

Aeris Advanced User Guide

Vendor's service manual is located on Confluence in the [XRD Lab Notebook AERIS](#) and on XRD lab computer Desktop>Aeris>Aeris Diffractometer References. Also on the orange Panalytical USB. Backup PDF's of the service manual are in IODP Share> IODP Technical Manual

- Introduction
 - Instrument Apparatus and Hardware
 - Aeris Start Up
- Sample Preparation and Holders
 - Bulk Powder Backloading
 - Cleaning and Disassembling Sample Holders
 - Small Sample Amount and Slurry/Smears
- Creating/Editing A New Measurement Program
 - Scan Parameters Setup
 - Hardware Setup
 - Changing Hardware
 - Beta Filter
 - Divergence Slit
 - Beam Mask
 - Beam Knife
 - Beta Filter (detector side)
 - Changing Hardware: Configuration confirmation
- Importing A Measurement Program to the Aeris XRD
 - PANalytical recommended measurement programs
 - Bulk Samples Scan Parameters
 - Clay Speciation Scan Parameters
 - Very Low Angle Data Scan Parameters (clays)
- Scanning A Sample in Aeris XRD
- Viewing and Analyzing Scans
- Uploading Data
- QAQC Quality Control Quality Assurance
- Maintenance
- Troubleshooting

Introduction

The X-ray Laboratory onboard the R/V JOIDES Resolution performs diffraction analyses of minerals, clays, and rock powders.

The X-ray laboratory uses the benchtop Malvern Panalytical AERIS Research Edition XRD. Associated software, HighScore Plus allows for powder diffraction analysis of minerals, including peak matching, mineral, and chemical compound identification. The X-ray Laboratory provides the scientist with a quick and reliable tool for mineral identification. In addition, XRD data can be used to determine mineral proportion.

Theory of Operation

A mineral is a three-dimensional structure that forms a crystal lattice. When a focused X-ray beam is directed onto the crystal structure of a mineral, part of the beam is diffracted. X-rays are diffracted differently depending on the atomic composition and arrangement within the crystal lattice. Each mineral has a unique fingerprint that is determined based on a characteristic set of d-spacings (space between adjacent planes of atoms in the crystal lattice). This is a fundamental characteristic of minerals that allow mineral identification through X-ray diffraction. X-rays are generated in a vacuum tube and directed to a powdered sample, when the X-rays hit the powdered sample, they are diffracted onto a detector. The X-ray detector then converts the signal to a count rate. The angle between the X-ray tube, sample, and detector are varied during measurement to produce an X-ray scan. Using the angle between the X-ray tube, sample, and detector (2θ , which can be measured) and the wavelength of a generated X-ray beam (λ , which is known based on the material generating the X-rays), the scientist can determine the d-spacings by using Bragg's law: $n = 2d\sin\theta$.

n = order of the diffracted beam

λ = wavelength of the incident X-ray beam

d = distance between adjacent planes of atoms (d-spacings)

θ = angle of incidence of the X-ray beam

The d-spacings generated by an X-ray scan provide the unique fingerprint of the mineral or minerals present in the sample.

Instrument Apparatus and Hardware

- Benchtop Malvern Panalytical AERIS Research Edition X-ray Diffractometer (XRD)
- Goniometer
 - The goniometer is the core of the diffractometer. The goniometer radius is 145 mm. The minimum step size for the angle of incidence (θ) and the scattering angle (2θ) is 0.001°.
- X-ray Source, Copper (Cu)
 - The X-ray tube is made of ceramic. The anode is at a maximum of 40 kV above ground potential and is cooled with a glycol mixture of 25% glycol and 75% water.
 - The window of the X-ray tube is made of beryllium and is 300 μm thick.
- PIXcel1D-Medipix3 detector
 - Soller slits
 - Beta filter
 - Divergence slits
 - Beam mask

- Beam knife
- Si standard
- Corundum standard
- Sample Holders

Safety

When properly operated within the specifications, the radiation level at a distance of 10 cm from the outside surface of the instrument is less than 1 $\mu\text{Sv/h}$.

The cover has a door lock to prevent accidents. As a safety measure, the instrument will not open the shutter of the X-ray tube if the cover is not correctly closed and locked. You cannot open the cover when the shutter is open, when a goniometer arm moves, or when the instrument initializes.

You **can** open the cover while the X-ray warning lights are on. The shutter of the tube remains closed which makes it safe to do so. The instrument will not allow you to run a sample or open the shutter while the lid is open (*Figure 1*).



Figure 1. Aerial touchscreen safety information. The closed circle icon on the screen indicates when the shutter is closed and it is safe to open the lid.

The "X-RAYS ON" lights on the top corners of the instrument and 2 more on the inside of the cover give a visual warning when high tension is applied to the X-ray tube. The "X-RAYS ON" lamps are 'fail safe' lamps and they are part of the safety circuit. The result is that if one of the lamps fails, no high tension can be applied to the instrument.

When you turn the HT key switch clockwise, the HT generator goes on. If the instrument is energized and the safety loops are closed, the HT generator is automatically set to 40 kV, 15 mA or 40 kV, 7.5 mA.

When the HT generator is in the ON condition, the indicators are as follows:

- The **kV** and **mA** indicators on the touchscreen show the settings (see *Figure 1*)
- The four X-RAYS ON lamps on the corners of the instrument are on.

When you turn the HT key switch counter-clockwise, the HT generator goes off.

The HT key switch is also used to reset the Xsafe system. If the HT key is removed from the instrument and an unsafe condition is sensed, you cannot switch the high tension on again.

NOTE: The HT key can be removed and kept in a safe place.



WARNING IONIZING RADIATION

X-RAYS ARE HARMFUL. THE INSTRUMENT PRODUCES X-RAYS WHICH CAN BE DANGEROUS TO HEALTH IF THE PROPER PRECAUTIONS ARE NOT TAKEN.

IT IS IMPORTANT FOR THE HEALTH AND SAFETY OF THE USER THAT THE RECOMMENDATIONS GIVEN IN THE INSTRUMENT MANUALS ARE CAREFULLY OBSERVED.

LOCAL SAFETY REGULATIONS MUST BE STRICTLY COMPLIED WITH.



WARNING TOXIC MATERIAL

X-RAY TUBE WINDOWS AND DETECTOR WINDOW CONTAIN BERYLLIUM.

BERYLLIUM AND COMPOUNDS OF BERYLLIUM (FOR EXAMPLE, BERYLLIUM OXIDE) ARE POISONOUS.

- DO NOT TOUCH, SWALLOW OR BREATHE IN BERYLLIUM.
- DO NOT GET BERYLLIUM ON YOUR BARE SKIN. ALWAYS WEAR GLOVES WHEN YOU TOUCH ITEMS THAT CONTAIN BERYLLIUM.
- IF THERE ARE DUST OR FUMES OF BERYLLIUM, USE A DUST MASK AND PROTECTIVE DISPOSAL OF BERYLLIUM MUST OBEY ALL APPLICABLE LOCAL REGULATIONS.

Aeris Start Up

The main power switch is on the rear lower right-hand side of the instrument (*Figure 2A*). This can generally be left on. The unit is plugged into the UPS power box and that should remain on at all times. The vendor recommends we leave the entire instrument and X-ray key switch on all the time. If restarting is necessary to clear an error, there is no need to toggle the key switch. Obviously, this should be done for dry dock, or when it's totally necessary, we can shut everything down. BUT, it's not good to be turned off and on often.

A.



B.

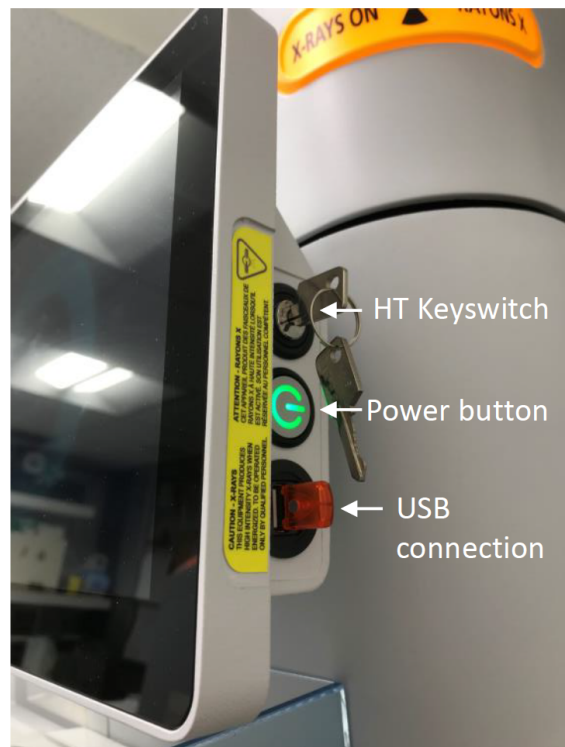


Figure 2. Turning on the Aeris XRD. A) Power switch in the back right of the instruments. B) Push the green Power button on the right of the screen to power on the screen. Turn HT keyswitch clockwise to start the X-ray generator (the key is vertical when it is ON).

1. Make sure at least one sample holder spot is empty before turning on (to allow the sample changer to zero itself).
2. Turn the Aeris front computer on by pressing the power button on the right side of the screen (*Figure 2B*). Let it initialize. Sample changer arm should move and initialize.
3. Screen will prompt you to turn the key-switch, turn it clockwise and the "X-RAYS ON" yellow lights around the instrument will light up. The Aeris screen goes to sleep when not in use, just move the mouse or touch the screen.
4. To turn off the instrument, press the power button on the side of the screen (*Figure 2B*), and then toggle the key-switch counter-clockwise.

Sample Preparation and Holders

Aeris sample holders cannot be interchanged with the Bruker sample holders. The size is slightly different and they are magnetic which lets the machine to know there is a sample in the slot.

See sample preparation instructions on the main XRD page here [X-Ray Diffraction \(XRD\)](#)

Bulk Powder Backloading

Most common method is backloading. The sample holder is in two parts, the top ring (B) and the base (C) which the top ring clips into and is loaded using the sample table (A) which is the small device with a center platform and three round buttons around it (*Figure 3*).

Follow the steps below along with the figures below to pack and assemble the backloaded sample holders (*Figure 4*).

- 1) Place the top ring upside down on the sample prep table and clip it in by pressing the button on the side. Take care not to scratch the center platform.
- 2) Mound the powder up slightly in a conical shape.
- 3) With a blade or glass slide make chopping motions on the powder to help spread it out evenly. Press it down with the cylindrical press.
- 4) Scrape off the excess with a blade or glass slide.
- 5) Brush away excess powder so that the outer ring is clean for attaching the sample holder base.
- 6) Press and clip the sample holder base into the top ring. It should snap in cleanly and easily.
- 7) Turn the entire sample prep table upside down, resting the sample holder base on the countertop, and press the release button on the side to release the completed sample holder. Sample surface should be flat and flush with the edge of the holder. With the brush or kimwipe, clean any powder off the ring surface because that comes in contact with the sample spinner.

Tips:

If the powder sticks to the sample table when you flip it upside down to release, try it again with a piece of weighing paper placed on top of the platform and then clip the top ring in. If you don't have enough power to fill the sample holder, after pressing down with the cylindrical press add one of the cardboard spacers so that the cardboard spacer is flush with the ring top and then clip in the holder base (*Figure 5*). See also [XRD Sample Preparation for a small amount of material](#) for mounting small amount of material. Also note that if there is too much sample the back of the holder will not attach.

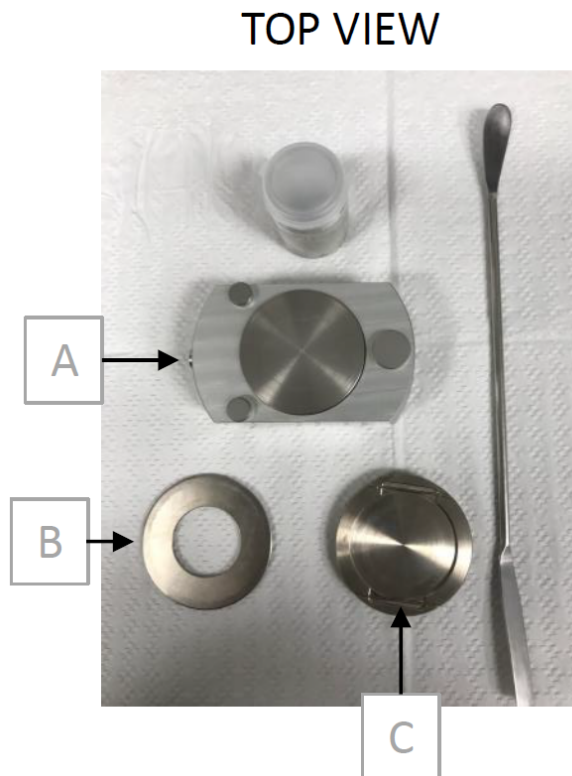
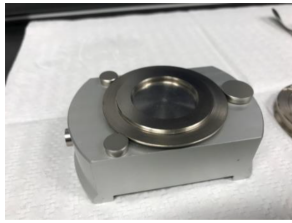
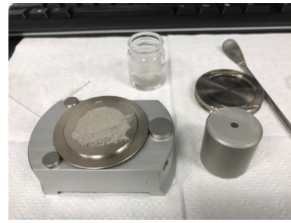


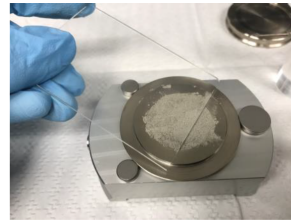
Figure 3. Backloading sample holder (view from the top). A) Sample preparation table, B) Top ring, C) Base which the top ring clips into.



