

Rigaku MiniFlex 6G Standard Operating Procedures



Concise Operating Procedures

1. Ensure you are familiar with safety requirements.
2. Login to the FOMLock Screen
3. Open SLS II if not already open. **Login: AMSEC PW: 4280_AMSEC**
4. Load your measurement flow or create a flow, if needed.
5. Ensure the Startup and Shutdown procedures for the X-Ray source are set in your flow.
6. Modify General Measurement for file save location and filenames. Make sure you have set the correct sample position, if you want it to spin or not, and spin rate.
7. Prepare samples.
8. Load samples. (see opening and closing the door).
9. Ensure the detector has been set for Standard or XRF Reduction
10. Begin run. Press Run Flow or Ctrl+F5.
11. Check the XRD chiller status before and after each run.

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1. Safety

This section will cover the inherent hazards when utilizing this piece of equipment as well as the best practices and procedures to avoid danger. These hazards will not include basic things that may be included in the basic safety training document that each user has attested to have reviewed at fom.wvu.edu/documents.

Lab coats are to be provided by the user unless special hazards exist in which case they are located at the PPE station.

1.1 Hazards

- Chemical exposure
- X-Ray exposure

1.2 Required PPE

Appropriate laboratory attire is required at all times in the AMSEC laboratories. Whenever chemicals are being used, an additional requirement of a lab coat is required. Lab coats are to be provided by the user.

Whenever a user is in the AMSEC labs, the minimum requirement for eye protection is wrap around impact glasses. Anytime liquid chemicals are present in the same room as the user without a direct barrier, all users in the lab must wear chemical splash goggles. Splash goggles must be approved by State of Washington Administrative Code (WAC 296-24-078).

If chemicals being used are considered toxic, caustic, corrosive, flammable solvents, carcinogenic, mutagenic, or teratogenic, a minimum of disposable nitrile gloves is required. Avoid chemical transfer by taking off gloves when using anything other than the chemical(s).

1.3 X-Ray exposure

The X-Ray Diffractometer utilizes X-Rays to carry out analyses. At any given time, X-Rays are being produced by the XRD. As long as none of the interlocks are defeated, the chances of exposure to X-Rays is nearly zero. The system has many different safety devices in place to shut down the X-Ray generator if any malfunction occurs. This means that at no time should anyone tamper with or attempt to dismantle or repair any part of the instrument. The only exceptions are for modifications to the instrument that are discretely outlined within these SOPs.

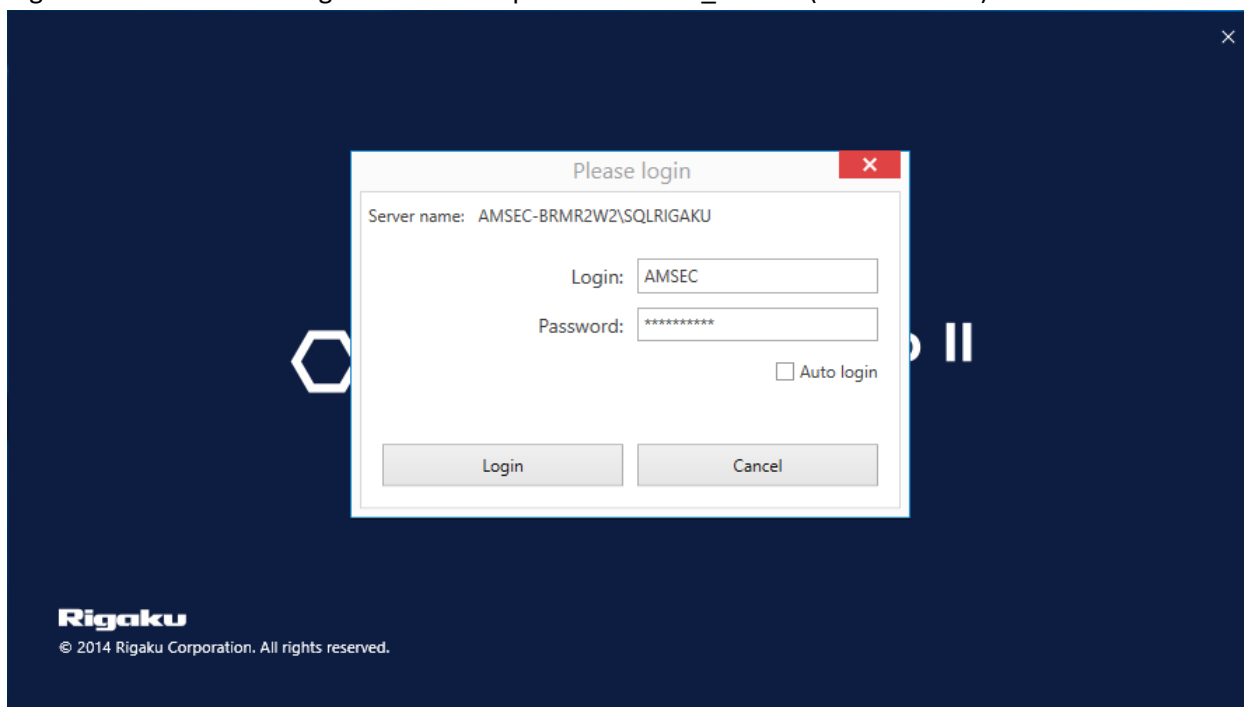
All users of the XRD are required to participate in the X-Ray radiation safety training course on Canvas. No user may have access to the instrument without passing the quiz at the end of the course with an 80% or higher. Contact the lab manager to access the quiz if you have not done so. If you already have access to the instrument but have never taken and/or passed the course, you must do so immediately.

