

Rigaku SmartLab

Introduction

The Rigaku SmartLab is a multipurpose XRD which can switch between focusing (Bragg-Brentano, BB) and parallel beam (PB) geometries. In addition, it has an in-plane arm ($2\theta/\omega$ axis) which rotates the counter around the axis perpendicular to the axis of 2θ rotation, effectively pulling the counter to the front allowing both in-plane and out-of-plane grazing incident diffraction (GID) for thin films. The system also has both source and detector monochromators.

The unit is fully interlocked for X-ray safety. The door cannot be opened when the X-ray shutter is open. *Do not force the door.* To open, press the door button and wait for it to respond.

Options and Capabilities

The system can do

- *Standard $2\theta/\theta$ diffraction for phase ID.* The tilt stage has tilt in x and y (R_x and R_y) that allow for a $\pm 5^\circ$ tilt in these values to correct for any offset. The D/tex detector can be used to collect faster scans.
- *Rocking curves for crystal quality analysis.* This experiment rocks ω around a fixed 2θ . The narrower the reflection the higher the crystal quality. Once ω is optimized 2θ is optimized. Finally the ω scan is repeated.
- *Grazing incidence diffraction (GID) for thin film analysis.* GID allows for a larger sample interaction volume. The incident beam is set a fixed critical angle (typically 0.3 - 0.6°) and the 2θ axis is scanned. In-plane GID is performed by rotating the $2\theta/\omega$ axis to the appropriate angle and scanning 2θ .
- *Pole figure analysis and ϕ scans.* These data are collected by varying ϕ and $2\theta/\omega$ around a fixed $2\theta/\omega$ reflection to determine texture and/or epitaxy.

A *monochromator* is on the source side and an *analyzer* is on the detector side. These options require removal of detectors. Do not perform this task unless properly trained and authorized.

- *Graphite analyzer* - The graphite analyzer is used for reducing background radiation from fluorescing samples. This option is installed in front of the SC detector. The selector on the analyzer must be turned to “curved crystal” for BB mode and to “flat” for PB mode.
- *Ge (220)x4 4-bounce monochromator* - This option is used to obtain a highly monochromatic beam before it interacts with the sample. However, $>90\%$ of the beam intensity is lost as result. (Please do not install this option unless properly trained and authorized)
- *Ge (220)x2 2-bounce analyzer* - This option is used to obtain a highly monochromatic diffracted beam after it interacts with the sample. Again, some beam intensity is lost. As a result it is suggested that the user not use this option unless necessary (i.e. analysis of highly epitaxial thin films).

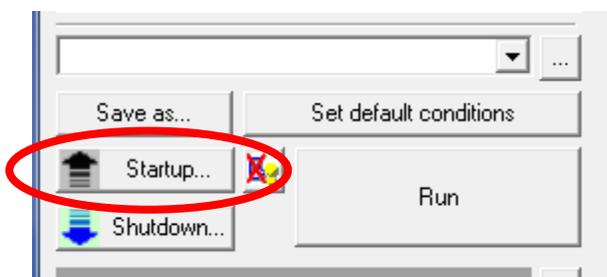
- *k_β filter and slits* - The k_{β} filter helps to eliminate the k_{β} peaks by passing the diffracted beam through a thin Ni foil. The incident slits limit the size of the incident beam and thus prevent large background signals. The parallel slits help limit beam divergence (either in x or y on both the incident and receiving optics).

Standby Condition

- X-ray generator at **20 kV** and **2 mA**.
- Goniometer arms at 0 degrees.
- Log out of Guidance software.

Startup

- Log in and start the SmartLab Guidance software if necessary. Wait for the instrument to calibrate if necessary.
- Select **Startup** and bring up the X-ray Generator. Choose the appropriate warm-up time (takes about 15 minutes)



Sample Loading

- Sample Loading

Basic Scans

Most standard scans for phase ID are $\theta/2\theta$; $2\theta/\omega$ scans are more useful for obtaining information on lattice planes parallel to the sample surface (e.g. thin films on a substrate and/or buffer layers). Note that $2\theta/\omega$ are only useful if performing a rocking curve on a substrate or sample peak.