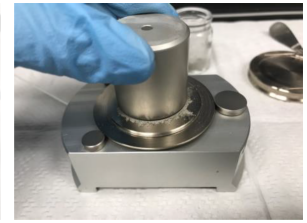
1. Top ring upside down



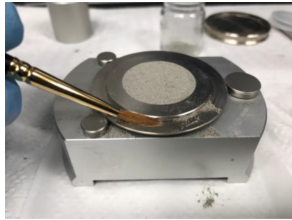
2. Mound powder up



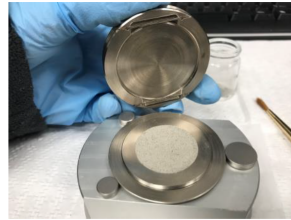
3. Spread sample out evenly and press it down



4. Scrap the excess



5. Brush away excess



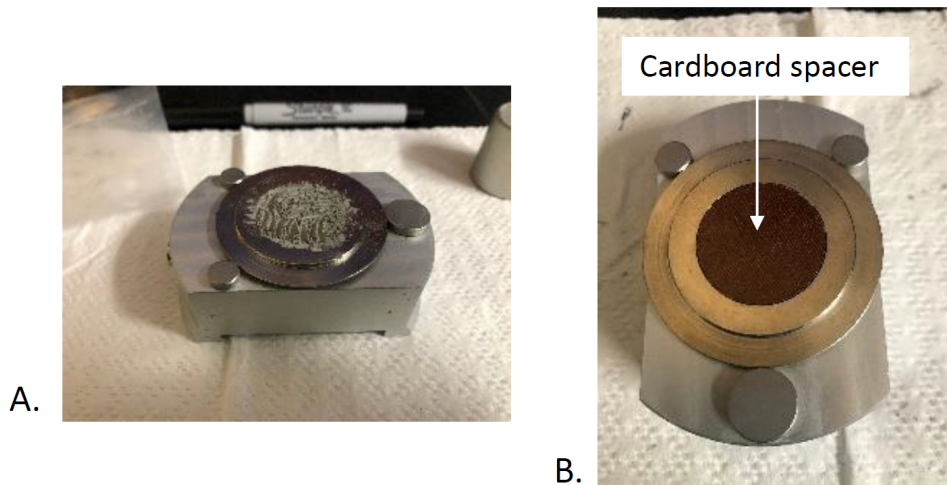
6. Press and clip the base



7. Sample prep table upside down and release sample holder



Figure 4. Sample preparation for Aeris backloading sample holders.



A.

B.

Figure 5. If there is not enough sample powder to fill the holder (A), use a cardboard spacer (B) to fill area

Cleaning and Disassembling Sample Holders

When the measurement is finished, first check the scan to make sure the counts and other parameters are satisfactory. If the scan is acceptable, pour the sample back into the sample bag. Next, remove the top ring. The ring is magnetically connected and should pop off by hand very easily (Figure 6A). Disassemble the rest of the sample holder (Figure 6B). In Figure 6 a cardboard spacer was used. To clean the sample holder, rinse each piece with DI water and then with isopropyl alcohol with kim wipes. Isopropyl alcohol should remove the marker (Sharpie) label on the front of the sample holder. Once clean and dry, store the sample holder back in the box or use it to make next sample.

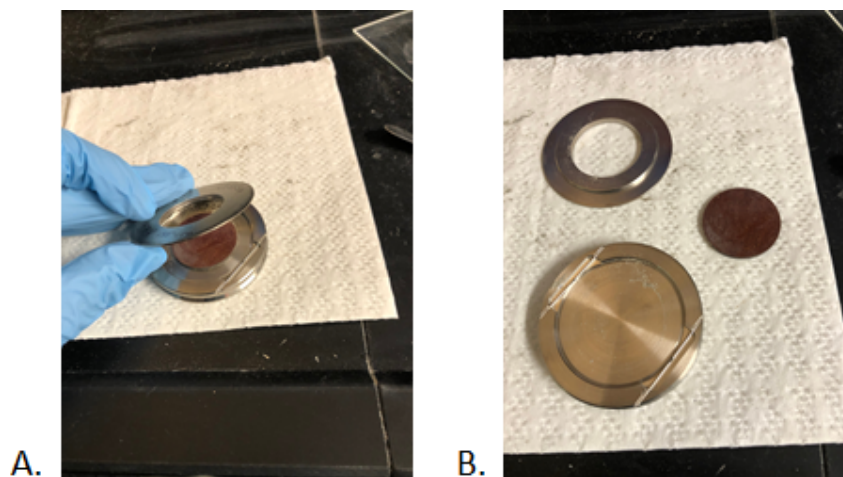


Figure 6. Disassembling the sample holder.

Small Sample Amount and Slurry/Smears

The following technique is modified from the *U.S. Geological Survey Open-File Report 01-041, A Laboratory Manual for X-Ray Powder Diffraction*. For a very small amount of sample material (i.e., end of a tooth pick), samples may be ground to a fine talc-like powder and smeared onto one of the silicone disk inserts or Aluminum spacers. Although not useful for semi-quantitative analysis, this method is useful for rapidly determining mineralogy.

1. Place a small amount of sample powder in the center of a silicone or single crystal quartz disk.
2. Add 2–3 drops of 70 % isopropyl alcohol or distilled water to the sample. **Note:** Isopropyl dries faster than water.
3. Spread the sample to a thin layer using a glass rod. The slurry should be spread evenly across the disk. If there is not enough material to cover the entire disk, concentrate the sample in the middle of the disk where the X-rays will contact the sample.
4. Place the sample in the desiccator to dry before running in the XRD.

Use the sample rings, clip, aluminum spacer, and zero background silicon disks (Figure 7). **Do not scratch the mirrored silicon disks, always use an aluminum spacer so the clip does not scratch the surface.** A tiny amount of powder can be mixed with alcohol to make a slurry and then smeared in the center of the silicon disk.

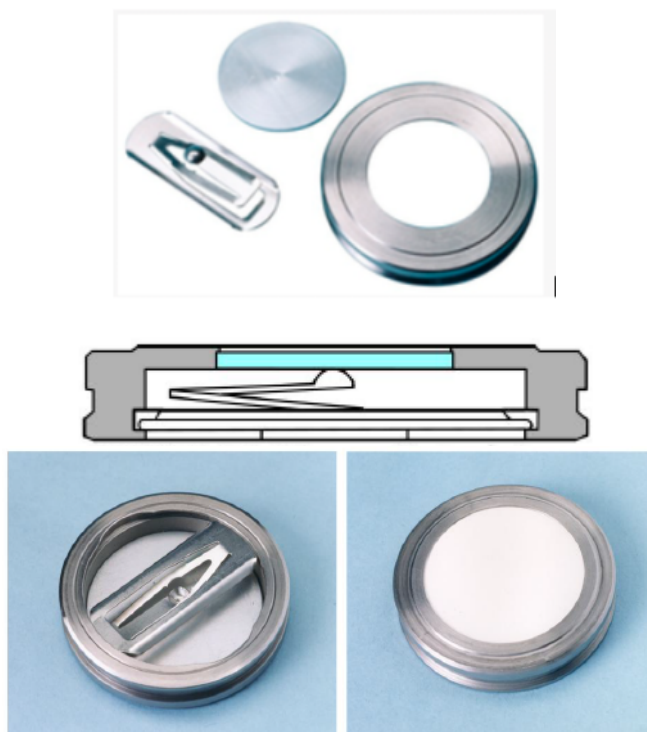


Figure 7. Sample ring, clip, zero background disk and aluminum spacer.

Turn the ring upside down and place the silicon disk in the indentation. Use the flat side or the side with the shallow well for your sample (amount of sample will determine if you use the flat or well), depending on how much you have. Put the aluminum spacer on the silicon disk, then press the clip in and turn it to secure both in place.

If doing heating steps (clay analyses) remove the disc with sample and place on tray to go in the oven. **DO NOT heat the sample holders** they will deform and will no longer be of use.

Don't heat the holders! Only silicon disks can be heated!

Ceramic disc: Ceramic disc are specifically for clay separations. The ceramic is porous and preferentially orientates the clay crystals. We currently have not developed an in-house sample preparation method for using the ceramic disc. Creating our standard slurry and drying them in the desiccator should be ok. Another method is to dry using a vacuum system, which has not been developed on board.

Creating/Editing A New Measurement Program

With Aeris XRDMP creator (*Figure 8*) you can open previous expeditions scan parameters or set up a new parameter file (*Figure 9*). This is where the scan range and step time is set. Name your file with the expedition number for future reference. If starting off with one of the Panalytical predefined programs located in C: > Panalytical > XRDMP Creator > Panalytical Predefined Programs > Aeris and listed below in the section "**Importing a measurement program to the Aeris XRD**", remember to only use the files marked Cu Mineral (copper X-ray source which is what we have). The Co indicates a cobalt source that our Aeris XRD does not have.



Figure 8. Aeris XRDMP Creator (measurement parameter set up) icon



Figure 9. Home Screen: Measurement Program selection window to create or open a measurement program

Scan Parameters Setup

Scan Parameters include the range of scan angles and step time. Vendor supplied scan parameters for Bulk and Clay analyses are listed below in the section "**Importing a measurement program to the Aeris XRD**".

Discuss with your scientist the scan parameters (scan angle range and step time) they wish to use for the samples (*Figure 10*). Direct the scientist to previous expeditions to obtain scan parameters or review previous expedition's files on the XRD PC for examples. The scientists can also decide if they wish a single scan or repeated scans for a sample. Save the measurement program with a name including the expedition number and selected parameters. For instance, in *Figure 10* below, the measurement program is named "X391 5-100deg 1/4 div 21 min" to describe a measurement program used during Expedition 391, with a scan angle range between 5 and 100° 2, a divergence slit of 1/4°, for an estimated time of 21 minutes per sample.

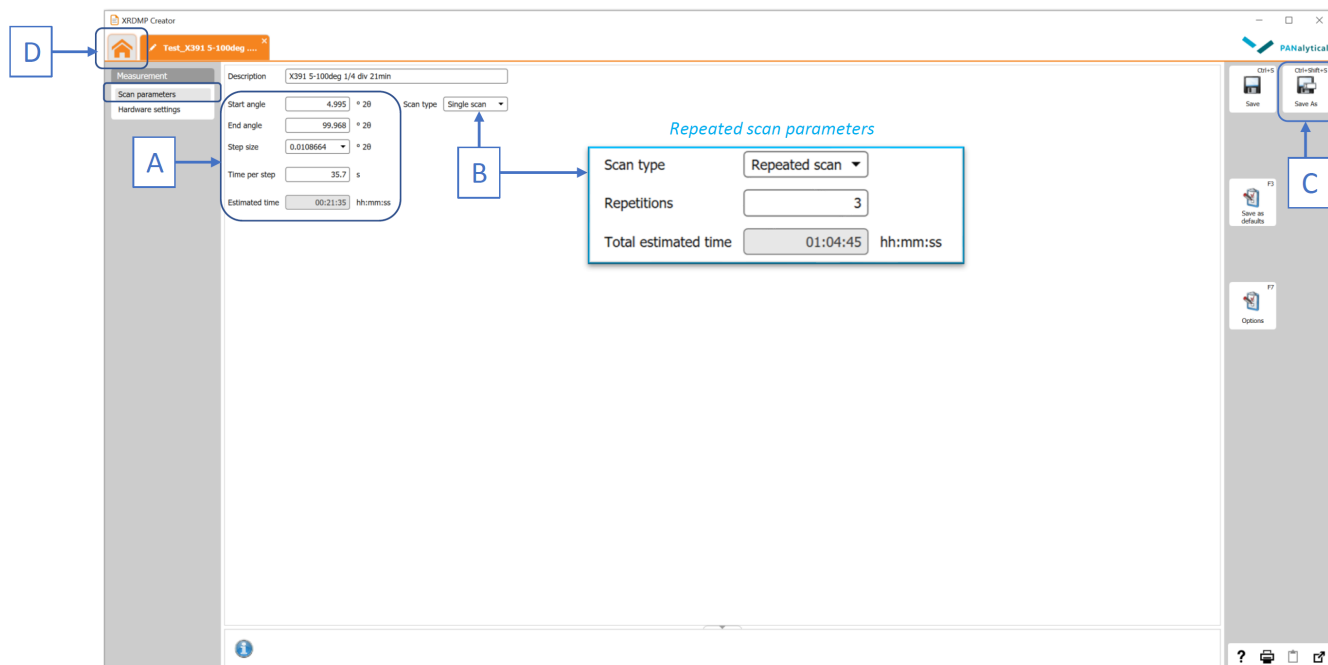


Figure 10. XRDMP Creator. Edit or create new scan parameter files: A. Setting scan angle range and step time, B. Single or Repeated scans (note that the estimated time is per sample), C. Save button, D. Home button

Hardware Setup

The Excel file Aeris Setup Guide ([Aeris Setup Guide.xlsx](#)) helps the user choose the best hardware setup for your scan parameters (Figure 11). Hardware is generally only changed when your scan angles change. For example scanning at angles less than 5° you will need to lower the knife (a.k.a. anti air scatter screen on the Bruker). You will scan at lower angles for clay identification, so switching from bulk sediment analyses (angle range of 5 to 100°) to clay analyses (angle range of 2 to 30°) will require a hardware change. Use the Aeris Setup Guide to determine the best hardware set up.

That being said, if you did not change the hardware, you would not damage the instrument, and the diffractograms may be ok, but they could be better if you follow the suggestions for hardware from the setup guide. Start in the first Excel tab, "Slit Guide" and enter the desired information such as start and end angles, the diameter of the sample holder and the sheet will provide suggestions for recommended hardware options. The other values provided in the Excel sheet are not something we need to change or take into account for our XRD scanning onboard.