2. Software

2.1 Login

The computer should be logged onto the local AMSEC account with login: “.\AMSEC” and password: “4280_AMSEC”. Do not login with your personal account. The instrument is controlled via FOM and has a lock screen installed which prevents unauthorized usage. Reservations must be made in advance on FOM to reserve the instrument. Otherwise, it is first come, first serve.

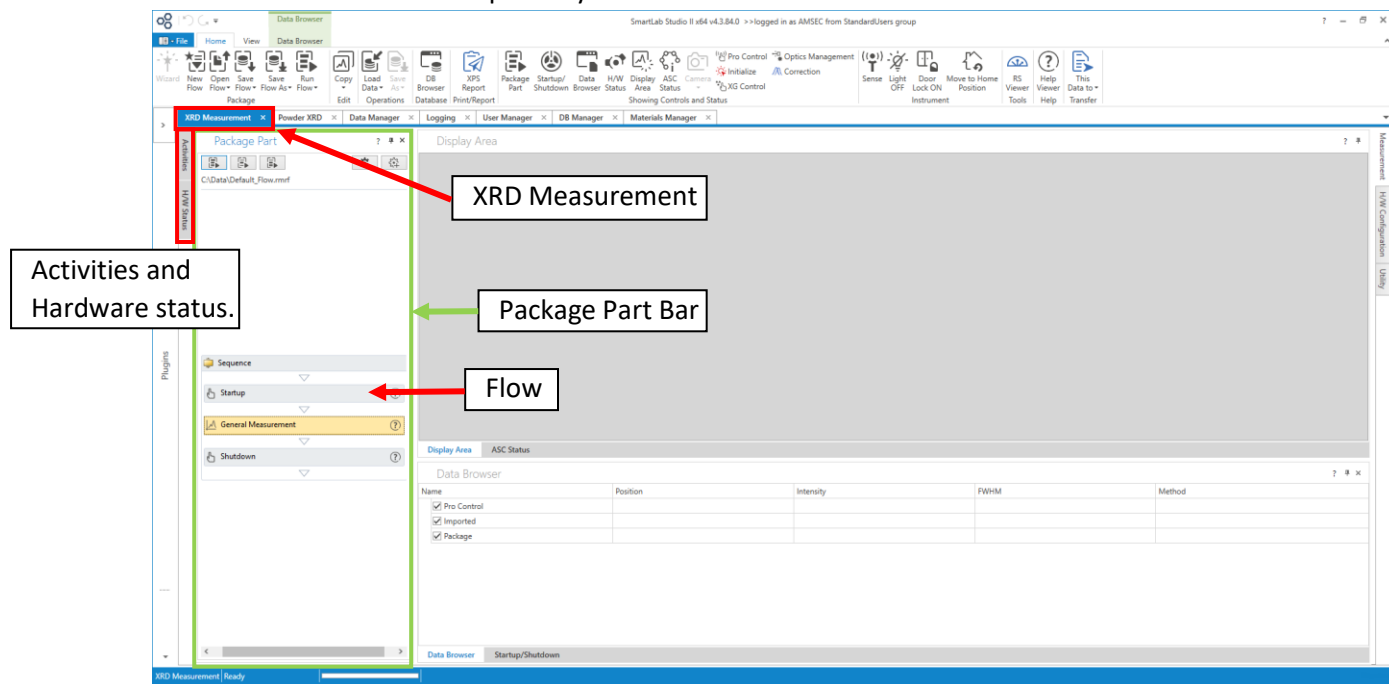
The instrument control software is called Smart Lab Studio II (SLSII) and a link should be on the desktop. Please leave the software open at all times. There is no reason to close it in between users. The login for the software is login: AMSEC and password: 4280_AMSEC (case sensitive).



The login screen is shown above and requires the login and password to load the software. Once entered, the software will connect to the instrument for several minutes and the blue part of the screen above will be show. Do not attempt to close the software while it is connecting.

2.2 Software Layout

Upon opening, the software layout may change or reset. The layout that will be shown in this SOP may be different than what you see, but the functionality will be the same. The software window may look like the image shown below. You need to make sure you are in the XRD Measurement Plugin. This can be accessed by clicking the tab named XRD measurement. If that isn't showing, hit the plugins bar on the far left of the screen, it is vertical, and a window should pop up that has XRD Measurement on it. The tab should show up after you click that.

