The User Manual

This manual is meant to aid you in your experiments after having received the training to use the diffractometer. The manual does not replace the training and you are not allowed to use the diffractometer before receiving a training from an operator of the XRD.

Contents

Before getting started...

Before starting to use the Rigaku SmartLab x-ray diffractometer you must get the user training.

After having the user training and reading this manual, you should be able to:

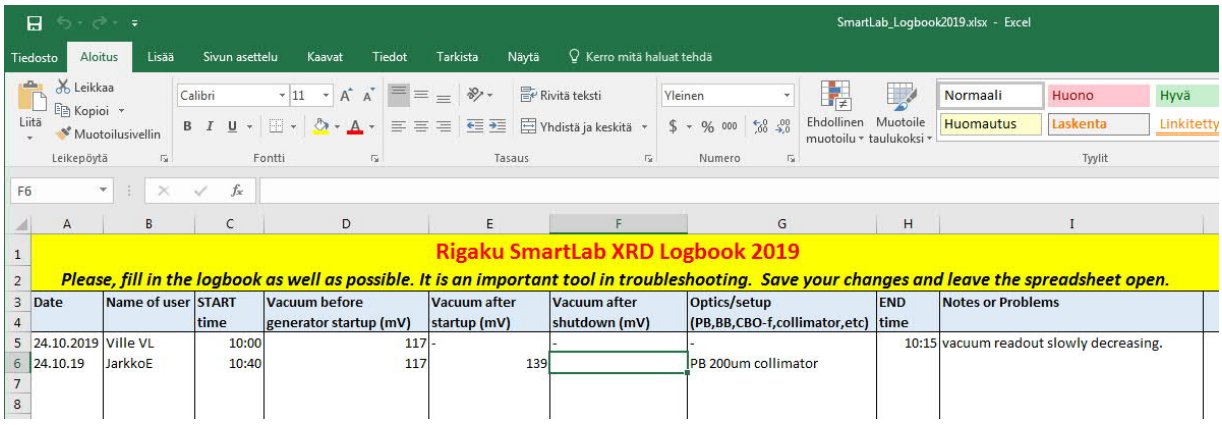
1. See if the device is on.
2. The risks (health/property) of using the instrument, and what are the measures to avoid accidents/damage to health and property.
3. Carry out instrument start-up and shut-down safely.
4. Know the basics of how to control the instrument manually (move the goniometer, manual scan).
5. Be capable of changing the setup. I.e. how to assemble and disassemble various x-ray optics components.
6. Carry out basic experiments successfully.
7. Know how to transfer data from the instrument computer (you are not allowed to use USB drive to directly transfer data from the instrument computer).
8. Who to contact in case of problems when using the device/accidents. Do not hesitate to ask for help. In case of emergency, call 112.
9. Make reservations on the device.
10. Know where to find information and manuals.

These points should be checked after the training.

Starting up the x-ray generator

NO STARTUP, IF INSTRUMENT IS ON.

1. If the instrument control software is not running, double click on SmartLab Studio on the Desktop. The username is “User1”. Press “Login” with no password.
2. Click once on SmartLab Guidance. Wait until the software is ready and no window asks you to close the doors of the radiation enclosure.
3. Go directly to step 4., if the x-ray generator is on (check voltage/current in software panel and the yellow light on top of the instrument). Otherwise, check that the vacuum level is OK (<140 mV, in the component status panel) before proceeding to Startup. The vacuum level can be too high. In that case you either wait or contact staff.
4. Fill in user/measurement/instrument information in the logbook excel sheet which should be open. Update the information/make notes during the experiment, when needed. (Otherwise you will find it on the Desktop). Save the changes. Pay special attention to the vacuum level, which should stay in ca 120-160 mV range during operation and never exceed 200 mV.