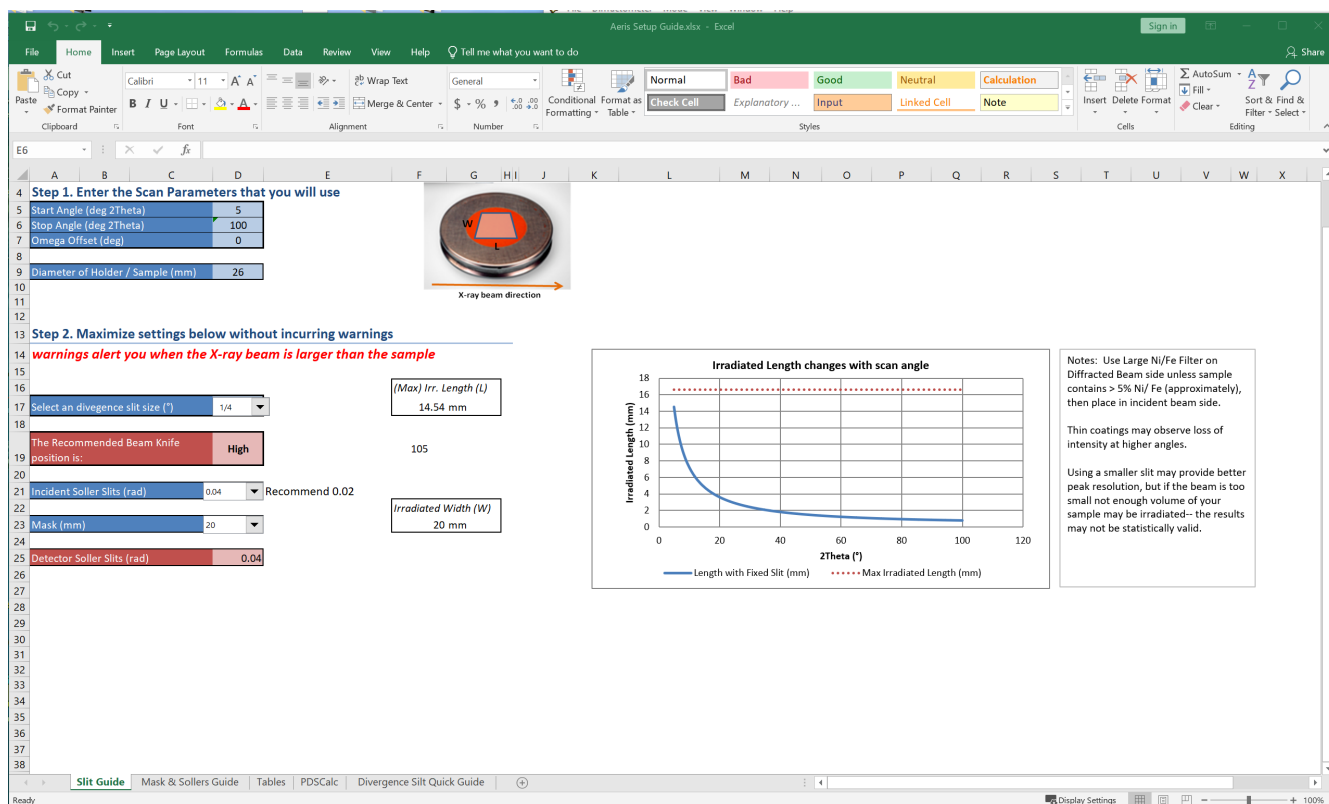


Figure 11. Aeris Setup Guide for hardware. The scan angles will determine the beam knife height and other hardware such as divergent slit.

Changing Hardware

Adjusting the hardware will allow for optimum results. Use the [Aeris Setup Guide.xlsx](#) to obtain the best setup for the scan parameters as discussed above in "Hardware Setup".

If you change the hardware, please remind to update the hardware settings in **XRDMP Creator** as well (Figure 12).

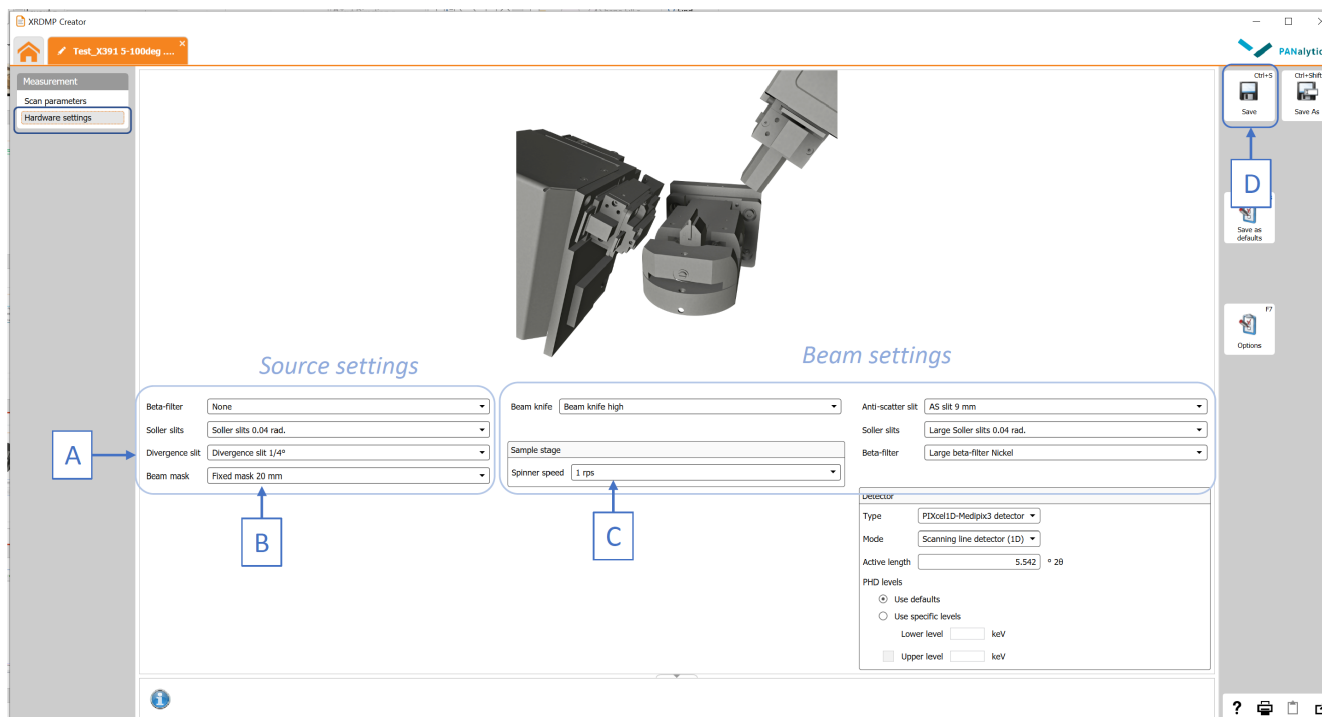



Figure 12. Changing hardware settings in XRDMP Creator: A. Divergence slit, B. Beam mask, C. Spinner speed, D. Save button

To access the internal drives and hardware:

On the Aeris touchscreen, press the 'Menu' button  on the top left corner and press the Advanced mode option (Figure 13). A password is needed to go to the Advanced Mode (Figure 14).

Password is: **panalytical**

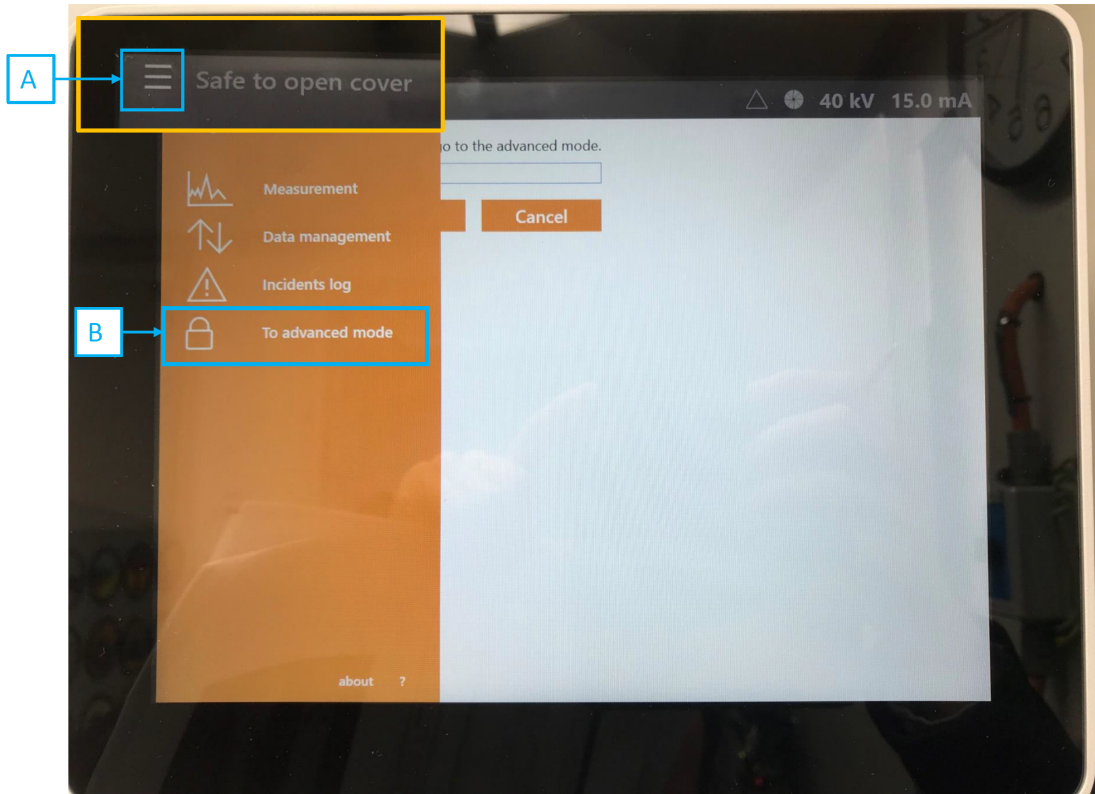


Figure 13. To go to the Advanced Mode. Press 'Menu' (A) then (B). Note that the lock indicates protected access by password.

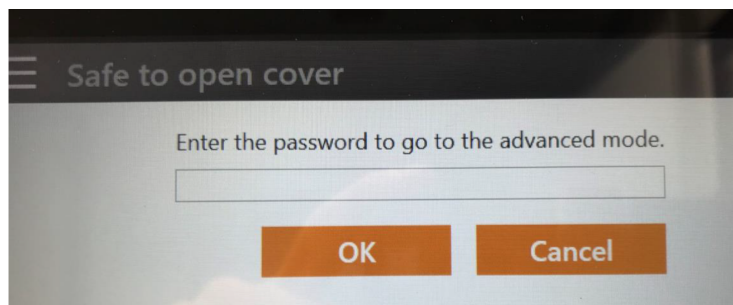


Figure 14. Entering password to go to the advanced mode

In the Advanced Mode, select 'Manual Control' (Figure 15, arrow A), and then 'Unlock Cover' (arrow B). Then lift the cover slowly until it stops on the ceiling. Use canned air to dust off any parts before inserting, the divergence slits tend to get dust in them.

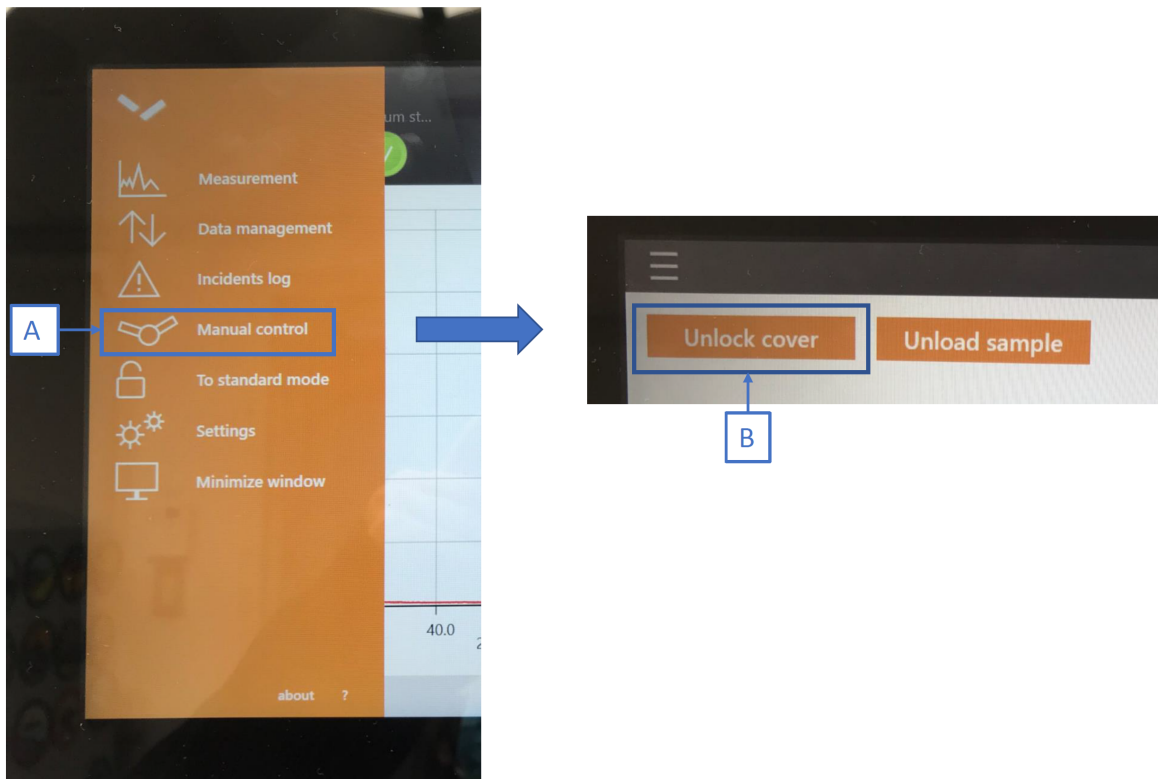


Figure 15. Unlock cover to access the hardware

Beta Filter

Typically a beta filter is not needed on the source (tube) side. Two filters are supplied: a Ni and Cu filter (Figure 16). Ni filter may be used to run the Si standard blank for a system check. If you do need to insert or remove this beta filter it should slide in an out completely freely without resistance and proper placement is sliding all the way in. The writing will be facing the beam knife.