Choose the appropriate measurement package.

- **Tasks | Package Measurement | <package>** (and choose the appropriate package).
- **General (Bragg Brentano focusing)** works for basic measurements (with additional options).

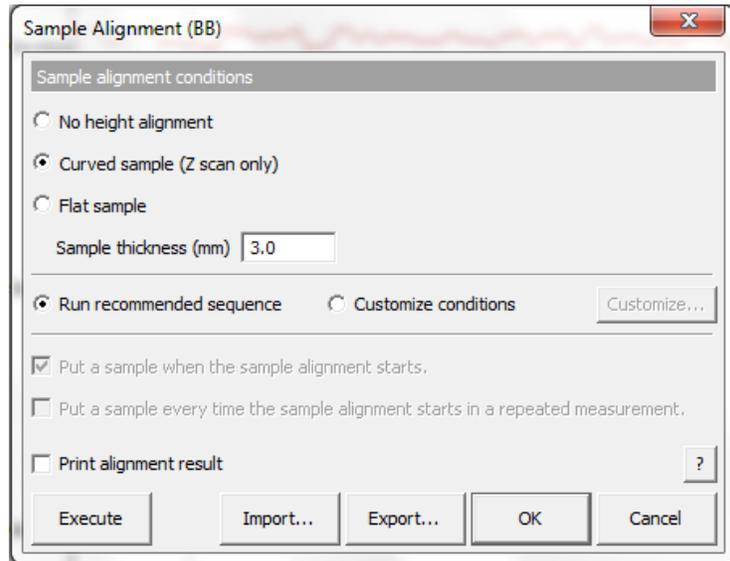
Optics Alignment: (if necessary; takes 5-15 minutes)

- Select **Optics Alignment** and verify that *Current Attribute* is correct.

- If there is any doubt about the alignment, you may select the correct Optics alignment name for your analysis (i.e. @BB Focusing) and check “Change optics without alignment” that uses the latest saved alignment settings for that measurement package.
- Follow the instructions and change the centering slit when prompted.

Sample Alignment: (takes ~5 minutes for full alignment)

- Choose the appropriate sample holder and mount your sample.
- Select **Sample Alignment**. Required only for first sample IF your samples are mounted identically. If sample thickness or geometry may vary, run for each sample.
- Choose approximate sample thickness (including holder, typically 2-3 mm), choose the recommended sequence and **Execute**.
- Note: For most samples, check the box for **Curved Sample (Z scan only)** This adjusts only the “Z” and skips the sample rocking part of the alignment. Full sample alignment, “Flat Sample” alignment takes about 5 minutes.
- The ? icon will bring up the help documentation.



Data Collection

- From **Tasks | General Measurement**, select “**General (Bragg-Brentano Focusing)**”

Sample Measurement Data: File name is the full path to the where the file will be saved. Save all data to the “M:” drive. RAS is the file extension for the SmartLab data; files are also saved automatically in Rigaku’s ASCII (ASC) and RAW formats. Sample name and Memo should contain some information about the data, minimally duplicating the file name. Sample name has a 30 character size limit for most displays of data.

Manual exchange slit conditions: Includes options for: Soller/PSC Slit (Incident); Incident Slit; PSA adapter; and Soller Slits (Diffracted).

Measurement Conditions: For a single sample, choose one line to configure and check the “Exec.” Box. Clicking on the “?” box pulls up the help for the measurement settings dialog.