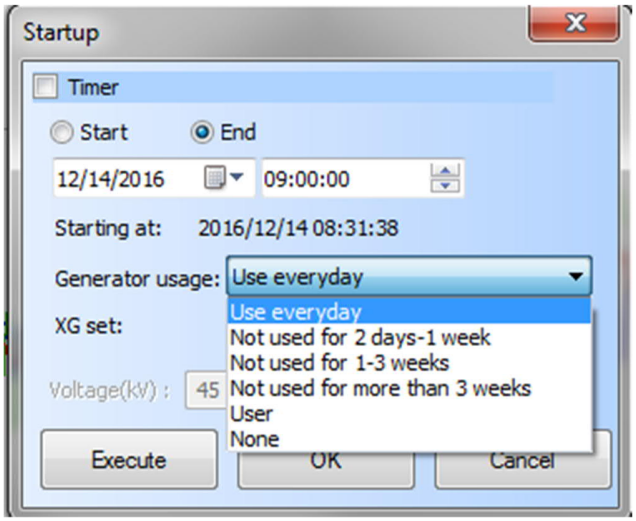


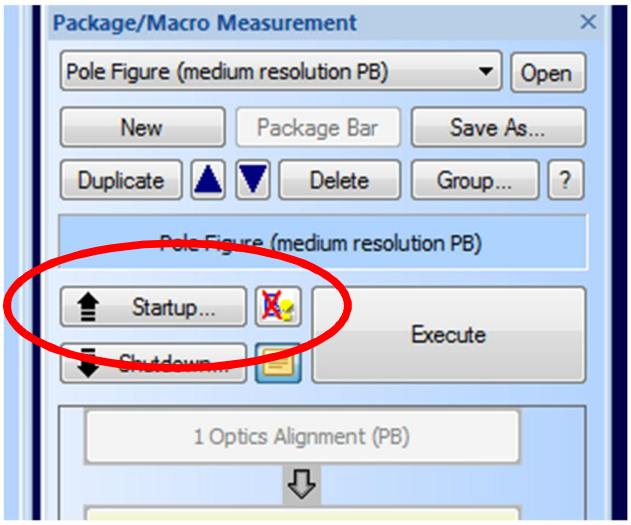
Rigaku SmartLab XRD Logbook 2019

Please, fill in the logbook as well as possible. It is an important tool in troubleshooting. Save your changes and leave the spreadsheet open.

Date	Name of user	START time	Vacuum before generator startup (mV)	Vacuum after startup (mV)	Vacuum after shutdown (mV)	Optics/setup (PB,BB,CBO-f,collimator,etc)	END time	Notes or Problems
24.10.2019	Ville VL	10:00	117	-	-	-	10:15	vacuum readout slowly decreasing.
24.10.19	JarkkoE	10:40	117	139		PB 200um collimator		

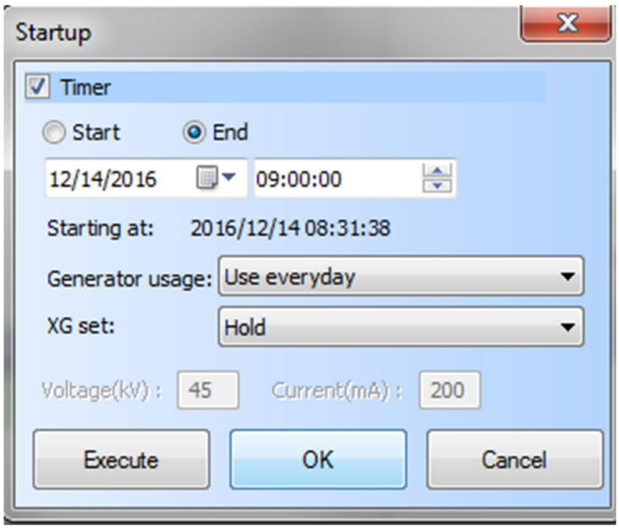
5. Click on the Startup button on SmartLab Guidance. A Startup window appears.





6. You can either start heating up the x-ray generator directly by pressing Execute in the Startup window after you have selected the correct option for generator usage (depends on when the diffractometer was used last time, check the reservation calendar), or
7. you can plan ahead and set up a timer for example in the previous evening, so that the heating up is ready for you when your reserved time starts, by ticking the Timer box, selecting the correct time and by clicking OK button in the Startup window. The timer is active if the Startup button has a 

red arrow.



8. After pressing the Execute button in the Startup window or after the timer activates, it typically takes about 30 minutes to reach standard operation voltage (45 keV) and current (200 mA). The “Hardware Control” – window disappears when the heating up (or “aging” as it is called in the software) is ready. Then you can start measuring.