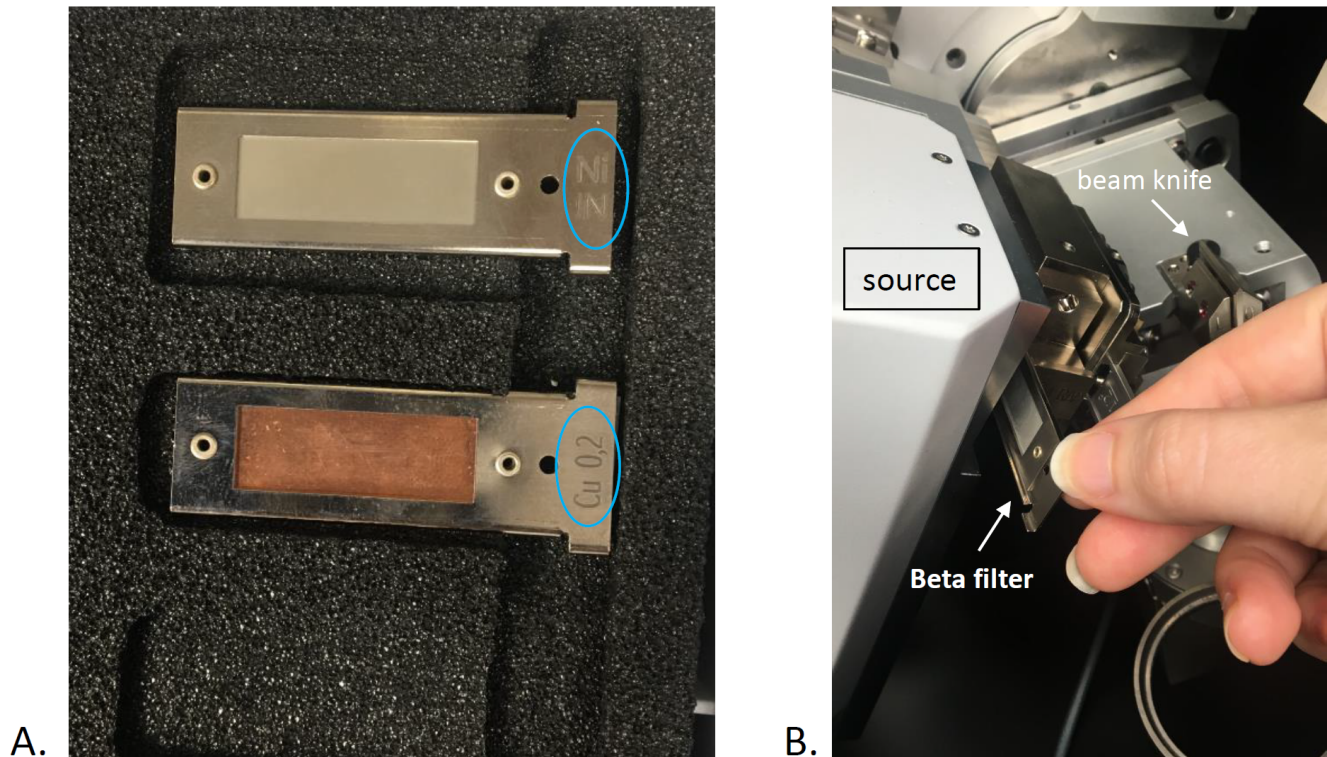


Figure 16. A. Ni and Cu Beta filters, B. Writing (circled in blue in A) facing the beam knife

Divergence Slit

You may need to change the divergent slit to run a low angle scan (*Figure 17*). For instance, a $1/4^\circ$ slit is typically used for bulk samples, and a $1/16^\circ$ for clay speciation. There is writing on both sides of the slit, but when looking at it from the side of the beam knife the writing will be right side up. Slide it in until the first small click. It is possible to push it further and feel another click but that is too far and should be backed out.

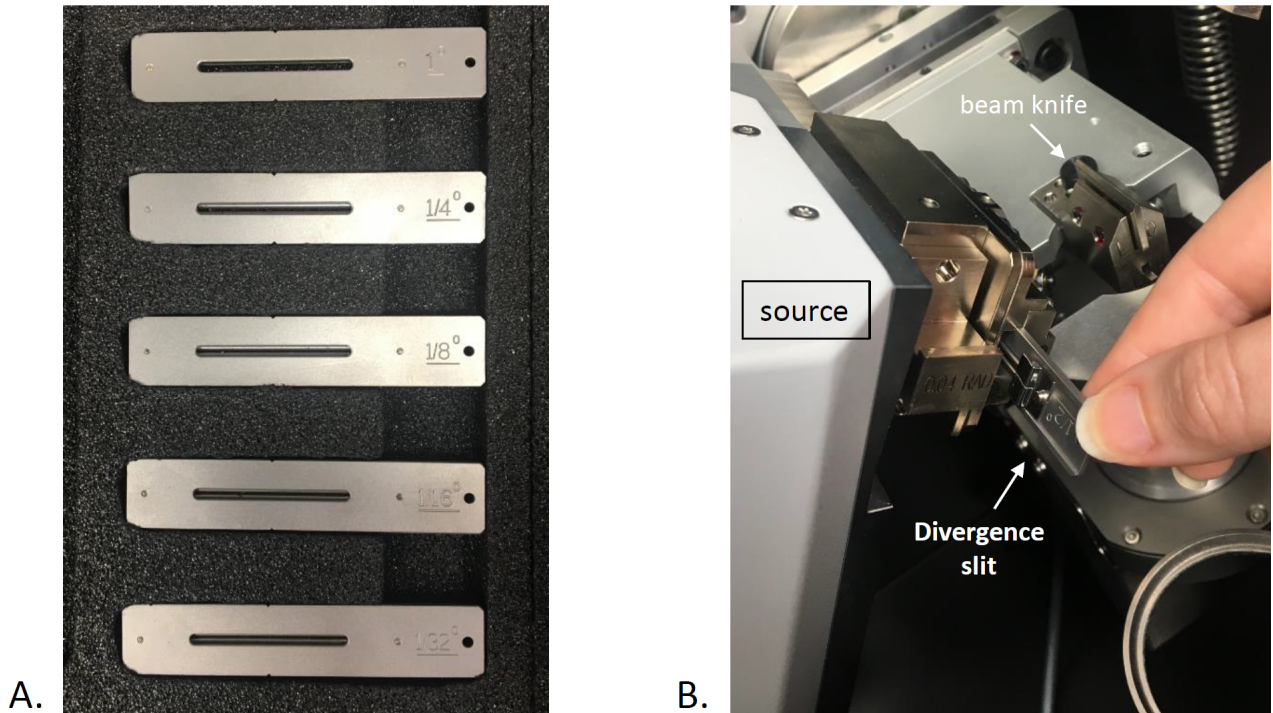


Figure 17. A. Set of divergence slits, B. the writing " $1/2^\circ$ " appears upside down and this is correct.

Beam Mask

Beam mask may need to be changed if the diameter of the exposed sample is different in different sample holders (*Figure 18*). For classical measurement with backloaded samples, a beam mask of 20 mm is usually used. The writing should be facing the beam knife (*Figure 18B*). It should feel tight when inserting or removing, you need to put your finger in the opening to pull it out. When inserting it should slide all the way down, no clicks or anything but you won't be able to push it too far. The picture on *Figure 18C* is what it looks like fully inserted.

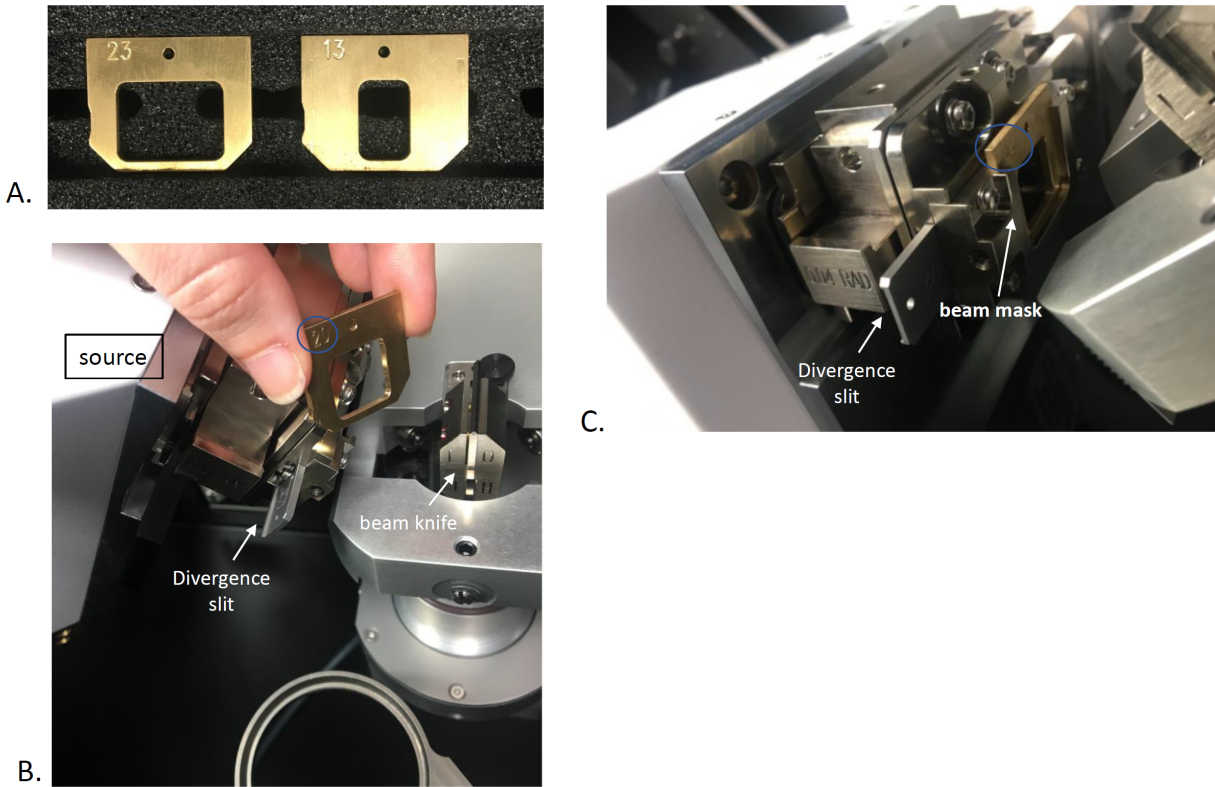


Figure 18. A. Beam masks, B. Inserting beam mask, C. Fully inserted beam mask with diameter information facing the beam knife.

Beam Knife

Beam knife (Figure 19A) reduces air scatter and noise in the detector, it may need to be changed for low angle scans (e.g., for XRD on clay separations) or system checks. Knife goes in the position you can read (Figures 19B and 19C).

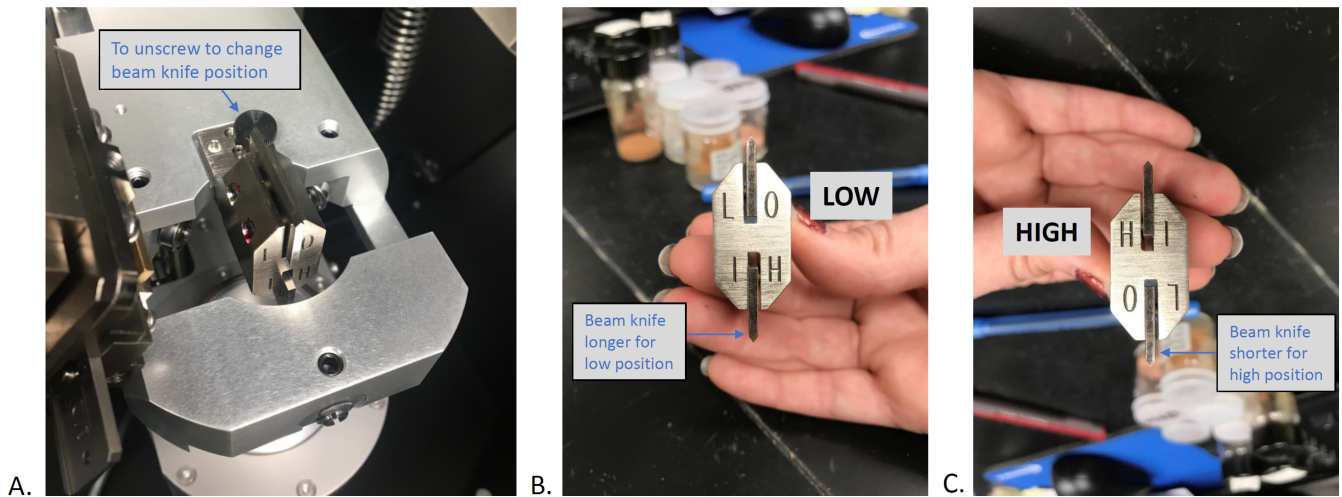


Figure 19. A. Beam knife in the Aerie in low (LO) position. B. Beam knife in LO position. C. Beam knife in high (HI) position.

Beta Filter (detector side)

This Ni filter should **never** need to be removed except when doing a Factory Acceptance Test or instructed to do so by PANalytical service techs for troubleshooting. When sliding in and out it should slide freely. When inserting you will feel a click in the middle and continue to push all the way in.