- Check the box for “Drive the 4 axes to the current zero positions after the measurement is completed.”
- **Single measurement** - set parameters desired, click on the **Execute** button to start collecting data. The data file (RAS) will be created and data collection will begin. After a few seconds a real-time display of your data will appear.
- **Multiple measurements** - repeat the procedure, changing filenames and data collection parameters as appropriate for your samples. Failure to change the filename will cause previously collected data to be over written!
- After data collection is completed, you may close the general measurement window by clicking **Cancel**. DO NOT CHOOSE OK. It will overwrite your data file.
- If you wish to reuse this setup, you may **Export** it prior to closing and save it. To reload it later, **Import** it from the General measurement window. All previous settings (including filenames) are saved in the “Export” file so these need appropriate editing when reused to avoid overwriting existing data files.
- Note: The SmartLab Guidance software does not incrementally save data to disc as it is collected, but must complete the data collection of the previous step before files are saved. For this reason, it is important to not interrupt the data collection until the system has finished the job. When done, you will have three data files. These will be: *filename.RAS, filename_Theta_2-Theta.RAW, and filename_Theta_2-Theta.ASC.*

General Measurement (BB)

Save measurement data

File name

Sample name

Memo

Manual exchange slit conditions

Soller/PSC (deg) IS L (mm) PSA (deg) Soller (deg) Monochromatization Data acquisition mode

5.0 10.0 Open 5.0 K beta filter method Diffracted beam monochromator method OD

Read current slits

Measurement conditions

Exec.	Scan axis	Mode	Range	Start (deg)	Stop (deg)	Step (deg)	Speed (Duration time)	IS (deg)	RS1 (deg)	RS2 (mm)	Attenuator	Comment	Options	Voltage (kV)	Current (mA)
1	<input checked="" type="checkbox"/> Theta/2-Theta	Continuous	Absolute	10.0000	90.0000	0.0200	1.0000	2/3	2/3	0.300	Open		Set...	40	44
2	<input type="checkbox"/> Theta/2-Theta	Continuous	Absolute	3.0000	90.0000	0.0200	4.0000	2/3	2/3	0.300	Open		Set...	40	30
3	<input type="checkbox"/> Theta/2-Theta	Continuous	Absolute	3.0000	90.0000	0.0200	4.0000	2/3	2/3	0.300	Open		Set...	40	30
4	<input type="checkbox"/> Theta/2-Theta	Continuous	Absolute	3.0000	90.0000	0.0200	4.0000	2/3	2/3	0.300	Open		Set...	40	30
5	<input type="checkbox"/> Theta/2-Theta	Continuous	Absolute	3.0000	90.0000	0.0200	4.0000	2/3	2/3	0.300	Open		Set...	40	30
6	<input type="checkbox"/> Theta/2-Theta	Continuous	Absolute	3.0000	90.0000	0.0200	4.0000	2/3	2/3	0.300	Open		Set...	40	30
7	<input type="checkbox"/> Theta/2-Theta	Continuous	Absolute	3.0000	90.0000	0.0200	4.0000	2/3	2/3	0.300	Open		Set...	40	30
8	<input type="checkbox"/> Theta/2-Theta	Continuous	Absolute	3.0000	90.0000	0.0200	4.0000	2/3	2/3	0.300	Open		Set...	40	30
9	<input type="checkbox"/> Theta/2-Theta	Continuous	Absolute	3.0000	90.0000	0.0200	4.0000	2/3	2/3	0.300	Open		Set...	40	30
10	<input type="checkbox"/> Theta/2-Theta	Continuous	Absolute	3.0000	90.0000	0.0200	4.0000	2/3	2/3	0.300	Open		Set...	40	30

Drive the 4 axes to the current zero positions after the measurement completed.

Calculated scan duration : 01:20:00

Execute Import... Export... OK Cancel

Fluorescing samples

To reduce sample fluorescence you will need to use a monochromator (DBM) on the detector side of the sample. The DBM unit attaches in the detector bracket. Note that the DBM reduces count rates by $\sim 1/2$ relative to the standard scintillation (SC) detector.

- From **Tasks | General**, select “**General (Bragg-Brentano Focusing)**”
- Run **Optics Alignment** and **Sample Alignments** as needed
- Select **General Measurement (BB)**. A window (see above) will appear through which data collection is setup and executed. Select the “**Diffracted Beam Monochromator method**” check box and choose your file name and scan parameters.
- Check the box for “Drive the 4 axes to the current zero positions after the measurement is completed.”
- Verify that the detector bracket is at 351.5 mm and that the DBM is set to “Bent” mode.
- Remove the standard SC detector and mount the DBM unit on the detector bracket. Remove the mounting bracket from the SC detector and mount the detector unit onto the DBM.