Shutting down the x-ray generator

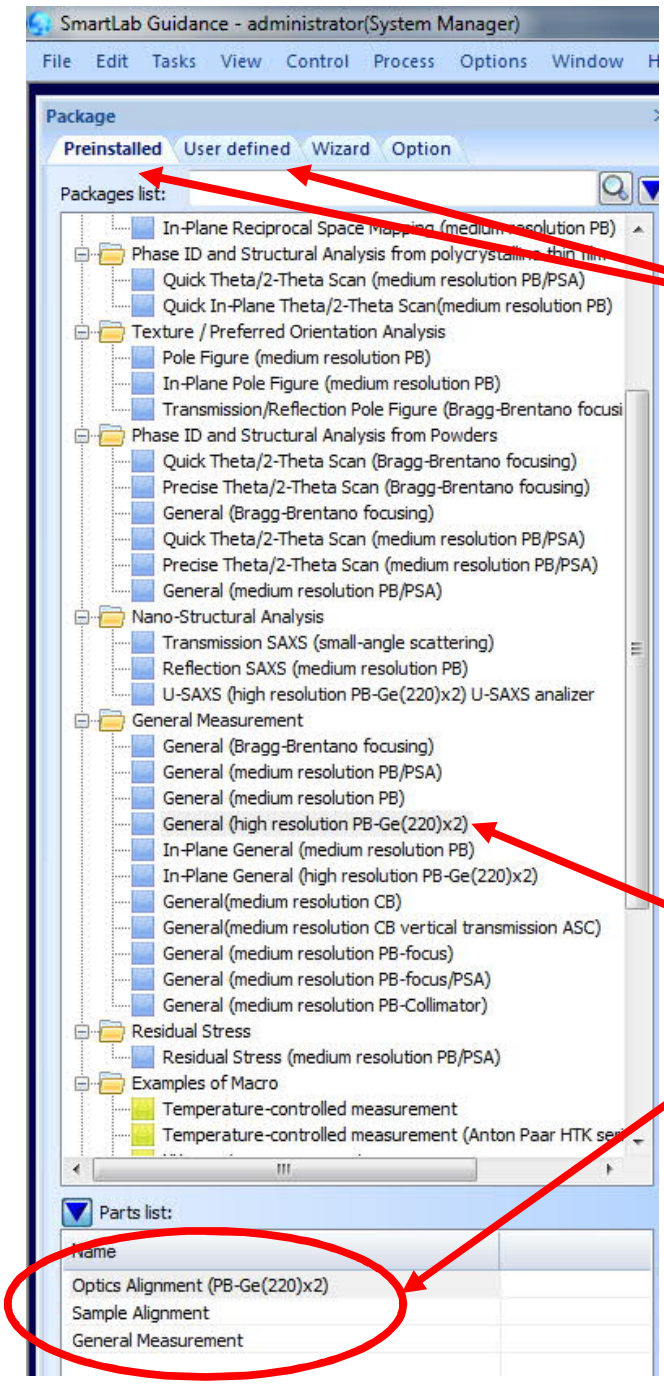
After your experiment is over, check the reservation schedule if somebody else has reserved the setup. If the reservation is right after you or a couple of hours later, you can leave the x-ray generator on and the software as it is. Otherwise, you must switch off the x-ray generator so that the filament and anode are not worn out without reason.

The ShutDown procedure has been updated 10.02.2020 due to repeated vacuum problems. Now the last user of the day should shut down the x-ray generator MANUALLY using the software instead of using the “ShutDown”-button. This procedure takes ca 15-20 min, which the last user of the day needs to prepare for.

1. Go to "Control"-drop down menu > "XG Control". You can from there manually control the accelerating voltage (kV) and current (mA).
2. Carry out the ShutDown in the following steps:
   - Set Voltage=45kV / Current=170mA
     (you need to click "set" every time to change the actual values)
   - Wait 2 minutes
   - Set Voltage=45kV / Current=140mA
   - Wait 2 minutes
   - Set Voltage=45kV / Current=110mA
   - Wait 2 minutes
   - Set Voltage=45kV / Current=80mA
   - Wait 2 minutes
   - Set Voltage=40kV / Current=80mA
   - Wait 1 minute
   - Set Voltage=40kV / Current=60mA
   - Wait 1 minute
   - Set Voltage=35kV / Current=60mA
   - Wait 1 minute
   - Set Voltage=35kV / Current=40mA
   - Wait 1 minute
   - Set Voltage=35kV / Current=20mA
   - Wait 1 minute
   - Set Voltage=35kV / Current=10mA
   - Wait 1 minute
   - Set Voltage=20kV / Current=10mA
   - Wait 1 minute
   - Click X-Ray: "OFF"
3. Write the final pressure (mV) value in the logbook.

Optics alignment

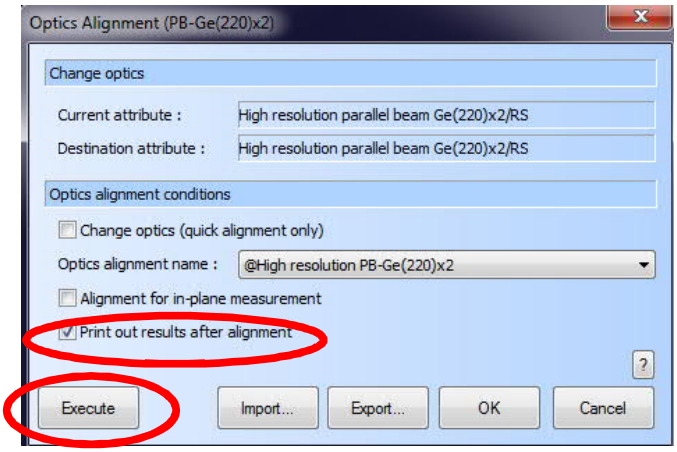
Select the macro you want by double clicking, to replace the existing macro. If you click only once, you can select the parts you want from the macro in the field below.