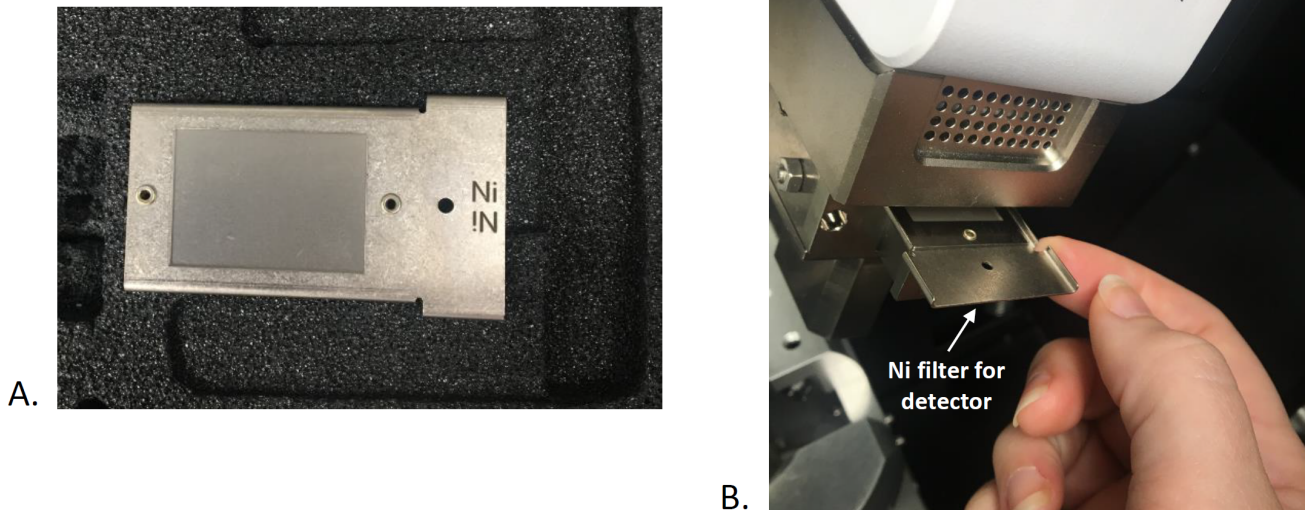


Figure 20. A. Ni beta filter for the detector. B. **NEVER** remove from the detector unless necessary.

Changing Hardware: Configuration confirmation

When you change a measurement program which uses different hardware, for instance the divergence slit changes from bulk sample to clay separation measurement, the Aeris computer will ask to confirm the hardware exchange (Figure 21A). Open the cover (see Figures 13-15) and change the optical components indicated by a double-arrow. When it is done, press 'Continue' on the touchscreen (Figure 21B).

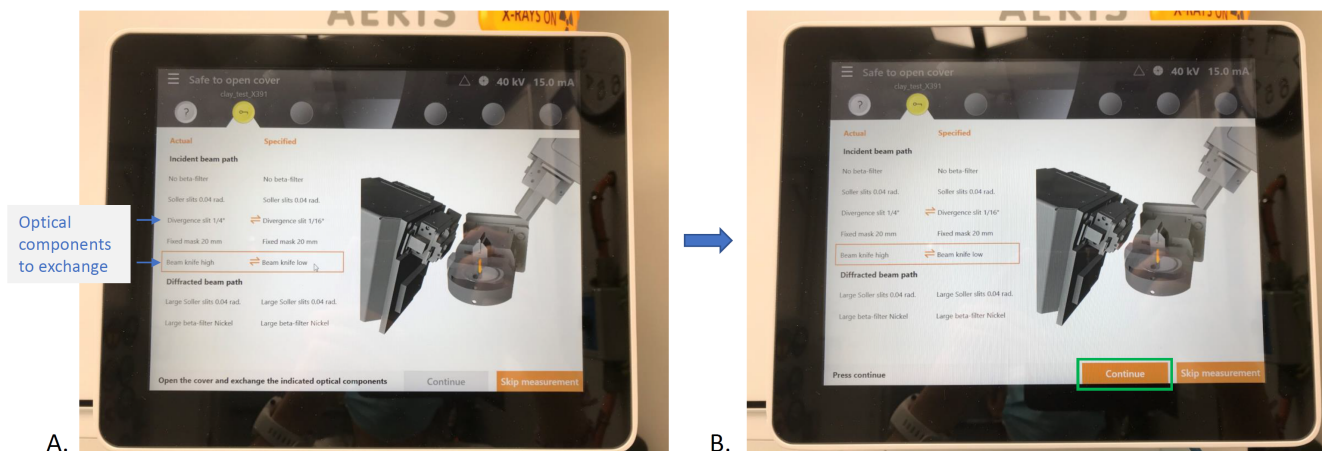



Figure 21. Confirmation of exchanged hardware in the Aeris XRD.

Importing A Measurement Program to the Aeris XRD

1. Navigate to C:/ PANalytical/XRDMP Creator/ to open an existing file on the XRD computer to edit or rename by clicking OPEN. OR open the XRDMP creator software and open a previous parameter file. See 'Creating/Editing A New Measurement Program' and Figure 10 above. Make any needed edits to the start and end angles and click 'Save As' (Figure 10). Rename for the current expedition.
2. Save the creator parameter file onto a USB drive. The USB can then be inserted into the side of the Aeris touchscreen (see Figure 2B).

3. Click on the **Menu** button  and select '**Advanced Mode**'. The password is **panalytical**
4. Click on Menu again and select '**Data Management**' (Figure 22, arrow A). Select the '**Import Programs**' tab (Figure 22)
5. Source should auto populate as the USB drive (Figure 22, arrow B). Select the measurement program (arrow C), and then click '**Import Programs**' (arrow D)
6. Click **Menu** to return to Measurement screen.

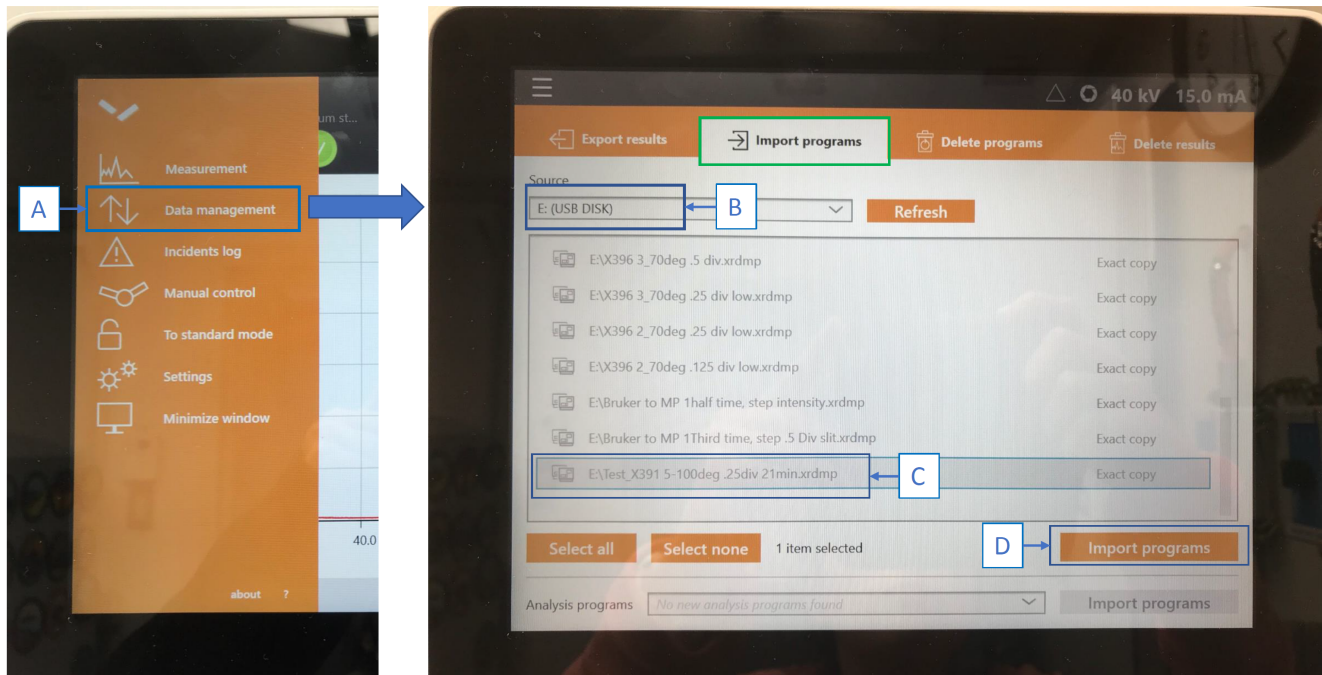


Figure 22. Importing a program from a USB drive to the Aeris XRD.

PANalytical recommended measurement programs

PANalytical recommended measurement program settings are as follows. *This is from a document named AERIS_Suggested_Config.docx, from the factory/ installation. Please update your parameters as necessary.*

Based on this document, for bulk samples the scan rate is 40 sec/step (leading to a 17-min scan from 5 to 90° 2) and you might use a scan rate as slow as 200 sec/step for really challenging samples (or repeat the scan at 40 sec/step five times and sum the results).

Bulk Samples Scan Parameters

- Start angle: 5° 2
- End angle: 90° 2
- Step Size: 0.0108664° 2
- Time per step: 40 sec/step (longer as needed)
- Incident (Tube) Side
- Beta Filter: None
- Soller Slits: 0.04 rad
- Divergence Slit: 1/4 °
- Beam Mask: 20mm
- Sample Stage
- Beam Knife: Beam Knife high
- Spinner speed: 1 rps
- Diffracted (Detector) Side
- Anti-Scatter Slit: AS Slit 9mm
- Soller slits: 0.04 rad
- Beta Filter: Large beta filter Nickel
- Detector
- Type: PIXcel1D-Medipix3 detector
- Mode: Scanning line detector (1D)
- Active Length: 5.542° 2
- PHD levels: Use defaults

Clay Speciation Scan Parameters

- Start angle: 2° 2
- End angle: 30° 2
- Step Size: 0.0108664° 2
- Time per step: 40 sec/step (longer as needed)
- Incident (Tube) Side
- Beta Filter: None
- Soller Slits: 0.04 rad
- Divergence Slit: 1/16 °
- Beam Mask: 20mm
- Sample Stage
- Beam Knife: Beam Knife low

- Spinner speed: 1 rps or NONE (if peaks seem broad because sample is wobbling)
- Diffracted (Detector) Side
- Anti-Scatter Slit: AS Slit 9mm
- Soller slits: 0.04 rad
- Beta Filter: Large beta filter Nickel
- Detector
- Type: PIXcel1D-Medipix3 detector
- Mode: Scanning line detector (1D)
- Active Length: 5.542° 2 for fast (8 min) or 0.978° 2 for slow (40 min) with better background
- PHD levels: Use defaults

Very Low Angle Data Scan Parameters (clays)

- Start angle: 1 to 0.5° 2
- End angle: 30° 2
- Step Size: 0.0108664° 2
- Time per step: 40 sec/step (longer as needed)
- Incident (Tube) Side
- Beta Filter: None
- Soller Slits: 0.04 rad
- Divergence Slit: 1/32 °
- Beam Mask: 20mm
- Sample Stage
- Beam Knife: Beam Knife low
- Spinner speed: NONE (if peaks seem broad because sample is wobbling)
- Diffracted (Detector) Side
- Anti-Scatter Slit: AS Slit 9mm
- Soller slits: 0.04 rad
- Beta Filter: Large beta filter Nickel
- Detector
- Type: PIXcel1D-Medipix3 detector
- Mode: Scanning line detector (1D)
- Active Length: 0.5 to 0.109° 2 (make the active length slightly smaller than ½ the start angle)
- PHD levels: Use defaults

(Note: the Bruker instrument should not be used to scan at angles < 5° 2)

Scanning A Sample in Aeris XRD

Place sample into one of the six slots on the sample changer. Be careful not to touch the center arm that comes in and out of the shutter door (Figure 23A).