- Select **Execute** to start the scan.
- When finished remove the monochromator and remount the SC detector.

Thin Film Conventional Diffraction

The X-ray source is set up as a parallel beam, Omega is fixed at a small angle, and 2Theta is scanned to collect the diffraction pattern.

- Select **General | medium resolution PB**
- Insert centering slit and PB slit
- Perform **Optics Alignment**
- Set the sample height slit to match your sample.
- Perform **Sample Alignment**
- Select **General Measurement**
 - Input filename & description
 - Select **Set** and set the Omega origin to 1-2°
 - Set up scan as “2Theta”
 - Select box “Drive 4 axes to zero after scan”
 - Start Scan

Thin Film Conventional Diffraction with D/tex

The X-ray source is set up as a parallel beam, Omega is fixed at a small angle, and 2Theta is scanned to collect the diffraction pattern.

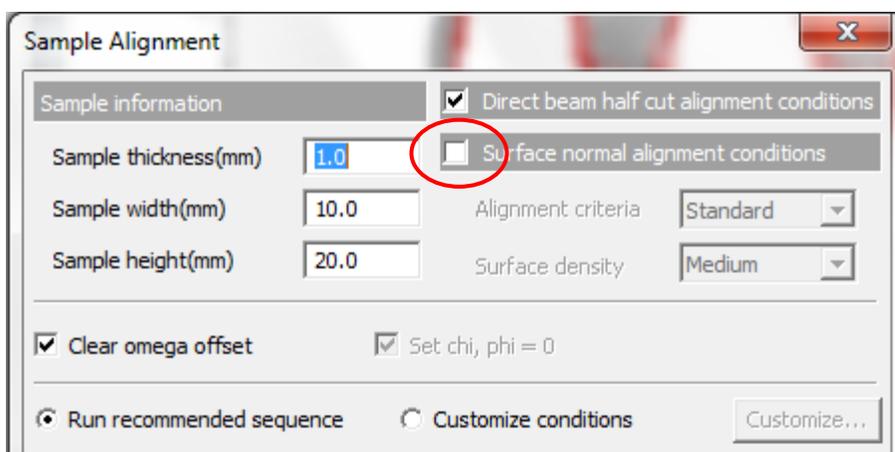
- Select **General | medium resolution PB**
- Insert centering slit and PB slit
- Perform **Optics Alignment**
- Set the sample height slit to match your sample.
- Perform **Sample Alignment**
- Note the Z value for the sample position
- Select **Control | Manual** and drive Z down by several mm
- Mount the D/tex detector and perform **D/tex Centering**.
- Select **Control | Manual** and reset the sample to the original Z value
- Select **General Measurement**
 - Input filename & description
 - Select **Set** and set the Omega origin to 1-2°
 - Set up scan as 2Theta, slits = 1, 10, 10
 - Select Drive 4 axes to zero after scan
 - Verify that the detector is set to D/tex
 - Start Scan