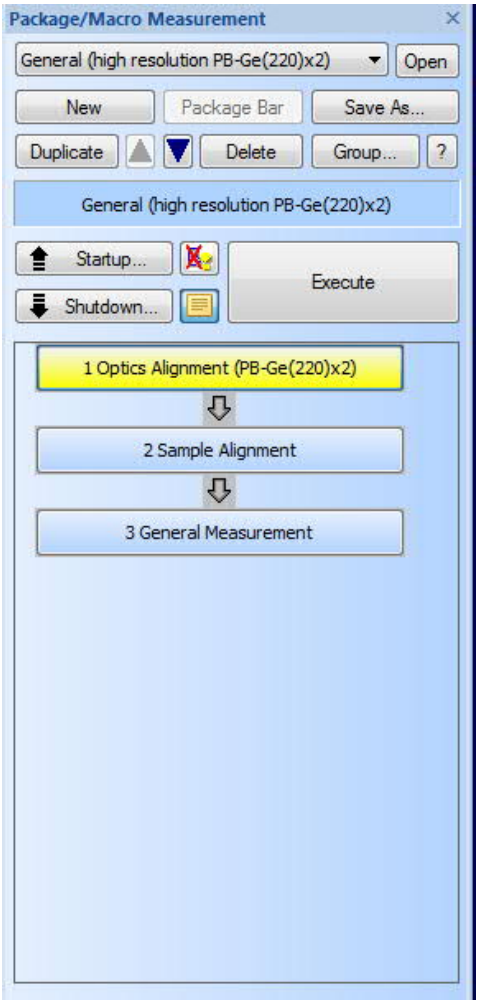
Choose one of the preinstalled macros or a user-defined macro

When clicking once the macro name, you see the list of parts below

The parts of the macro are shown in the Package / Macro Measurement window. Yellow part is the active one. Align first optics by pressing the Optics Alignment part.



Make sure the Print out results after alignment is ticked in order to save the alignment result and press Execute.



Choosing the right setup for your measurement

X-ray diffraction measurements are always a compromise between intensity and resolution. If you need to resolve the diffraction peaks very precisely and you have big crystals (i.e. thin diffraction lines), use the high resolution measurement options with germanium double-bounce monochromator (Ge (220) x 2). The monochromator should also be used usually in x-ray reflectivity measurements unless you have very thin film (e.g. thinner than 5 nm). When using the monochromator, we eliminate all other wavelengths from the x-ray source but the Cu $K\alpha_1$.

Sometimes you need a lot of intensity, for example if your powder contains only five volume percent of the interesting phase. Then it is better to use the parallel beam medium resolution options (PB). This gives 10 – 100 times more intensity than the Ge (220) x 2, but you have two wavelengths in the beam, Cu $K\alpha_1$ and Cu $K\alpha_2$. At high angles you will hence see two peaks from each crystal lattice plain. The analysis software PDXL is able to handle this, but for a publication, it might be nicer to show data measured with the monochromator instead. However, sometimes it is impossible to get data within reasonable time the monochromator setup. Then one must use the PB mode.

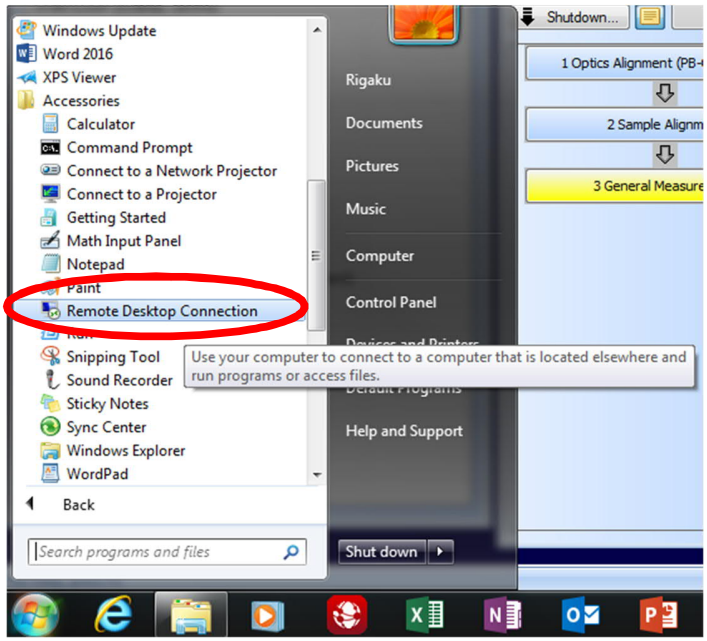
General guidelines for changing making changes in the setup