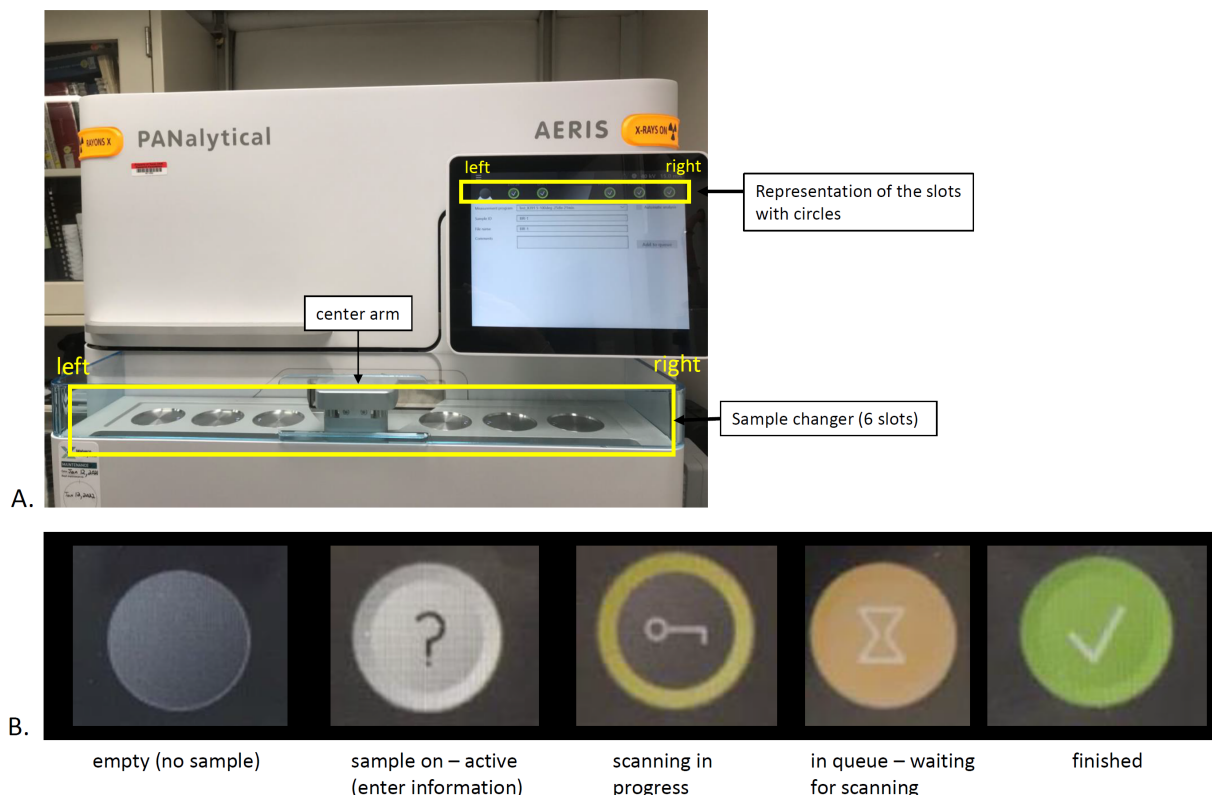



Figure 23. A. PANalytical Aeris XRD in the Xray lab. B. How to 'read' the slots

1. Click on the MENU button  in the top left of the Aeris touchscreen and go to **Measurement** (Figure 24, arrow A).
2. The 6 circles along the top of the screen correspond to the six sample slots and have meanings (Figure 23B). Click on an active one to get started (with a question mark). You can't click on one that is empty (grey) (Figure 23B).
3. Select the appropriate measurement program in the dropdown menu (Figure 24, arrow B).
4. Use the barcode scanner (on the right side of the Aeris) to scan the sample ID label into the sample ID field, or type it in with the keyboard (Figure 24, arrow C). The file name will auto populate. Add any comments as necessary such as clay separation, heated, standard, test. NOTE: If you are running clay samples you will end up with multiple runs that have the same sample info if you do not add extra info in the sample name. To make sure you can differentiate this in the database you must add extra text **before** the Text_ID so that it looks like the examples below. Do this step on the Aeris touch screen so that it gets included in the metadata of the file. See '**Uploading Data**' below for more information on file format.
5. Click **Start/Add to Queue** and the program will run right away (Figure 24, arrow D). The screen changes to a live view of the scan in progress (Figure 25A). If the sample is not in the sample changer you will not be able to click Start.
6. While the instrument is running the first sample, click on another circle where there is a sample and add the sample information and click **Add to Queue** (Figure 24, arrow D).
7. When the sample has finished running, click on **Next Measurement** (Figure 25B, arrow C) and you can add the next sample .

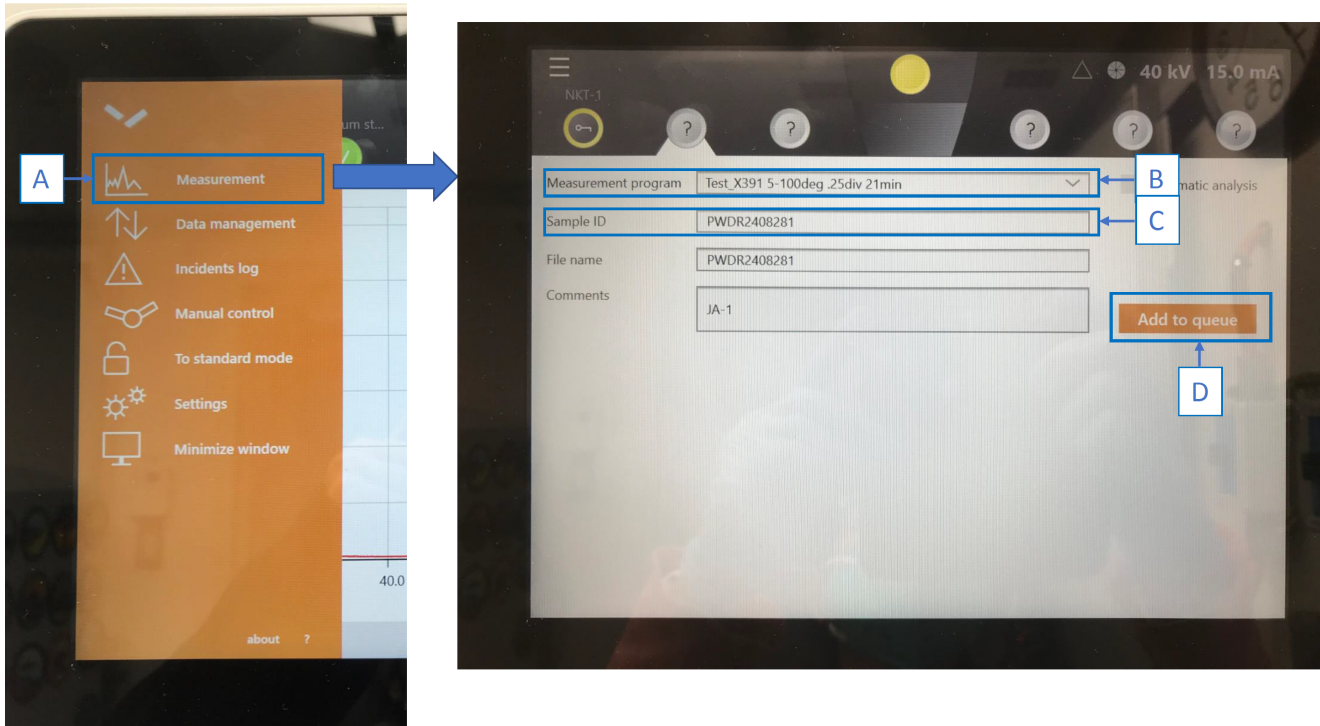


Figure 24. Starting a scan on the Aeris XRD. Sample ID set up screen.

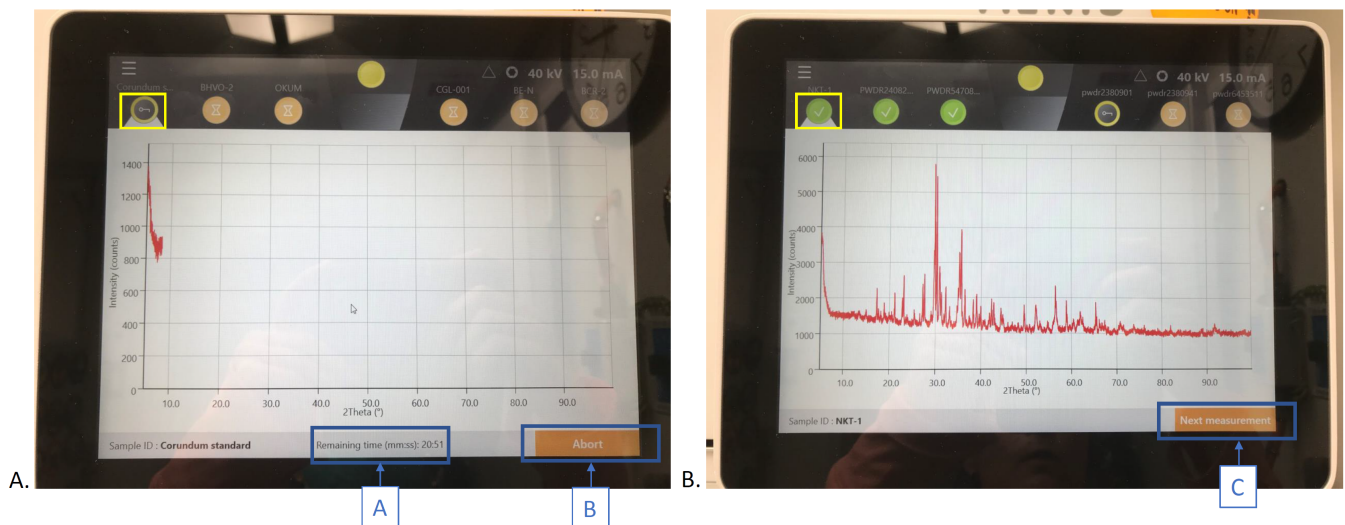


Figure 25. A. Live view of sample diffractogram (note the remaining time and the Abort button). B. Finished scan and Next measurement option.

Tips:

- Once a sample is added to the Queue you cannot cancel it. If you decide there is a mistake and you don't want to run a sample that has already been added to the queue, simply remove the sample from the sample changer. When the instrument gets to the empty slot it will show a warning that no sample is present and skip to the next sample. Then you can place a sample back in the slot, click on the corresponding circle, and enter the new information.
- You cannot enter any sample information into a slot that doesn't have a sample holder placed in it. The sensor must sense a sample holder and then you will be able to add information and add it to the queue.

If the instrument is in Manual Mode: Due to the malfunctioning sample changer, the Aeris has been placed into "manual mode" by the service reps. You can bypass the sample changer and run one sample at a time by placing it gently on the center arm (Figure 26) and then filling out the measurement fields on the screen as described above. The arm swings out of the chamber whenever the screen is activated, take care not to bump it. In this state, the screen permanently shows 1 active error "Motion control error on the sample changer gripper axis".



Figure 26. Manual Mode, one sample at a time placed directly on the sample changer arm.

Viewing and Analyzing Scans

When the scan is finished, the data file automatically transfers to the XRD lab computer. Make sure that the data are saved in the correct folder (Figure 27) and can be found in **C:\XRD Data\XRD data**. Extension is **.xrdml**.

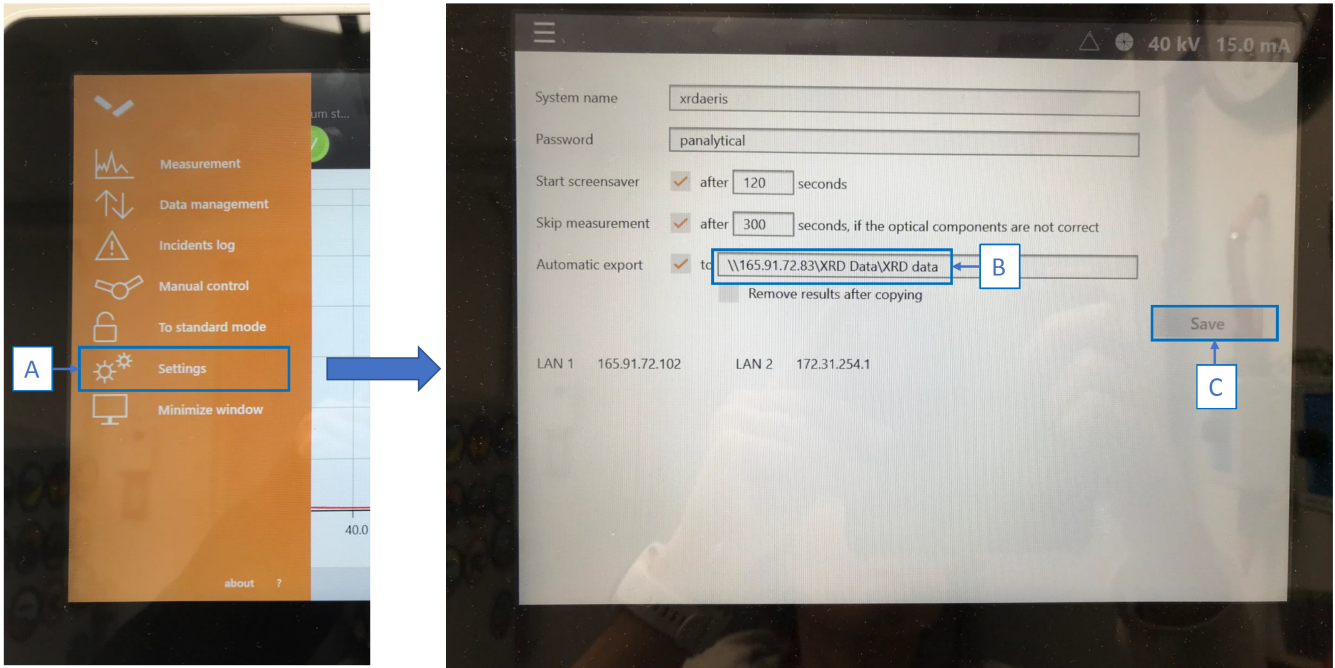


Figure 27. Directory to transfer data from the Aeris to the XRD lab computer.

Two file types, **.xrxml** and **.jpg**, must be uploaded per sample.



To create a **.jpg** file, open **Data Viewer**. Click 'File', then 'Open' (Figure 28, arrow A). Select the desired **.xrxml** file, for instance in **C:\XRD Data\XRD data** (Figure 28, arrow B). The diffractogram will open. This can be a quick and easy way to print and save the raw image. You can open more files and view the scans on top of each other (Figure 29).