To use macro

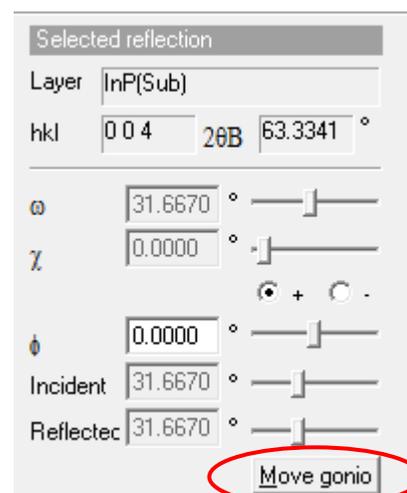
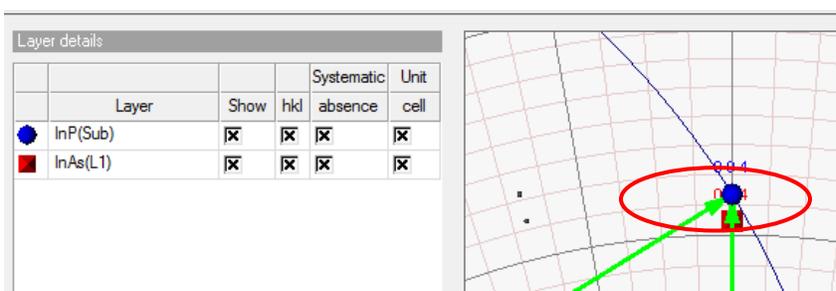
- Select **Tasks | Macro Measurement**
- From the dropdown menu select PB-Dtex
- Follow Flow buttons
- When finished, set back to **Tasks | Package measurement**

High Resolution RSM

- Select **Crystal Quality Analysis | Rocking Curve/Reciprocal Space Mapping (ultra-high resolution PB-Ge(220)x4**
- Mount the Ge(220) 4-bounce Monochromator on the source side
- Run **Optics Alignment** with alignment slit
- Run **Sample Alignment** (deselect surface normal alignment)

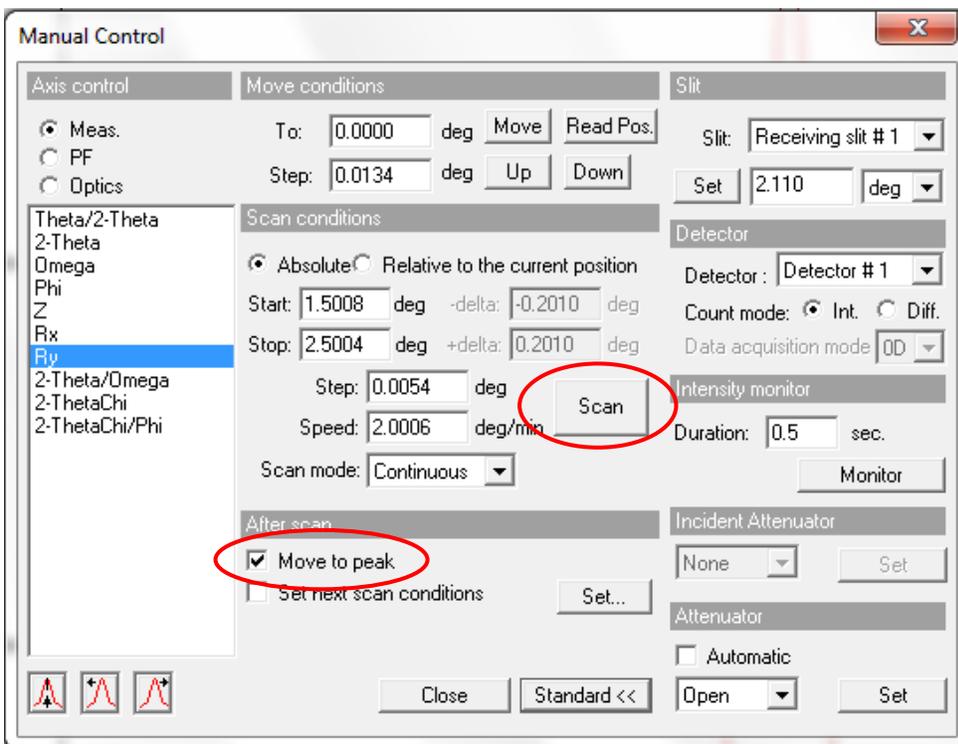


- **Process | Crystal Information**
 - Chose appropriate crystal & substrate.