- Ask for guidance whenever in doubt.
- Do not risk damaging the detector.
    - Always cover the detector with the metal cover plate, when operating close to the detector. E.g. changing parts on the receiving side, changing sample when no receiving side optics used, etc. The beryllium window is toxic and brittle.
    - When moving the detector, loosen it from the rail only as much as needed for sliding it to the right distance. Support the detector all the time, when loosened. Shaking the detector can break it.
    - Never expose the detector to a focused direct beam, without using the absorber. High x-ray intensity can harm the detector.
- Handle all components with care. Wearing out components or giving rise to even small damages (dropping the components etc.) will result in decreased intensity levels and inaccuracy.
- Only use the dedicated hex key/Allen wrench, which is stored inside the instrument. There are no components that need any other tools for adjustments.
- Tighten the screws lightly. Do not use excess force. It will just damage the components.
- Always before moving motors, check for collisions, especially when using a collimator or the anti-scattering slit, which reduces air scattering in powder measurements.
- If you use the fluorescence rejection mode of the detector (Basic parts > Setup HyPix detector > Energy mode > XRF reduction), always change back to standard mode after the measurement.)

General guidelines for powder measuements

- For Bragg-Brentano (BB) measurements the detector must be positionedexactly at distance 350 mm. Check the distance.
- The standard z-motor position for powder sample measurements is -0.05 mm.
- Always use 1D mode (except if you use a heating chamber at the sample stage, then use 0D). RS1 and RS2 can be fully open (RS = receiving slit).
- IS (incident slit) should be 0.5° if you measure at 2θ > 10° or 0.25° at smaller angles.
- The general measurement program does not remind you to insert the Cu Kβ-filter, but you must use it if you use the Bragg-Brentano geometry. Insert it after optics alignment.