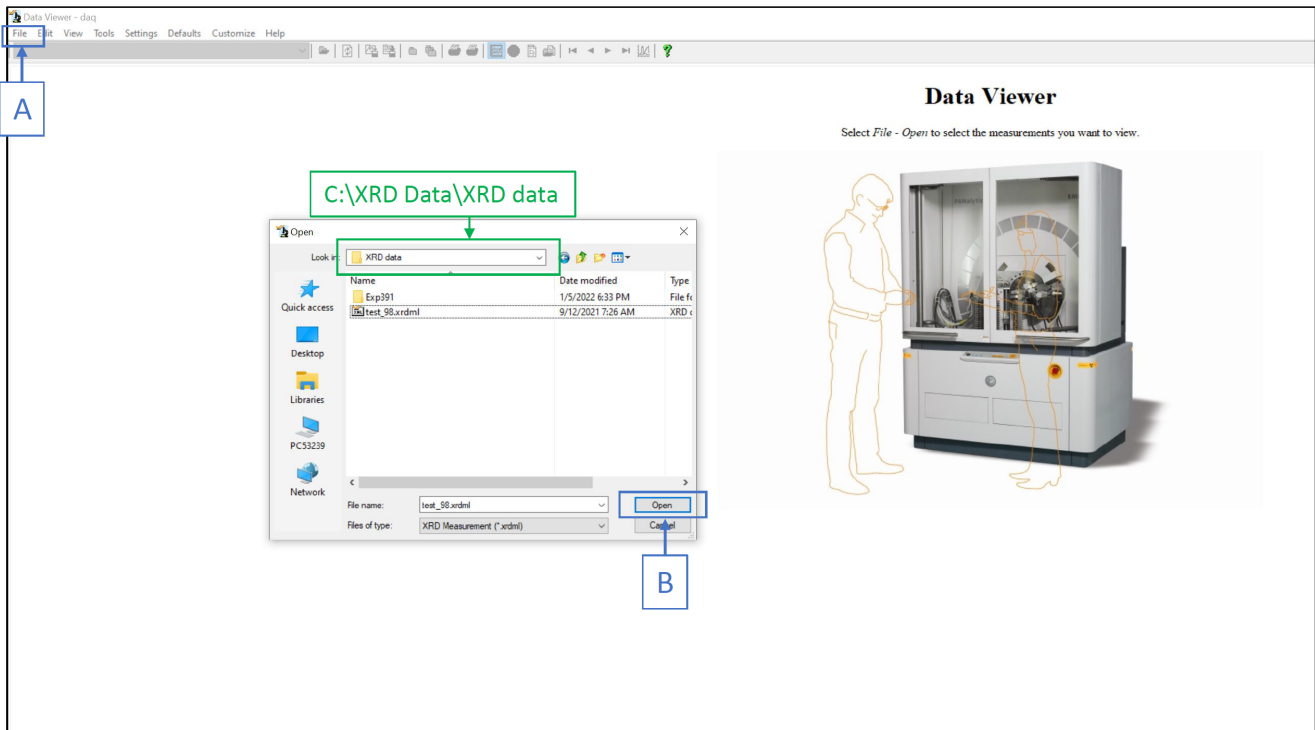


Figure 28. Opening a **.xrxml** file with Data Viewer.



Use the snipping tool to make a screenshot of the diffractogram. Save as .jpg. Click on the page icon (circled in purple in Figure 29), to view the complete report of parameters used in the scan. Scientists may want this information for their methods.

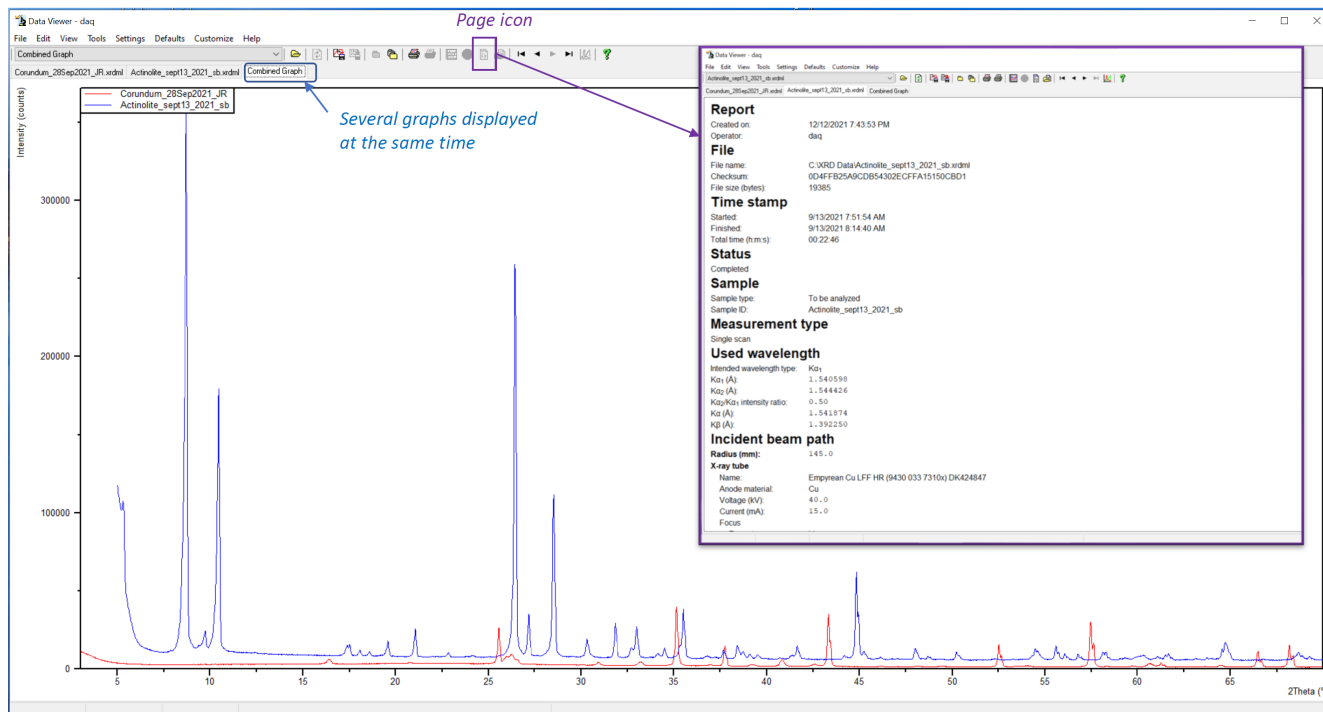


Figure 29. Viewing diffractograms with Data Viewer and accessing report of scan parameters for a sample.



For peak identification and advanced processing, use **HighScore** software. See the [HighScore Quick Start Guide](#). HighScore is available to the scientists via a virtual computer onboard. The IP address is 165.91.150.141, computer PCV0001.

Login: daq

Password: daq

It is possible to view and analyze Aeris diffractograms with **DIFFRAC.EVA** software (used for analyzing diffractograms obtained with the Bruker). For that, .xrdml files need to be converted into .xy files with **Data Viewer**. In **Data Viewer**, click 'File' then 'Convert'. A window pops up (Figure 30). Select the desired directory. Choose the conversion format by checking the box 'X-Y table' (.xy). Click 'Convert'.

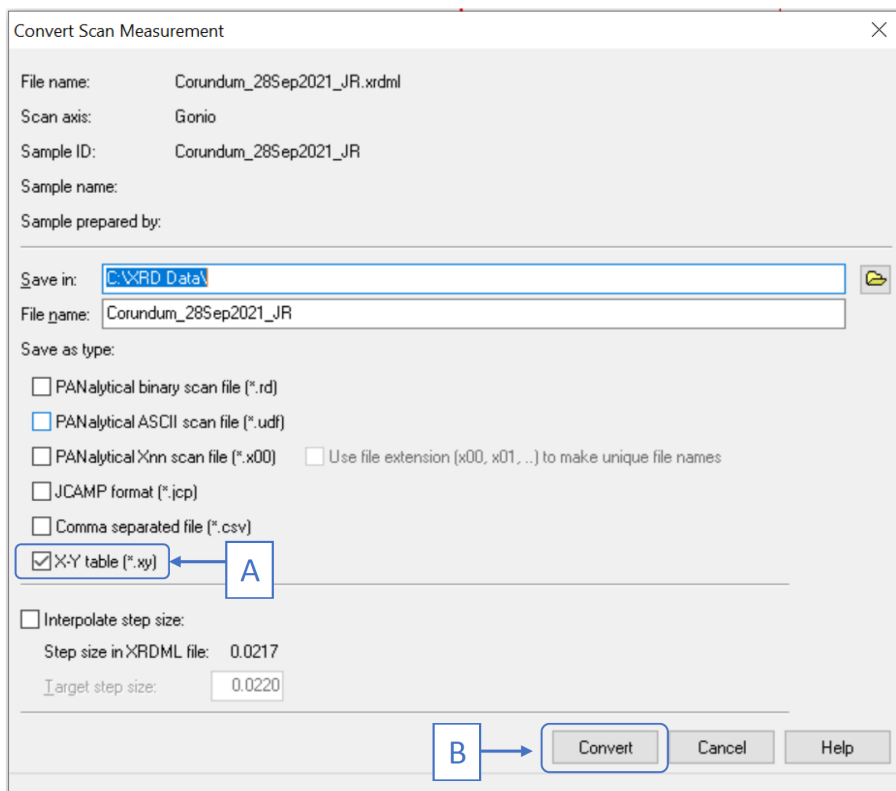
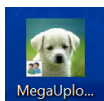


Figure 30. Converting Aeris file to be opened with DIFFRAC.EVA

Uploading Data

Two file types, **.xrml** and **.jpg**, are uploaded per sample. File name must be in the following format to be recognized by MUT and uploaded: U1536B_5H3_79_81_CYL19622183. The files are located by default in C:\XRD Data\XRD data. You can copy the data to C:\DATA\IN to have all Aeris and Bruker XRD scans in a same folder.



Upload files to the database through MUT. To be able to correctly upload the files to the database, make sure to have **both .xrml and .jpg files** in MUT with matching file names. The valid files have a green tick in the Status column (Figure 31). **Both .xrml and .jpg should be in MUT** to get the green tick. The files with a purple question mark have an error and cannot be uploaded. In Figure 30, the files in the purple box do not have the proper correct format. Select the correct project (Figure 31, arrow A) and upload to MUT (arrow B). Refresh when new samples are measured (arrow C).

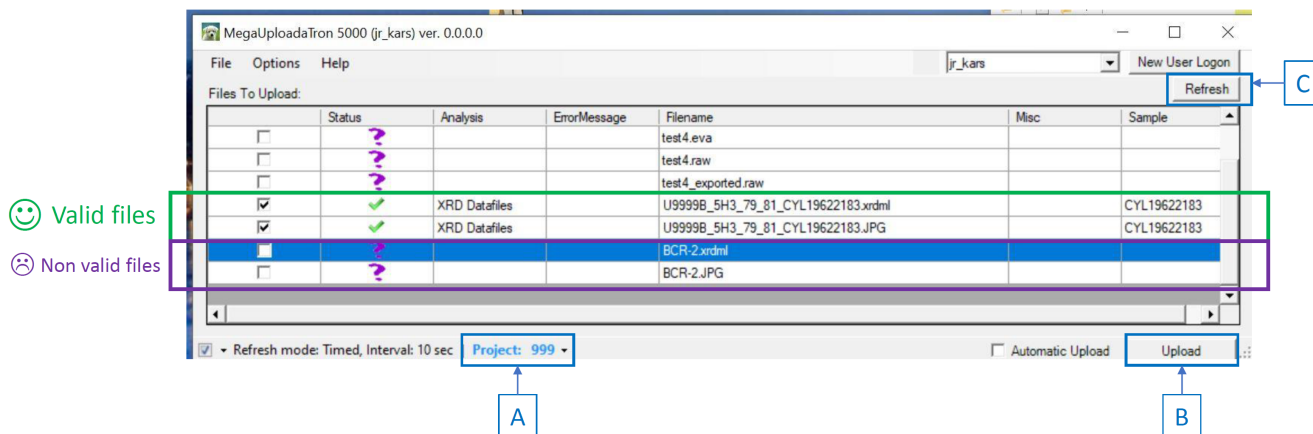


Figure 31. MUT window. Note the valid files with a green tick and non correct files with a purple question mark.

After uploading, MUT will transfer the files to either [C:\XRD Data\archive](#) (if you choose to save data in the default folder) or [C:\DATA\ARCHIVE](#) (if you choose to copy data in the same folder as for the Bruker). Organize the archived files by Site and Hole. At the end of expedition copy these into DATA1 which is used as a database backup (speak to programmer if you cannot find DATA1).

As noted above: If you are running clay samples you will end up with multiple runs that have the same sample info. To make sure we can differentiate this in the database you must add extra text **before** the Text_ID so that it looks like the examples below. Do this step on the Aeris touch screen so that it gets included in the metadata of the file. MUT will recognize the files written this way as long as there is a .xrdml and .jpg with matching file names.

There is a character limit on the Aeris so you may shorten the extra text to something like [heat](#), or [glyc](#).

Examples: U1565A_1R1_3_4_ [clay](#)_WDGE11053881.xrdml
 U1565A_1R1_3_4_ [heated](#)_WDGE11053881.xrdml
 U1565A_1R1_3_4_ [glycolated](#)_WDGE11053881.xrdml

On LIMS, the .xrdml file will be uploaded in the 'Raw file link' column ([Figure 32](#)). The 'UXD file link' column will be left empty because this column is for converted .raw files of the Bruker. Test instrument name is AERIS.

X-Ray Diffraction (XRD)

<< < > >> Show up to 200 rows.

Exp	Site	Hole	Core	Type	Sect	A/W	Top offset on section (cm)	Bottom offset on section (cm)	Top depth CSF-A (m)	Bottom depth CSF-A (m)	Top depth CSF-B (m)	Bottom depth CSF-B (m)	Viewable file (PDF or PNG) link	Viewable filename	Raw file link	UXD file link	Timestamp (UTC)	Text ID	Test No.	Sample comments	Test comments	Result comments
999	U9999A	1	H	1	W		51.5	53.5	0.515	0.535	0.515	0.535	60353041	U9999A_1H1_52_54_WDGE11241591.JPG	60353031		2021-12-27 11:01:53	WDGE11241591	120985911			

Reported on Mon Dec 27 2021 11:03:30 GMT+0000 (Coordinated Universal Time)

Figure 32. Aeris XRD files per sample on LIMS

QAQC Quality Control Quality Assurance

The Aeris is provided with its own corundum standard (do not use the Bruker standard). It is a round white disk. It should be kept in its sample holder to avoid getting scratched and damaged. Both corundum and the Si disk can be used to verify that the actual peaks align with theoretical peak angles. Any drifting on the x-axis is uncommon for the Aeris and vendor recommends making any corrections to the x-axis in HighScore and not making any hardware or settings adjustments. It is possible to change the system settings to correct this but only service technicians have access to that.