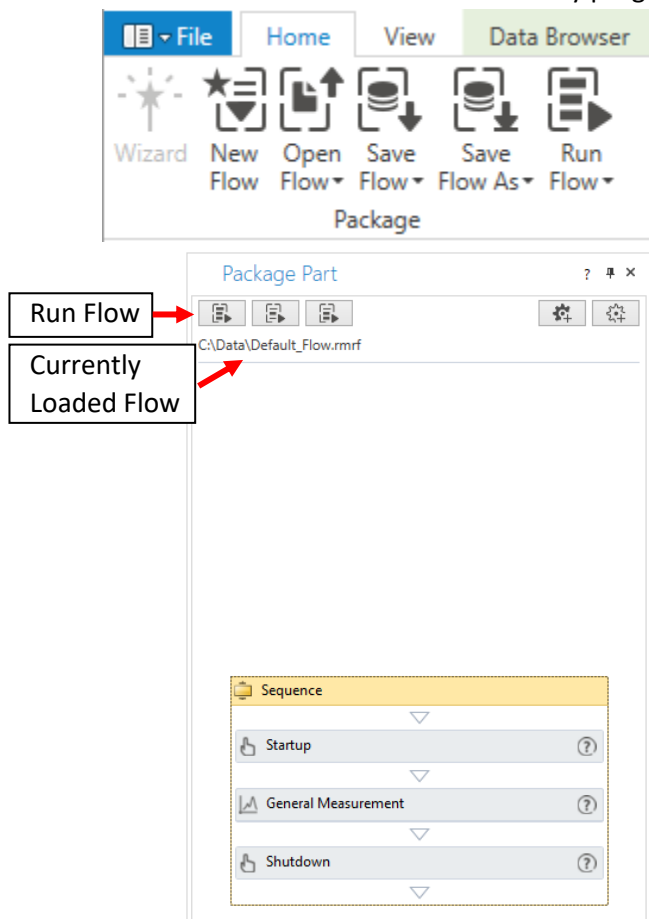


The main panel you will use is the Package Part panel shown on the left. The Activities bar is currently hidden on the left and would be used for making a custom flow. This will be unnecessary for most people. Using a default flow will work for most standard measurements. The Hardware Status bar is currently hidden on the left. The Hardware Status and Activities can be accessed by clicking on the vertical box labeled with the corresponding name.

Any window or pane described in the software can be closed by accident or not displayed upon software opening. They can be reopened and dragged into any configuration. To reopen any of the windows, use the "View" tab under the home tab, you will see Package Part and H/W Status. The Activities window can be accessed by pressing the Package Part button.

2.3 Package Part Bar

The Package Part bar is where the on-deck run will be shown. Everyone will store their flow in their own folder within the research folder in C:\Data. Each research group should have its own folder, not each user. Each user can have a folder within a research group folder. Each department should have its own folder for class related stuff. Any programs left in the general folder area will be deleted.



Regardless of if you are making a brand new flow, or if you are just going to load your premade flow, you will want to hit New Flow from the home tab to clear the area of whatever the last user had loaded. Hitting Yes on the dialogue that appears will clear the flow, hitting No will leave it how it was.

If you are making a new flow, load the "Default_Flow.rmrf" from C:\Data. This will have all the startup and shutdown procedures preloaded into it. You can load a flow of your own design, or the default one by pressing Open Flow and choosing File System. Then you will press on the General Measurement box to edit that section. See Section [2.4 General Measurement](#). Once you have made a flow that fits you, you can save it by pressing Save Flow As, and then choosing File System and save it within your own folder.

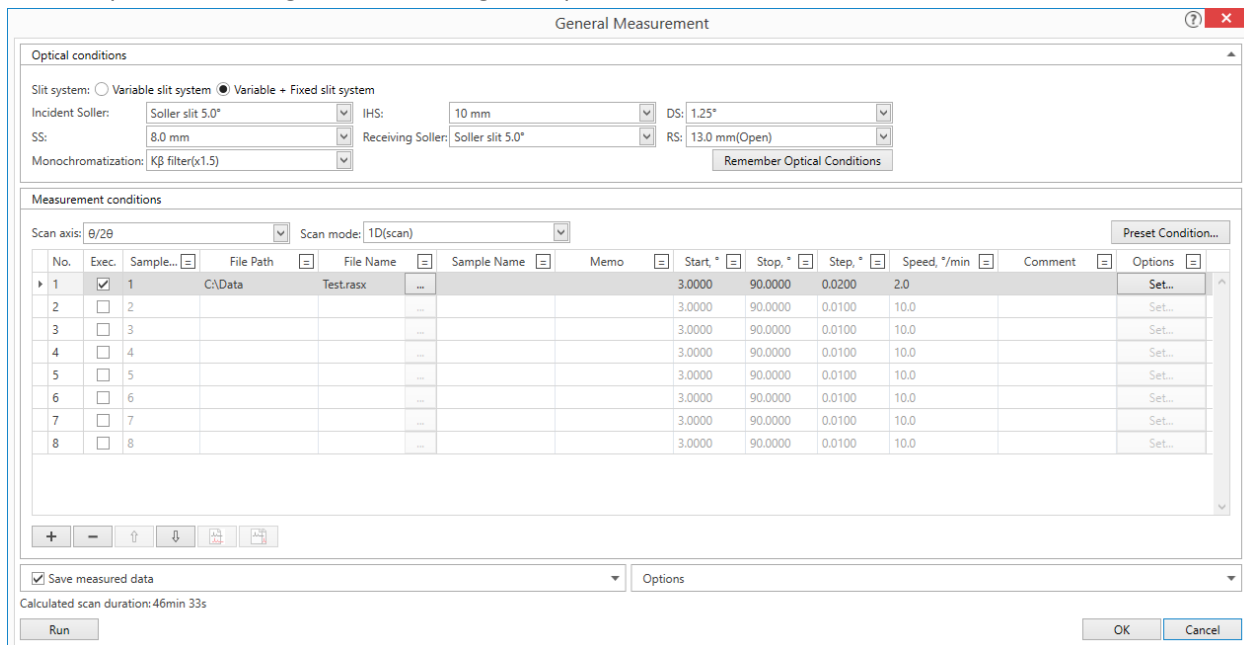
If you are loading a flow you already made, then just click Open Flow and then File System. Then you can edit the [General Measurement](#) as needed.