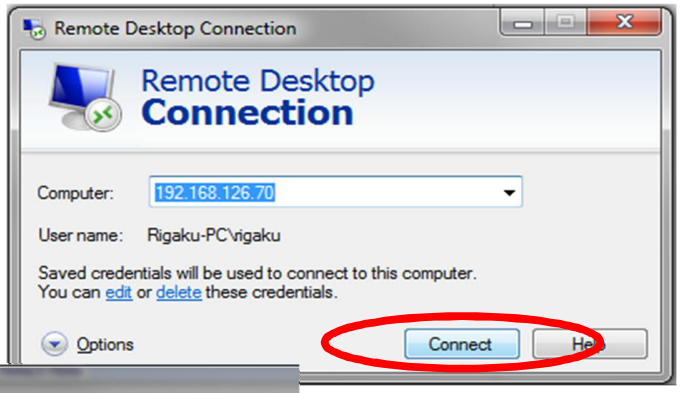
Checking detector status

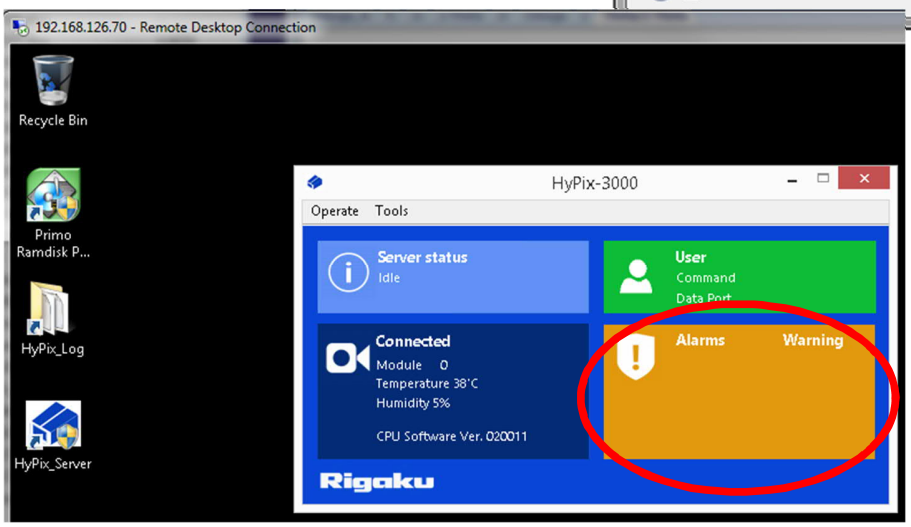


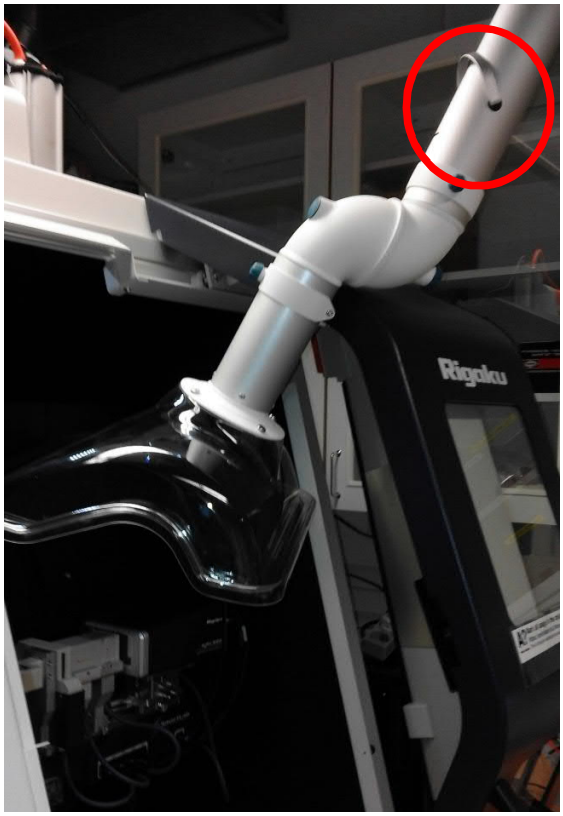
Ideally, the HyPix-3000 detector should have temperature 35 – 40 °C, but occasionally the detector heats up too much or there is some problem with communication and the SmartLab Guidance throws out an error. In this case, you can check the detector temperature by connecting to the detector computer via a Remote Desktop Connection.

Press Connect button when the Remote Desktop Connection window appears. The correct IP address is 192.168.126.70. User name is Rigaku, with no password.

Then the desktop of the detector computer is visible with the HyPix-3000 server window. If an alarm or warning code appears on the bottom right corner, write it down in the logbook and take screen shot and save it in errors folder.

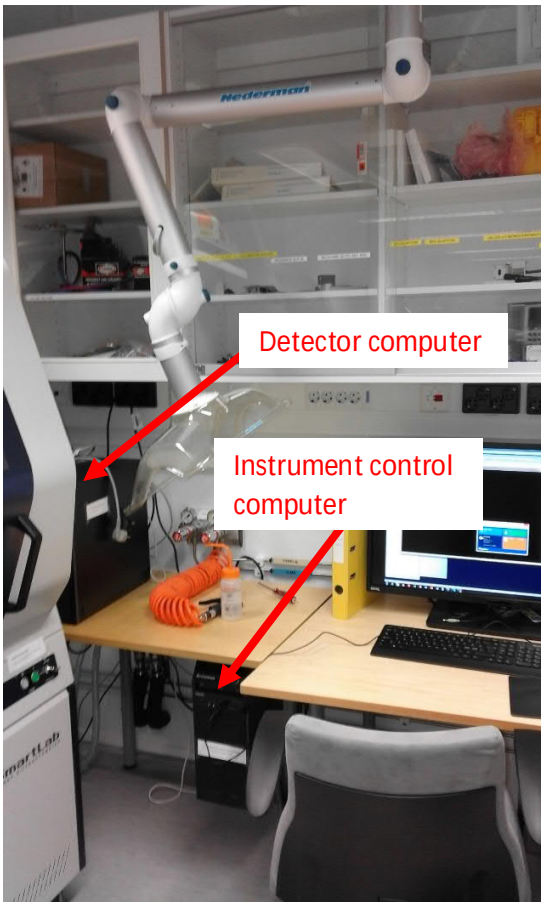






Movable exhaust ventilation system for cooling down the detector for a while. Set the ventilation on by the handle circled in red. Switch off ventilation when not in use by turning the handle back to bottom position.

To reset alarms and warnings, restart first the detector computer (by pressing the power button for a few seconds) and then the instrument control computer (from Windows Start button). If the software forgot the offsets of the motors after restarting the computers, you might need to do optics alignment and sample alignment again.



UPDATE THESE IMAGES WHEN THE LAB IS READY!

Saving measured data

Network storage at the measurement computer should be mapped as a network drive (Z:\), if not, you find it at:
\\tw-harley.org.aalto.fi\XRD

Username: AALTO\nmc-xrd Password: Q+SPjVk3yrE9

Once the network drive is mapped (usually it is), save your data in folders of the format

Z:\\year\yearmonthdaySurname\

e.g. Z:\\2017\20170816Vainio\

The "day" is the day when you start measuring. You can use the same folder for one "project". For example, if you reserve the device for two days, use the same directory for all measurements from those days.