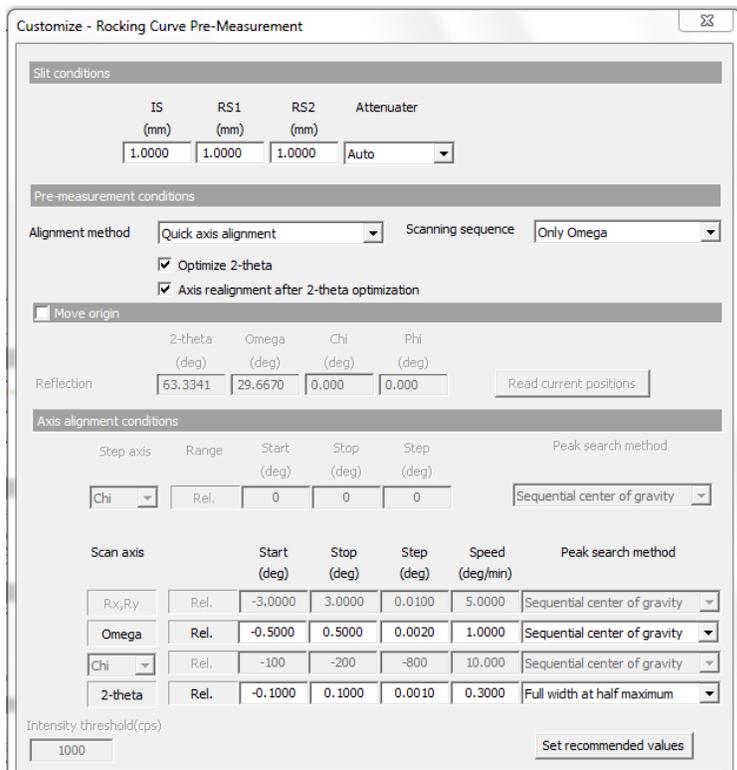
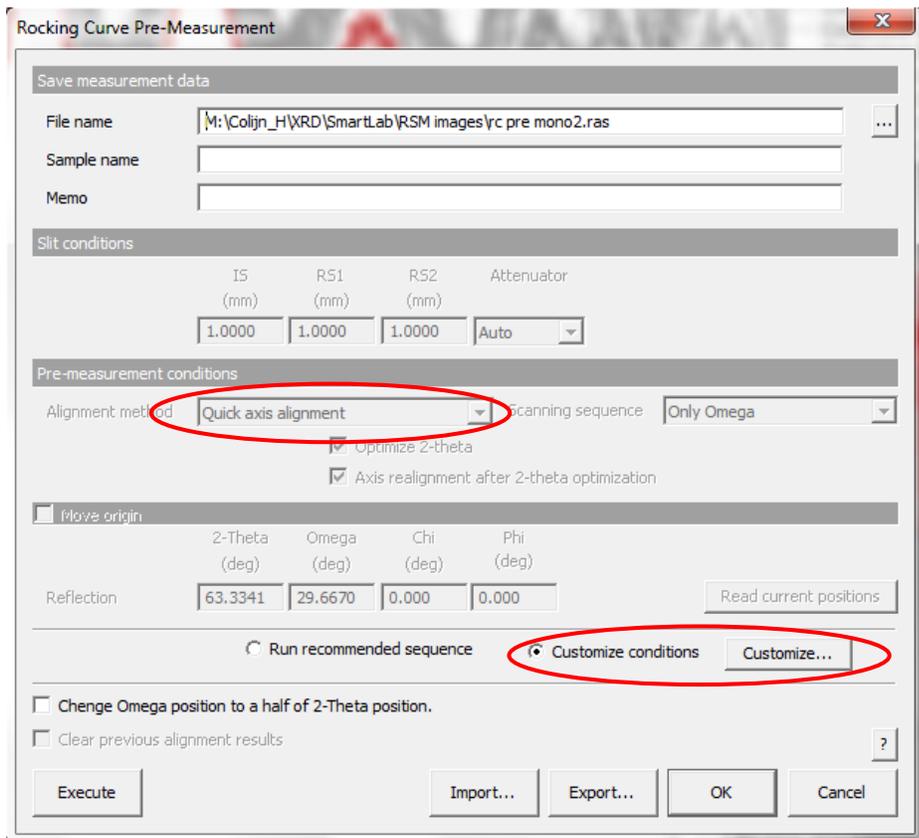


- **Process | Diff space simulation**
 - Select the appropriate substrate reflection

- **Move gonio** to the substrate angle
- Select **Control | Manual Control** to locate the substrate peak for off-cut samples
 - **Scan Ry and “move to peak”**
 - **Scan Rx and “move to peak”**



- Select **Rocking Curve Pre-measurement**
- Choose **Quick Axis Alignment** and **Customize conditions**.