2.3.1 Starting a Run

Once your flow has been setup properly, you just hit the run flow button on the top left of the Package Part bar or "Ctrl+F5". Make sure to hit the proper button, the one on the left. The other two will only run partial sections of the flow.

2.4 General Measurement

The general measurement box is accessed from the Package Part bar by clicking on the currently loaded general measurement box. This will bring up the window shown below. The optics section should be minimized when you open the window, but it should look like the one below unless you have been shown by the lab manager how to change an optic.



The screenshot shows the 'General Measurement' window. It has two main sections: 'Optical conditions' and 'Measurement conditions'.

Optical conditions:

- Slit system: ☐ Variable slit system ☒ Variable + Fixed slit system
- Incident Soller: Soller slit 5.0°
- IHS: 10 mm
- DS: 1.25°
- SS: 8.0 mm
- Receiving Soller: Soller slit 5.0°
- RS: 13.0 mm(Open)
- Monochromatization: K β filter(x1.5)
- Remember Optical Conditions button

Measurement conditions:

- Scan axis: $\theta/2\theta$
- Scan mode: 1D(scan)
- Preset Condition... button

No.	Exec.	Sample...	File Path	File Name	Sample Name	Memo	Start, °	Stop, °	Step, °	Speed, °/min	Comment	Options
1	<input checked="" type="checkbox"/>	1	C:\Data	Testrasx			3.0000	90.0000	0.0200	2.0		Set...
2	<input type="checkbox"/>	2					3.0000	90.0000	0.0100	10.0		Set...
3	<input type="checkbox"/>	3					3.0000	90.0000	0.0100	10.0		Set...
4	<input type="checkbox"/>	4					3.0000	90.0000	0.0100	10.0		Set...
5	<input type="checkbox"/>	5					3.0000	90.0000	0.0100	10.0		Set...
6	<input type="checkbox"/>	6					3.0000	90.0000	0.0100	10.0		Set...
7	<input type="checkbox"/>	7					3.0000	90.0000	0.0100	10.0		Set...
8	<input type="checkbox"/>	8					3.0000	90.0000	0.0100	10.0		Set...

Buttons: +, -, Up, Down, Print, Save, Load

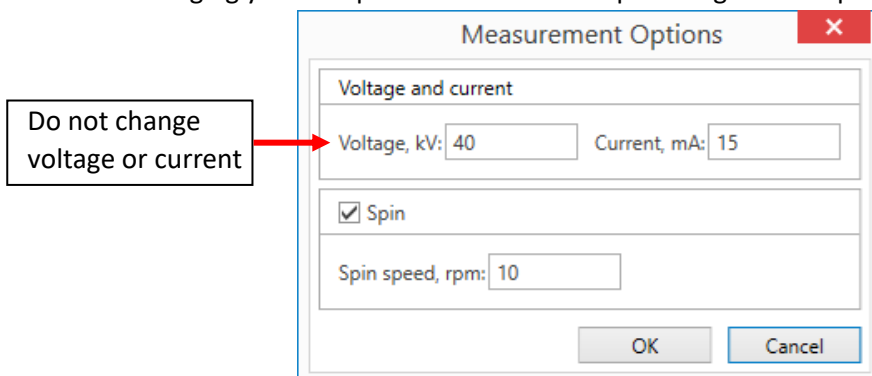
☒ Save measured data Options

Calculated scan duration: 46min 33s

Run OK Cancel

There are 8 slots for measurements, but more could be added if needed. We only have 8 positions in the auto sampler. Multiple scans could be ran on the same sample, but we typically just run one scan on each sample. Select the boxes on the left under the Exec. header to indicate how many samples you are going to run. The autosampler location is indicated in the Sample column. For each sample, you will indicate a save location under the Folder header, a file name, and a sample name. All data must be saved under C:\Data and further underneath the research (subdivided by research group) or class folder. Anything left in the main directory will be deleted. The equal sign on each header can be used to copy down an input.

The run conditions must be set for each run. The equal sign at the top can be used to copy down the first row to the rest. The option to spin a sample and the spin speed is available in the options column by clicking Set for each run. This box is shown below. This is useful to remove preferential crystal orientation. You can choose a spin speed between 10 rpm and 80 rpm in increments of 10. Slower is better to avoid flinging your sample off. Start with 10 rpm and get to 40 rpm if you can.



The screenshot shows the 'Measurement Options' dialog box. It has a red 'X' button in the top right corner.