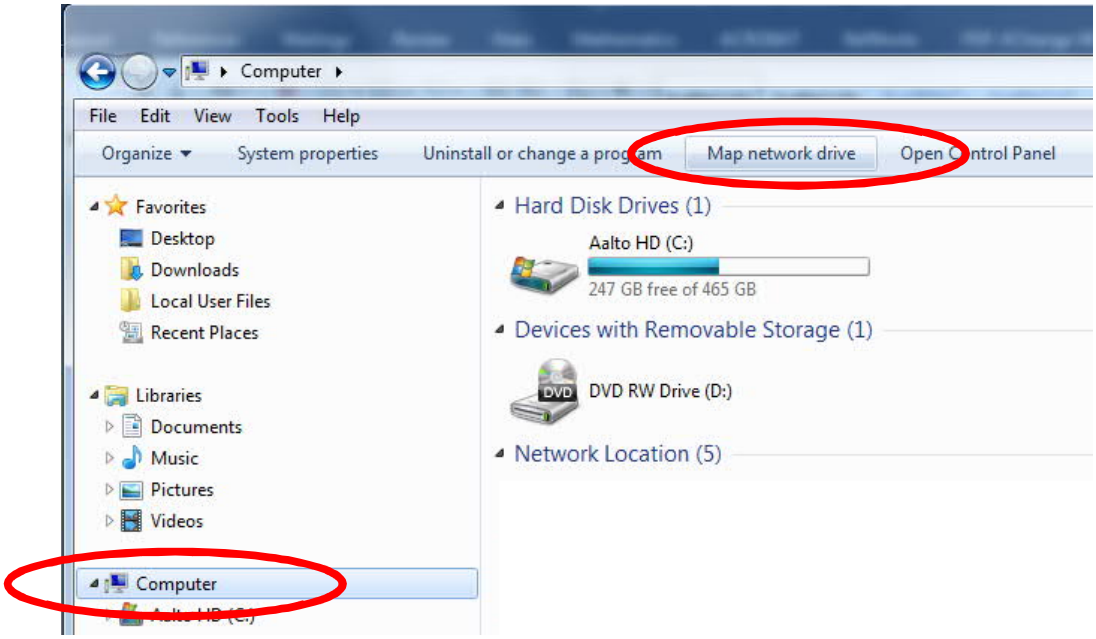
Save alignment data to directory Z:\\year\alignment\ and indicate the date and the type of optics used in the file name. This way we can track how the intensity has changed over time for each setup configuration.

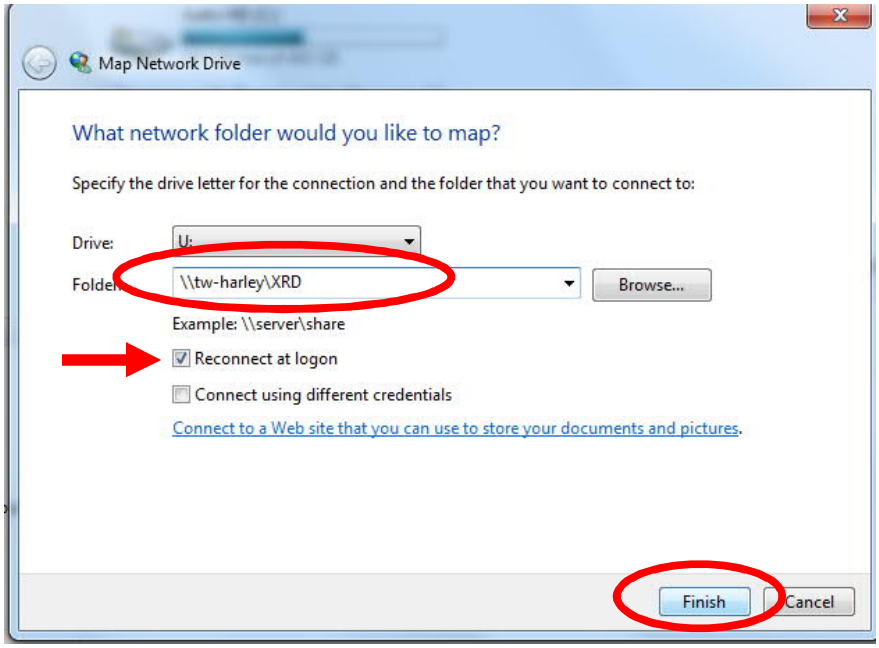
This system will allow us to track easily which people should be contacted if we notice some problems in the data afterwards.

If you cannot access the data, you have not been added to the user group yet. The device administrator has to ask the IT Servicedesk to add you to tw-harley-nmc-xrd-read-only.