- Choose **Quick Axis Alignment**. The Rx & Ry box should gray out.
- Choose
- Run Rocking Curve if desired

Rocking Curve Measurement

Save measurement data

File name: M:\Collin_H\XRD\SmartLab\RSM images\RC_after manual tweak.ras

Sample:

Memo:

Omega scan conditions

Specify 2-theta position: 69.1280 deg

Scan axis	Mode	Range	Start (deg)	Stop (deg)	Step (deg)	Speed (deg/min)
Omega	Continuous	Relative	0.0000	1.0000	0.0010	1.0000

2-theta/omega scan conditions

Scan axis	Mode	Range	Start (deg)	Stop (deg)	Step (deg)	Speed (deg/min)
2-Theta/Omega	Continuous	Absolute	63.0000	64.0000	0.0010	0.2500

Execute Import... Export... OK Cancel

Reciprocal Space Measurement

RSM Measurement

Save measurement data

File name: M:\Colijn_H\XRD\SmartLab\RSM test\InP RSM mono ana 1.ras

Sample name:

Memo:

Detector setting: Type: Scintillation counter

Measurement mode:

- Omega step / 2-theta/omega scan
- 2-theta/omega step / Omega scan
- Omega step / 2-theta scan
- 2-theta step / Omega scan

Analyzer setting:

- Use analyzer after alignment
- Use analyzer without alignment
- Do not use analyzer

Measurement conditions:

Step axis	Range	Start (deg)	Stop (deg)	Step (deg)
Omega	Relative	-0.5000	0.1000	0.0500

Scan axis	Mode	Range	Start (deg)	Stop (deg)	Step (deg)	Speed (deg/min)	Attenuator
2-Theta/Omega	Continuous	Relative	-2.0000	0.3000	0.0012	0.0200	Auto

Calculated scan duration: 00:59:50

Buttons: Execute, Import..., Export..., OK, Cancel

- Insert Ge(220) 2-bounce analyzer in Adapter Position 1 (replace open PSA); leave Soller slits as is.
- Select “Use analyzer after alignment”
- Verify Attenuator is set to **Auto**
- Be careful of your scan speed. The calculated scan duration shows only hours: minutes. If your scan is longer than 24 hours, you will not see the true time.

Analysis Parameters:

Scan Axis: Specifies scan mode. $\theta/2\theta$ (coupled) is default for powder data. Thin film analysis can be taken via $2\theta/\omega$ scans.

Mode: Choices are continuous or step. Either mode is possible with SC. Do not use step scan for scans faster than 1 deg/min (i.e., 1.2 sec/step with a 0.02° step size).

Range: Specifies range “mode” of scan as “Absolute” or “Relative”. Always choose absolute for normal scans.

Start: Specifies start (low) angle (2θ) for scan. The lower limit is $\sim 1^\circ$.

Stop: Specify stop(high) angle (2θ) for scan. The upper limit is 158° .

Step: Specifies step size in $^\circ 2\theta$ over which data is “sampled” for collection. Default is 0.02° and is generally best for most uses; may be varied to adjust resolution (smaller for slow scans or larger for fast scans).

Speed Duration time: Indicates rate of scan in $^\circ/\text{min}$ for continuous scan or time spent per step for a step scan. SC/Monochromator scan rates are typically $4^\circ/\text{min}$ or slower. High-resolution data require much slower scan rates.