Voltage and current

Voltage, kV: 40 Current, mA: 15

☒ Spin

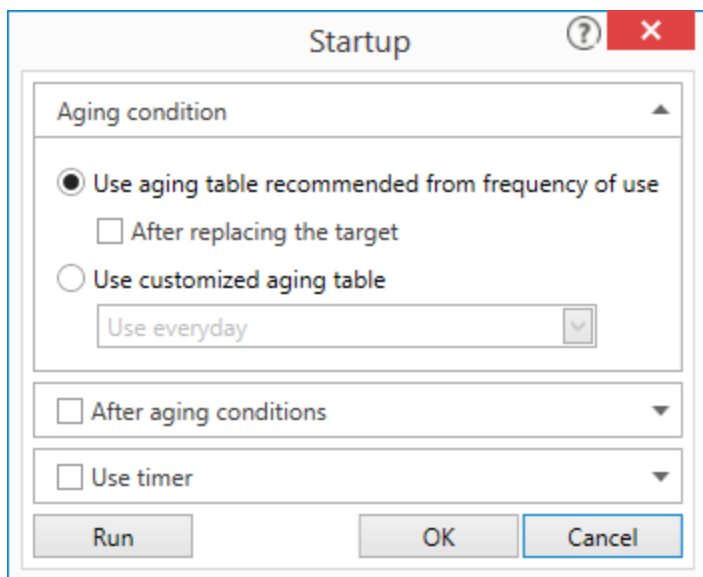
Spin speed, rpm: 10

OK Cancel

A red arrow points from a text box to the 'Voltage, kV' field.

Do not change
voltage or current

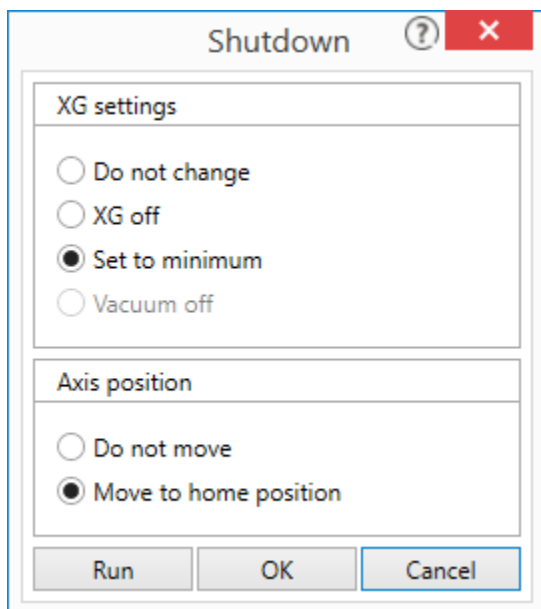
2.5 Startup



The startup section should be the first part of your flow. The window to the left can be accessed by clicking on that section of the flow. If you don't have the startup in your flow, load the [default flow](#) and start over. This window tells the X-Ray source to ramp up to operating power before starting a scan.

If you never mess with this window, the settings should be correct. It should be set as shown to the left.

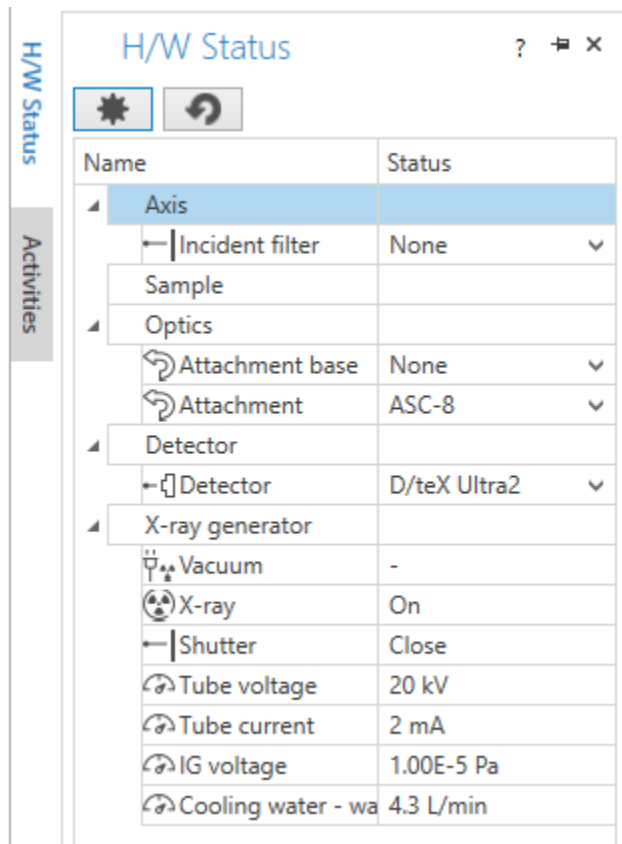
2.6 Shutdown



The shutdown section should be the last part of your flow. The window to the left can be accessed by clicking on that section of the flow. If you don't have the shutdown in your flow, load the [default flow](#) and start over. This window controls the behavior of the X-Ray source after measurements have completed. You will want to make sure that your shutdown window looks the same as the one to the left. This will ensure the source is set to its standby power which will prolong the life of the tube (~\$6k).