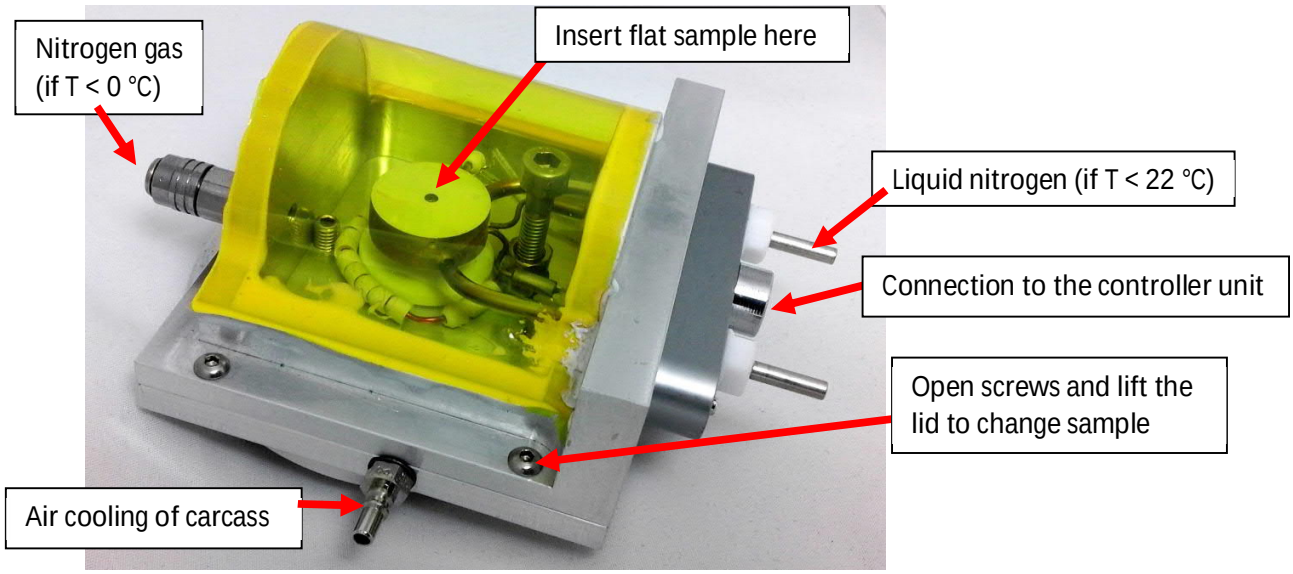
Accessing measured data

To access the data on your own Aalto computer, go to your computer, open Windows Explorer, select Computer and Map Network drive. Type in \\tw-harley\XRD in the Folder field, make sure Reconnect at logon is selected and press Finish.





Installing the 600 °C heater



This heater is based on a Linkam HFS600 stage. An aluminum carcass was built around it and the yellow Kapton window on top allows x-rays to pass. Only samples of about 0.5 mm in thickness or thinner then that can be measured with the heater, because of height limitations.

The temperature range of the heater starts from liquid nitrogen temperature or slightly higher (depends a little bit luck) and goes up to 600 °C, continuously. The heater is cooled down with liquid nitrogen if the dewar is installed inside the diffractometer enclosure.

When you insert the Linkam controller units (and possibly the dewar) on the bottom of the diffractometer enclosure, make sure all other items are removed from the bottom to be able to put the controllers are low as possible. This way the detector arm cannot hit so easily the controller units. If you heat the sample, make sure the air cooling tubes are installed and pressured air flows through the cooling channels of the heater. It is important not to heat the sample stage under the heater in order for the motors etc. to work also later on.

When cooling below 0 °C, insert the gaseous nitrogen hoses to the heater so that the sample chamber is filled with nitrogen. Flush the chamber first properly with nitrogen but keep the nitrogen flow as small as possible during measurements to avoid conduction of heat away from the sample. After the measurements, do not forget to the close the nitrogen and air shut off valves on the wall.

Always check after installation of cables or controllers that no cables or hoses block the moving of the detector and the source arms! When you switch on the two controllers, first switch on the cooling unit and then the main controller.

Installing the Linkam HFSX350-GI heater

For this heater which is in the original Linkam carcass, there is no option to put nitrogen atmosphere or to cool the carcass much, so do not heat it to higher than about 200 °C (and also there only for a short time) even if the maximum in principle is 350 °C.

Troubleshooting

- When an unexpected error or other unwanted event occurs, please, do mak a simple documentation of it:
    - Take a screenshot of the problem and save it to "C:\Users\Rigaku\Desktop\Error Messages – Screenshots"
    - Write a short description in the logbook spreadsheet. (E.g. measurement was interrupted and the system was automatically shutdown, Error message "…." was displayed.)
- PROBLEM: Software does not respond after optics alignment.
    - SOLUTION: The print-out of alignment results is not saved. The pop-up folder for saving the file might be hidden behind other windows on the screen. The program will respond after you save the print-out.

Troubleshooting comments

Please add comments or observed problems and solutions here.