IS (Incident Slit): Pulldown selects automatic slit control to maintain a consistent angular aperture (deg) or select a fixed aperture (mm). For SC/Mono the default of $2/3^\circ$ is generally a good choice.

RS1 (Rec. Slit 1): Pulldown selects automatic slit control to maintain a consistent angular aperture (deg) or select a fixed aperture (mm). For SC/Mono the default $2/3^\circ$ is a good choice.

RS2 (Rec. Slit 2): For the SC/Mono the default of 0.3mm is a good choice in BB mode.

Attenuator: Typically open (no attenuator) or Auto.

DBM: Diffracted Beam Monochromator

Comment: Optionally attaches comment to data for this run

Options: Allows setting specialized options for stages with those capabilities (i.e., rotations, tilts, positions, etc.)

Voltage (kV): Operating kV for scan (40kV is default)

Current (mA): Operating mA for scan (30kV is default; typically should be set at 44 mA)

Finishing

- Select **Shutdown** and “Set to minimum” IF no one is following you on the instrument. This will return the X-ray generator to 20kV, 2 mA
- Verify goniometer arms are at 0 degrees.
- Log out of Guidance software.

Notes

POWDL can be used to convert data files to various formats
(<http://users.uoi.gr/nkourkou/powdl.htm>)

For occupationally exposed workers, age 18 years and over, external radiation exposure will be restricted under normal conditions to the limits below

Part of the Body	Annual Dose Limit
Whole Body	5.0 Rem
Skin of Whole Body	50.0 Rem
Hands and Forearms, Feet and Ankles	50.0 Rem
Lens of the Eye	15.0 Rem

External personnel monitoring is required whenever an individual enters a controlled area and is likely to receive a dose from radiation exposure in excess of 10% of the applicable limits.

7. Personnel Monitoring (whole body or ring badges) Required? **No**

If personnel monitoring is not required, go to section 8.

8. Policy regarding occupational exposure of minors
Individuals under the age of 18 years are not allowed to be exposed to radiation levels in excess of 10% of the applicable adult limits
9. Radiation Safety is responsible for the following:
- Perform an initial audit and survey of all newly installed equipment or after any modifications to existing equipment.
 - Perform semi-annual inspections.
 - Perform an annual survey.
 - Perform an annual inventory of all inoperable units.
 - Maintain ODH registration of all industrial analytical radiation-producing devices.
Copies of radiation surveys and inspections performed by Radiation Safety are on file at room 103 Research Center Bldg, 1314 Kinnear Road, phone: 292-1284.
10. Emergency Contact Information
The 24-hour Emergency Response Number is 240-0705.

Any individuals who have non-emergency questions, concerns, or inquiries pertaining to radiation safety may contact the Radiation Safety Section of Environmental Health and Safety during normal working hours at 292-1284.

11. The Radiation Safety Section of EHS shall be notified immediately of any radiation producing device that is stolen, lost, or missing.
12. The Radiation Safety Section of EHS shall be notified immediately of any event that may have caused, or threatens to cause an individual to receive, in a period of 24 hours,
 - A total effective dose equivalent exceeding 5 rem,
 - A lens of the eye does equivalent exceeding 15 rem, or
 - A shallow dose equivalent to the skin or extremities or a total organ dose equivalent exceeding 50 rem.
13. Recognition of symptoms of an acute localized exposure

Most radiation injuries are "local" injuries, frequently involving the hands. These local injuries seldom cause the classical signs and symptoms of the acute radiation syndrome. Symptoms may include a skin lesion, erythema, blistering, dry or wet desquamation, epilation, and/or ulceration. Local injuries to the skin evolve very slowly over time and symptoms may not manifest for days to weeks after exposure.

14. Specific Standard Operating Procedures

Note to recipient of this template. Please attach a description on how to operate your radiation producing device for this section. Basic elements shall include:

- key control
- safety precautions
- hazards associated with the use of the unit
- operating log
- significance of warning lights, labels, and signals
- description of any portable shielding or barriers
- description of interlocks
- description of controlling access to the area / device
- how to operate the unit.