- Run the [corundum standard](#) at the **beginning, middle, and end of every expedition** and compare to the original file in C:\XRD Data\Aeris QAQC. The sequence to run for QA/QC is [Corundum Standard Scan 5-120deg .25div 28min SB.xrdmp](#). The original data file to compare with is [corundum 1_14_2021_SB.xrdml](#).
- Open the scans on top of each other in **HighScore** and they should line up exactly on top of each other ([Figure 33](#)). Over time the peak intensities may reduce slightly and that is normal. PANalytical service technicians advise that when to replace the X-ray tube is at our discretion if we feel the intensities are too low.
- If the scans do not line up you must figure out how much to correct the x-axis offset in HighScore and that must be done to any data files that might be affected.
- Email PANalytical service team that the x-offset must be corrected and they will be able to remote in to the instrument to make those corrections. See Laboratory Notebook for [service contact info](#).

The Aeris counts are lower than the Bruker's, this is normal and due to higher resolution. In order to make the counts higher, increase the size of the divergence slits (i.e., decrease resolution) and increase run time. The important part are the peaks' positions.

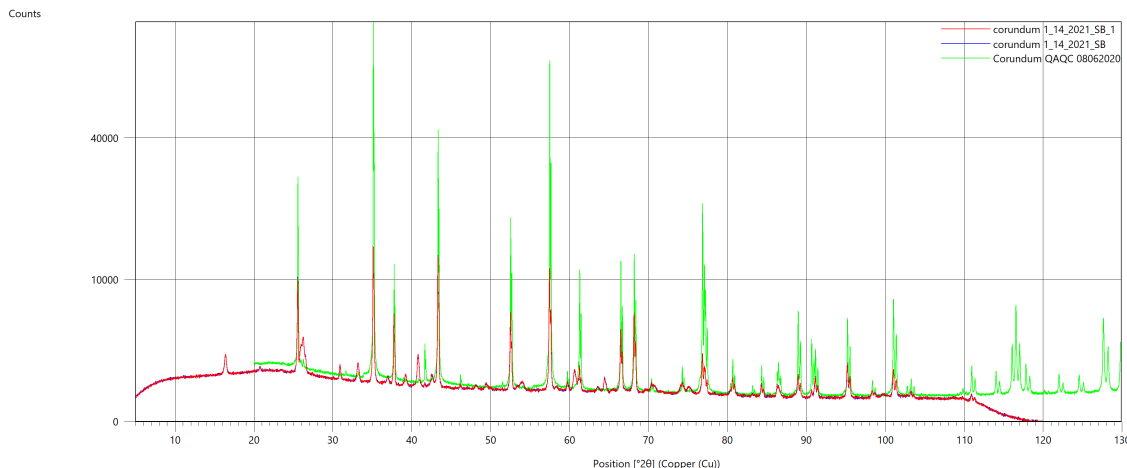


Figure 33. Corundum standard scans on the Aeris. Note the superimposition of the peaks.

Run the reference disk (Silicon standard disc) at the **beginning of every expedition**. This tracks intensity diminishes as the X-ray tube gets older. Run it using the measurement program Aeris provided. It is found in C:\PANalytical\XRDMP Creator\PANalytical predefined creator program\Aeris\Minerals Cu. Select the sequence **Silicon (Cu Tube).xrdmp**. You will need to unlock the cover (see **Changing Hardware** above) and insert the Ni filter if not already in.

Maintenance

1. Use a handheld vacuum to **clean any dust inside or outside the instrument**. The bearings that spin the sample on the sample stage gets dirty from sample dust. Clean them with isopropyl alcohol and a kimwipe (*Figure 34*). If you notice residue on the rims of the sample holders it means they need to be cleaned.
2. Check the **cooling DI fluid level weekly**. You can view and access the reservoir by opening the left rear panel (*Figure 35*). If the level is approaching the minimum limit then you must add more deionized water from the Barnstead dispenser in the chemistry lab.
 - Use a 6mm allen key to remove both the vent plug and fill plug (*Figure 35*) .
 - Use the gooseneck refilling bottle to slowly add DI water into the fill plug up to the high mark (*Figure 35*). Do not overfill. Aeris does not need a lot of water.
 - Replace the fill plug and vent plug. Replace the side panel.
 - Aeris needs water added in the middle and end of each expedition when being used regularly.

Clean and vacuum the dust filters at least once per expedition (*Figure 36*). If they need replacing we have spares in the black PANalytical XRD Service tool kit. If you use the spares make sure sure to order another set. In general keep the instrument clean and free of dust.

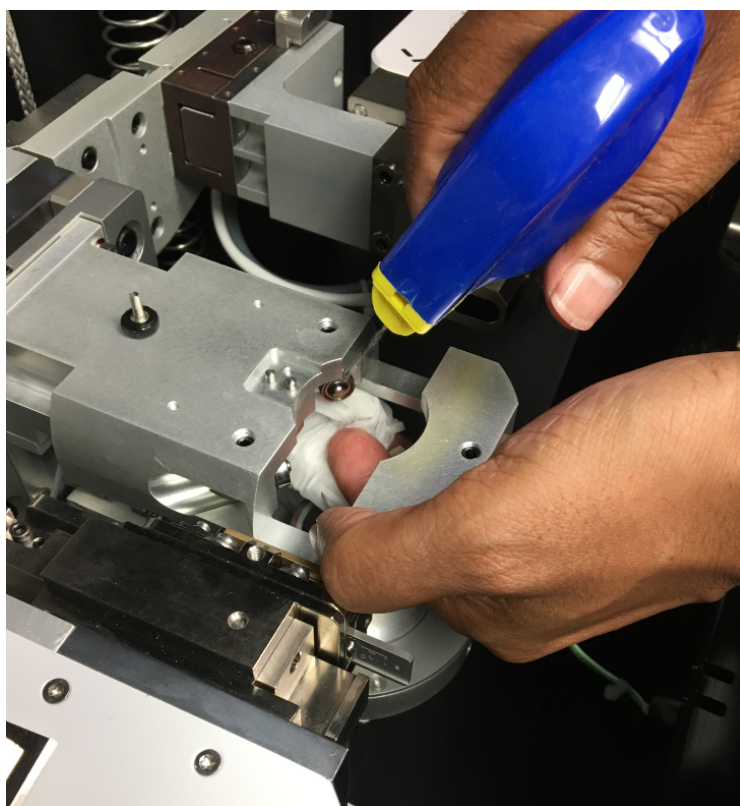


Figure 34. Cleaning the sample holder.




Figure 35. Cooling fluid reservoir. A. Vent plug, B. Fill plug, C. Coolant reservoir.



Figure 36. Cleaning Filters.

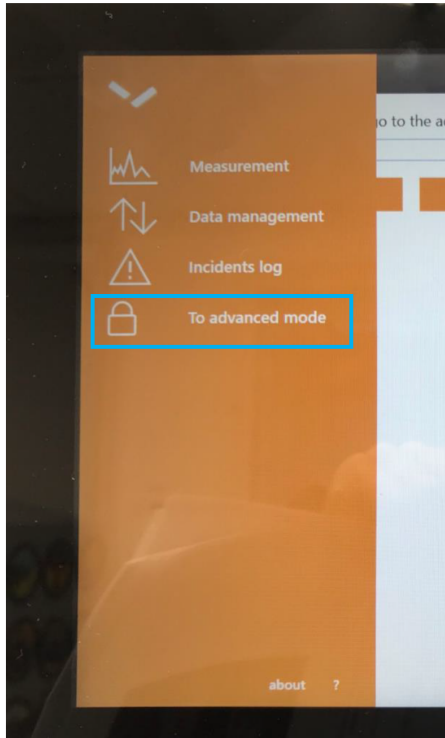
Advanced Mode

The Aeris XRD can be run with two different modes: Standard Mode and Advanced Mode. It is easy to switch between modes on the Aeris

touchscreen in the MENU  (Figure 37). Go to the Advanced Mode. The Advanced Mode is protected by a password. Password is: **panalytical**

In **Manual Control** the user can unlock the cover if it is necessary to change any hardware or to inspect a problem and manually unload a sample that got stuck or lost (see *Figure 15* in Changing Hardware).

Standard Mode menu



Advanced Mode menu

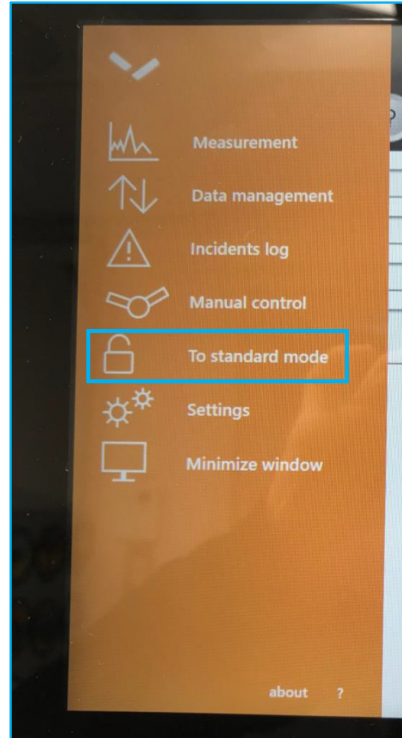


Figure 37. AERIS modes. Standard Mode (left) has less options than Advanced Mode (right). Note the lock (closed/open) for access.

In Advanced Mode the user can import and delete program files and delete old data that have already transferred to the PC. Select **Minimize Window** to gain access to the instrument computer (Figure 38). Click the Windows icon, and under Panalytical IMS select IMS Client. **IMS Client:** Click on Scenarios, Run. This is where the user can backup the system files, troubleshoot network connection, and collect logs if needed to send to the vendor for servicing. Some of the options are not available to us because you need a service dongle that PANalytical does not give out to their customers. If further access is needed, the instrument must be given internet access by the MCS so that service techs can remote in to the instrument.

Backup the instrument system files and copy to a USB at least once per expedition.

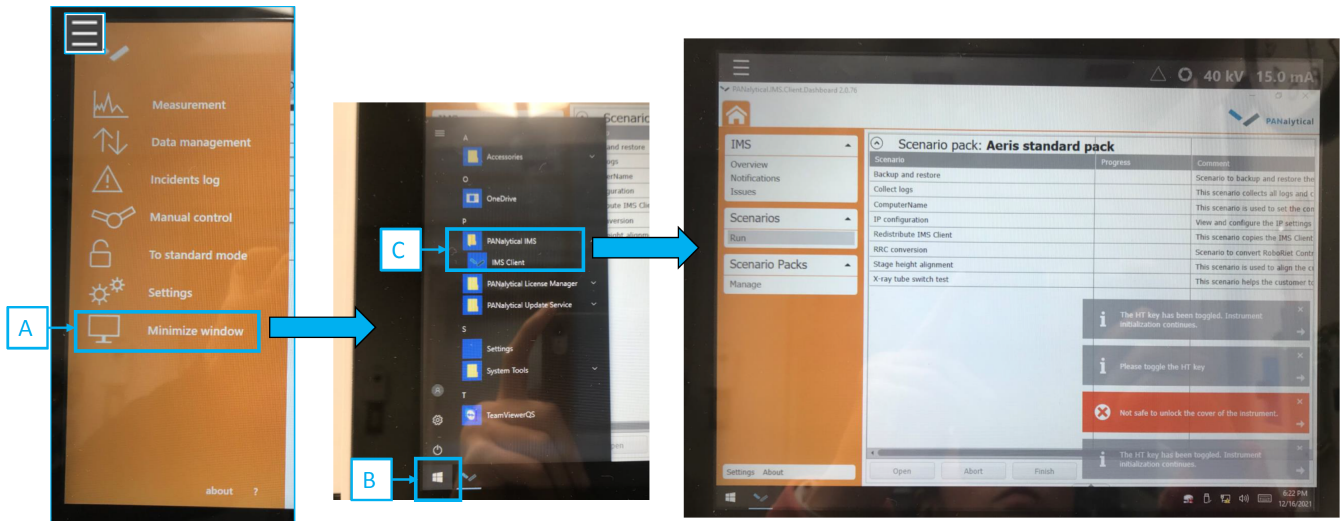


Figure 38. Accessing the scenario window on AERIS XRD for system files backup and logs to send to vendor if necessary.

Troubleshooting

Unfortunately, most troubleshooting options are only available to [Panalytical service techs](#). They can access the instrument remotely to fix or diagnose issues. Ask an MCS to give the instrument internet access to make this possible.

Error codes: Some error codes can be cleared by turning off and back on the front computer. Press the power button on the side of the screen, do not turn the key switch (see *Figure 2*). Wait a minute and press the power button to turn back on. If turning off and back on does not clear a goniometer motion control error, open the lid of the instrument and push on both the source and detector to move them. They are on cables and can be pushed and pulled by hand. Then turning off and back on the unit should clear the error.

Keyboard and mouse are wireless and run on batteries. If not working check the batteries and that the Bluetooth dongle is still in the Aeris, on the right hand side next to the network connections.

Files not exporting Error:

- Check the wires to make sure they are secure.
- The data will not export to the computer if the network is down. See the MCS if there is a network issue.
- After the instrument is turned off and back on, the file transfer does not work and needs to be reconnected.
 - To be able to transfer files you will need to go to "Advanced Mode"
 - Once in "Advanced Mode" go to file, menu, and minimize screen
 - In the bottom right hand corner you will see a red "x". Click on this icon and it will open up the file. You will see a file called "XRD Data"
 - Double click on the "XRD Data" file, it will open and reconnect itself.
 - The files will be able to be transferred again.
 - If the issue persists see the MCS.