to Si(004) peak position.
8. Select *Measure* → *Manual Scan.*
9. Start with **Omega** Scan. In *Manual Scan* window from the *Scan Axis* drop down menu select *Omega*. Enter *Range* 2°, *Step Size* 0.01°, and *Time per Step* 0.2sec. Then press *Start.*
10. After scan is completed. Si(004) diffraction peak should be visible. Right click on mouse. Using *Peak Mode* or *Move Mode* move **Omega** to the center of the mass of the peak.
11. Next perform **Chi** Scan. In *Manual Scan* window from the *Scan Axis* drop down menu select **Chi**. Enter *Range* 6°, *Step Size* 0.03°, and *Time per Step* 0.2sec. Then press *Start.*
12. Right click on mouse and select *Move Mode*. Move *Scan Axis* to the center of the mass of the peak.
13. Repeat *Omega* Scan. In *Manual Scan* window from the *Scan Axis* drop down menu select *Omega*. Enter *Range* 0.2°, *Step Size* 0.0005°, and *Time per Step* 0.2sec. Then press *Start.*

14. Move **Omega** to the center of the mass of the peak.
15. Select *User Settings – Sample Offsets*. Enter in **Omega** and **Chi** fields theoretical Si(004) values. Click *OK.*

VIII. Measurement – Symmetrical Scan using RC beam path.

1. In the **Incident Beam Optics** tab set *Beam Attenuator Usage* – "Preset Intensity" with *Activate Level* = 500,000 and *Deactivate Level* = 450,000.
2. 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.
3. To perform *2Theta-Omega* scan first move *Scan Axes* **2Theta** and **Omega** to middle positions of the scan range. For a symmetrical scan always **Omega** = (**2Theta**)/2.
4. In **Manual Scan** window select *Scan Axis* **2Theta-Omega** and appropriate *Range*, *Step Size* and *Time per Step*. Click *Start.*
5. When scan is completed, save it through *File* → *Save As* menu. Manual Scan will be lost if it is not saved.
6. To do *Omega* scan on the diffraction peak, first move **2Theta** and **Omega** to the diffraction peak position.
7. In *Manual Scan* window select *Scan Axis* **Omega** and appropriate *Range*, *Step Size* and *Time per Step*. Click *Start*.
8. When scan is completed, save it through *File – Save As* menu. Manual Scan will be lost if it is not saved.

IX. Measurement – Symmetrical Scan using TA beam path.

1. After alignment on Si(004) peak is completed using RC beam path (part VII), switch **Diffracted Beam Optics** to TA beam path.
2. In *Manual Scan* window from the *Scan Axis* drop down menu select *2Theta*. Enter *Range* 0.2º, *Step Size* 0.0005º, and *Time per Step* 0.1sec. Then press *Start.*
3. Move **2Theta** to the center of the mass of the peak.
4. Select *User Settings – Sample Offsets*. Enter in **2Theta** field theoretical Si(004) 2Theta value. Click *OK.*
5. In the **Incident Beam Optics** tab set *Beam Attenuator Usage* – "Preset Intensity" with *Activate Level* = 500,000 and *Deactivate Level* = 450,000.
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 perform *2Theta-Omega* scan first move *Scan Axes* **2Theta** and **Omega** to middle positions of the scan range. For a symmetrical scan always **Omega** = (**2Theta**)/2.
8. In **Manual Scan** window select *Scan Axis* **2Theta-Omega** and appropriate *Range*, *Step Size* and *Time per Step*. Click *Start.*
9. When scan is completed, save it through *File* → *Save As* menu. Manual Scan will be lost if it is not saved.
10. To do *Omega* scan on the diffraction peak, first move **2Theta** and **Omega** to the diffraction peak position.
11. In *Manual Scan* window select *Scan Axis* **Omega** and appropriate *Range*, *Step Size* and *Time per Step*. Click *Start*.
12. When scan is completed, save it through *File – Save As* menu. Manual Scan will be lost if it is not saved

X. Logging out

1. Close the shutter.
2. Beam *Attenuator – Usage* = "Do Not Switch" and *Status* = "Activated".
3. Move all angles to zero positions and **Z** to 5 mm.

4. Lower the power of the x-ray tube to 40 kV and 20 mA.
5. Close **Data Collector**.
6. *Disable* instrument in **Badger**.

For more advanced x-ray diffraction measurement techniques such as asymmetrical scans and reciprocal space maps please contact X-ray Lab manager.