If there is a user scheduled after you or you intend to do more runs, you can change this to "Do not change" if the next user is not too far away from the current time. If the user is a few hours away, you should choose "Set to minimum".

2.7 Hardware Status



Name	Status
Axis	
Incident filter	None
Sample	
Optics	
Attachment base	None
Attachment	ASC-8
Detector	
Detector	D/teX Ultra2
X-ray generator	
Vacuum	-
X-ray	On
Shutter	Close
Tube voltage	20 kV
Tube current	2 mA
IG voltage	1.00E-5 Pa
Cooling water - wa	4.3 L/min

The Hardware Status pane shows information about the instrument. It shows basic information about the instrument and isn't crucial to operation. The main thing of interest is to make sure the X-rays are on and that there is measured cooling water flow.

2.8 X-Ray Fluorescence and Reduction

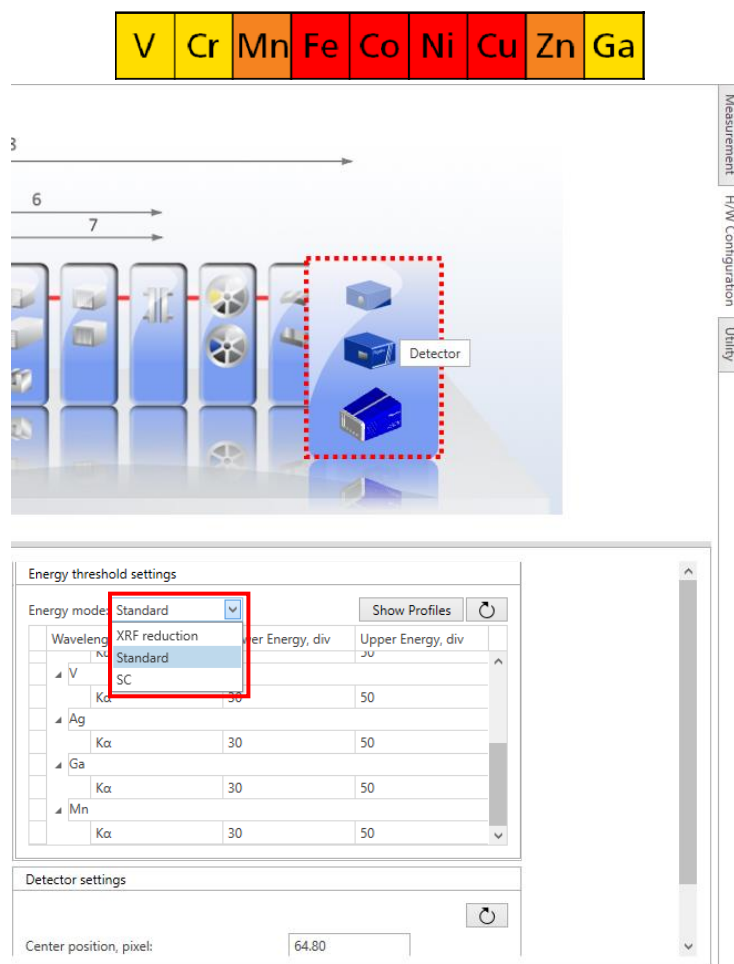
Note to all users: You will need to check this setting before every run. If you do not have the elements listed below in your sample, you will need to turn off the XRF Reduction mode. It will decrease your signal. If you do have these elements, you will need to check and turn it on before a run. There is no way to turn it on or off in the middle of a run.

If your samples contain the following elements, they will fluoresce under the copper X-Ray source that our XRD uses. The color indicates the severity of the fluorescence and how intensely it will impact your data. The fluorescence will cause you to have a high, variable background that will make it difficult to do proper phase identification and amorphous content determination.

V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga
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There are two ways to fix this issue. The first is to insert a monochromator in the instrument and run it in OD mode. This is the same setup that the PANalytical XRD has and you will need to run it on that instrument. The signal to noise will be similar on either instrument in OD mode with the monochromator. The second method is preferred if the fluorescence isn't too intense due to the concentration of the above elements in your sample. This involves changing a mode in the Rigaku Hardware (H/W) Configuration menu. There is a built-in function for reducing X-Ray fluorescence called XRF Reduction mode.

On the right side of the screen in the XRD Measurement tab, click the H/W Configuration tab. Once in that tab, click on the detector icon on the right (dotted red box below). The lower pane will change to the settings dialogue for the detector. You will choose between Standard mode for samples without the elements listed, and XRF Reduction mode for samples containing the elements listed.



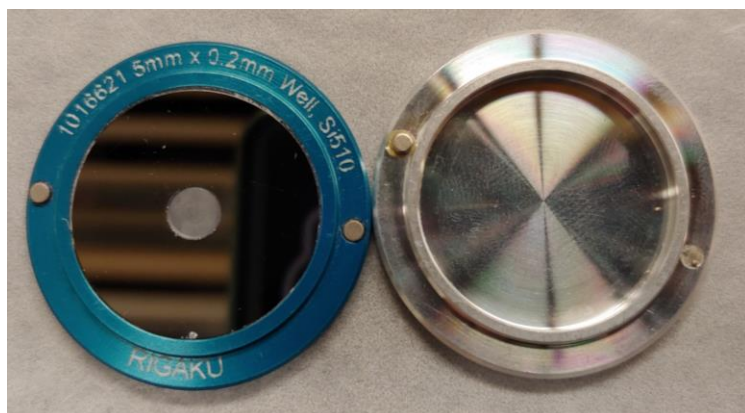
3. Sample Preparation

NOTE: Metal spatulas or anything that will scratch the sample holder must not be used. All care must be taken to keep powder from leaving the samples holders while in the instrument. Any spills must be directly reported to the lab manager via email or in-person contact.

NOTE: Solvent can only be used to wipe down the holders on a kimwipe. Holders must never be submerged in solvent or else the magnets may come loose and be lost.

NOTE: You MUST return at the end of your runs to clean off the sample holders before the next user. We don't have enough to leave sample in them for very long.

3.1 Sample Holder Preparation



There are two different types of sample holders for standard analysis. The one on the left is the zero background holder. This holder is designed for very small sample loadings. The mirrored surface must not be scratched at all costs. The dimple in the middle is where the sample will go.

The holder on the right is the standard holder. We should avoid scratching the surface as much as possible.



The procedure for the zero background and standard holders is pretty much the same. The goal is to get your sample level with the top of the recess the sample it meant to inhabit. Use a kimwipe with some solvent on it to clean the sample holder. Use a spatula or simply dump a little powder onto the sample holder ensuring you do not touch the sample holder with the spatula.

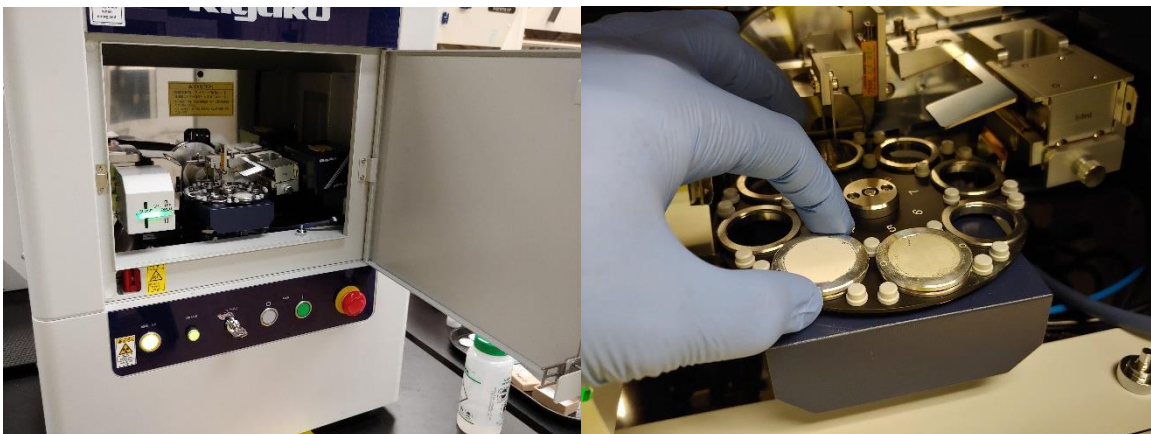


Take a clean slide and use it to smash the sample into the holder. Pressing down and using a circular motion helps distribute the powder. It is easier to add more versus remove extra so start on the safe side for the amount added.

The image to the left has been leveled successfully. The edges need to be cleaned up to make sure that excess powder does not make its way into the instrument. The image on the right shows a completed sample that can now be loaded into the instrument.

3.2 Inserting a Sample

To place a sample into the instrument, you must open the doors first. See [4.2 Opening the Door](#) for more information.



Open the door and place the sample in the 8-position auto sampler. The sample holders have magnets in them and they will snap down into the receiving rings. Take care not to allow them to snap down as this can send your sample flying all over the place.

If you need to use all 8 spaces and a sample needs to be placed under the beam knife, lift the beam knife to place the sample. Set the beam knife down gently so it doesn't snap down. This could damage it if done repeatedly. See [4.3 Beam Knife Usage](#) for more information.

4. Instrument Operation.

4.1 Instrument Overview



The XRD system contains the main unit (left) and the chiller unit located under the table. The chiller is discussed later in [4.4 XRD Chiller](#). The main unit has the X-Ray source which has a copper anode with a 1.540593 Å wavelength for K α with a minor amount of K β that we filter out.

The front consists of the door and the control panel located below the door. The orange button on the left is to unlock and lock the door.

The operate button should be illuminated at all times solid. If it is flashing, there is an error and the lab manager should be notified immediately.

The HV Enable key should be horizontal and should not be turned or removed for any reason.

The white O button and the green I button are to shut down and turn on the instrument, respectively, and should not be touched for any reason.

The big red button on the right is the emergency shutdown button. This can be used in the event of an emergency such as a radiation containment break. This should only be pressed in the event of an emergency.



4.2 Opening and Closing the Door

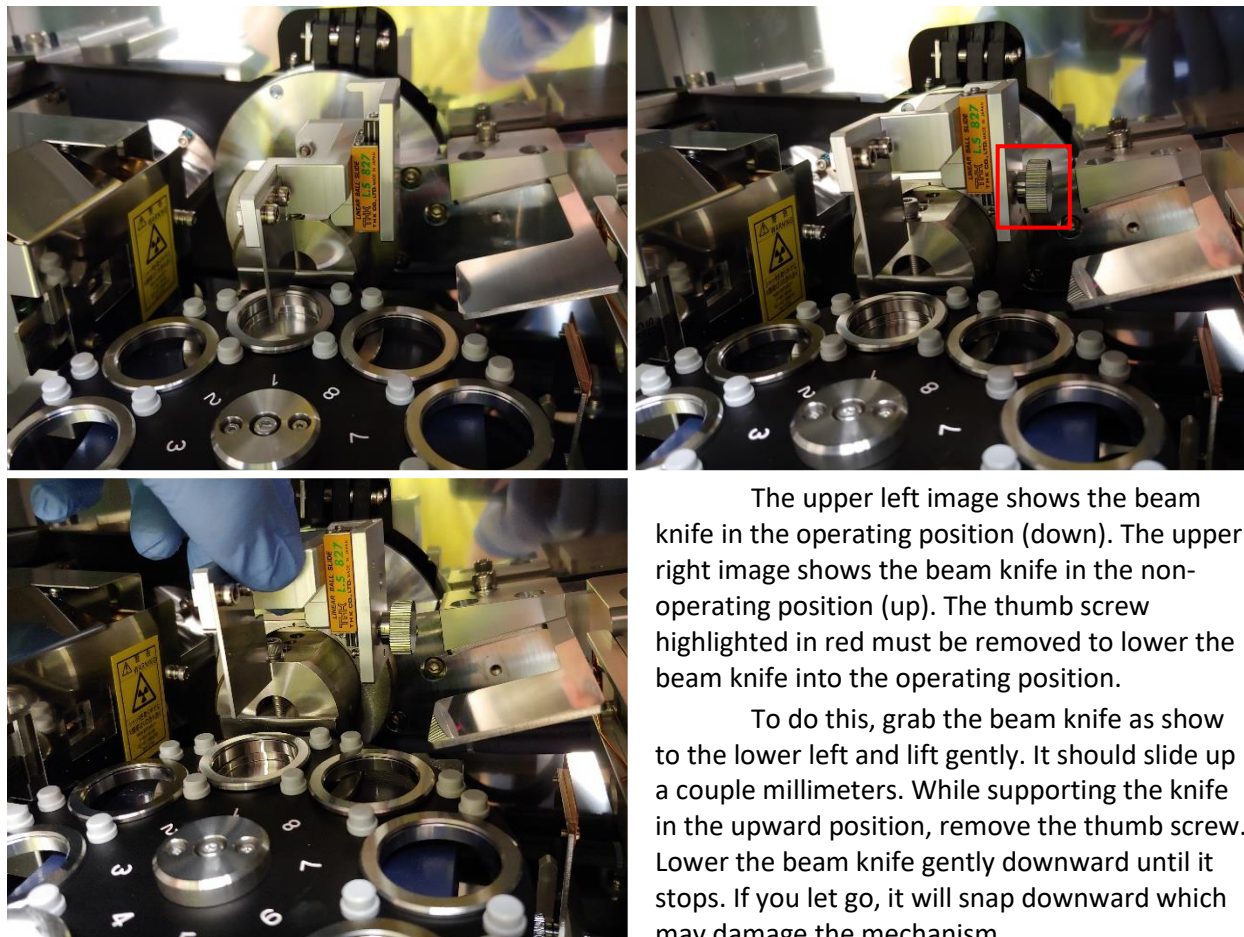
Opening the door requires that not scans are in progress and the instrument isn't moving at all. While the doors are open, the software cannot be used and the X-Rays will not be allowed out of the X-Ray tube.

To open the door, simply press the orange Door Lock button. Wait until the beeping begins before pulling on the handle. The beeping will continue until the door is locked. This cannot be bypassed and should not be bypassed by anybody.

To lock the door, ensure the door is fully closed and press the Door Lock button. The beeping should stop and the software should be usable again. If those conditions do not happen, try unlocking, fully closing, and locking the door again.

4.3 Beam Knife Usage

The beam knife will typically be used for all measurements. The beam knife prevents X-Ray scattering at low incident angles. Ensure the beam knife is down before your run starts. You should do this when loading your samples.



The upper left image shows the beam knife in the operating position (down). The upper right image shows the beam knife in the non-operating position (up). The thumb screw highlighted in red must be removed to lower the beam knife into the operating position.

To do this, grab the beam knife as show to the lower left and lift gently. It should slide up a couple millimeters. While supporting the knife in the upward position, remove the thumb screw. Lower the beam knife gently downward until it stops. If you let go, it will snap downward which may damage the mechanism.

The thumb screw should be left just inside the door on the far right next to the spot where the ATT cable plugs in. When setting the beam knife position to non-operating, the thumb screw must go in the lower hole on the right side.

4.4 XRD Chiller



The XRD chiller is located under the table the instrument sits on. It must be checked before and after your runs to ensure it is operating correctly. The chiller cannot go over 95 °F as displayed on the front. If it is getting close to the 95 °F limit, notify the lab manager immediately.

No other actions should be taken with the chiller. Users should never add or remove water or otherwise tamper with the chiller.

If the alarm sounds, the system should be shut down immediately by pressing the white O button.