

Scintag X-ray Diffractometer USER GUIDE – Revised

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home page \diamond www.geology.wisc.edu/~xray/xrd.html
sign up \diamond www.microscopy.wisc.edu/cgi-bin/calendar/geologyxray/calendar.cgi

Powder Diffraction Charges:
\$10/hour of tube time
\$40/hour of technician assisted time

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Follow these operating instructions in the order given. Make sure you understand the safety issues for this equipment (see Appendix I). If you hear a “clicking” sound from the machine and messages on the power supply console blink on/off, lower the kV by 5 (40 to 35), and contact John or Liz. If anything else unusual happens, contact John or Liz, and wait (if you definitely smell smoke and John or Liz is not immediately available, then turn off the power – either press the red “mushroom” power button, or flip the wall power switch off -- down).

1. **Detector LN.** Check the red LED on the front of the detector; if illuminated, you are out of luck...the detector is warm and will need to be refilled and then there will be a 30-45 minute wait before you can proceed. Liz and John are responsible for keeping the LN filled during normal operating periods.

The detector is a Germanium (Ge) solid state detector. The detector should be at or near 90° to be filled; move it to that position with software controls (5e below) if necessary, though we have programmed DataScan to automatically return to that position after the end of each run.

In the event that you must fill the LN dewar, see Appendix VII.

2. **Power status check.** Check the power supply for the X-ray tube: normal operating procedure is to keep some power ON to the tube 24/7, unless the machine is to be unused for an extended length of time (i.e. >5 days). Therefore, you should see the ‘X-ray On’ lit red and the ‘Intlk Closed’ indicator lit (this refers to the cooling water interlock being closed because water is flowing). If these are NOT the conditions, then go to Appendix II (“Cold Start up”).
3. **Warm up.** You want to go from the standby settings of 10 kV and 2 mA, to 40 kV and 35 mA, gradually, over a minute or so: 1) turn HV to 15 kV and current to 4 mA, then wait a few seconds; 2) then 20 kV and 6 mA, wait; 3) then 25 kV and 8 mA, wait; 4) then 30 kV and 10 mA, wait; 5) 35 kV and 12 mA, wait; 6) 40 kV and 14 mA, wait; and 7) now turn up current to 35 mA
4. **Start MDI DataScan Acquisition Software on the Dell computer.** (First check that it is not already running, just minimized on the bottom menu bar.) There is an icon on the desktop. Once you have the DataScan window open, click the Controller Menu tab at the top right. Do NOT change any values here, except for the ones indicated next.
5. **Goniometer Verification:** Verify that the actual angle positions on the instrument agree with the software:
 - a. Go to the instrument and read off the exact values of the inner sample holder ring (=theta), and the outer detector ring (=2-theta). Be sure to look at the fine-tuning verniers on the front of the goniometer, to be sure they are sitting at zero ($\pm .01$).



- b. Also check the chi and the phi (rotation around 2 different axes), they should be at zero (0).
- c. Go back to the Controller Dialog Box. Verify that the 'Current Axis Angles' of the 4 axes are the same as the angle position readings on the hardware. Normally, 2-Theta will be at 90.0 and Theta at 45.0. So if this is what you found on the instrument, there should be no adjustment necessary. Look down near the bottom for 'Current CHI Axis Angle' and 'Current PHI Axis Angle', which should both be 0.0

If the actual values do not match software, but are WHOLE numbers (vernier knobs=00), then:
Match Goniometer reading on software to actual value

1. Determine the actual angle position
2. Type in the actual value you found in the relevant box

If actual value is not on a whole number (vernier knob/s not 00), then do the following:

1. Determine the actual angle position (say the ring is between 12 and 13 and the vernier is at 50 for one axis.
 2. Type in **last** whole number to where the axis really is (e.g. 12)
 3. Repeat for other axis if necessary
 4. Click 'Calibrate Angle' and let the machine do its thing
 5. Determine the actual angle positions again, both the coarse (ring) and fine (vernier), and if they are the same as the software says, you are done. If now the wrong value but a whole value (zero on vernier), just type in the actual number in the box in the Dialog window.
- d. Before closing this window, it is a good idea to verify once that communication is working and the alignment remains correct. Now, in the **Drive to** window, type in a value a short distance away from the 2-Theta value shown, e.g. if it is at 90, type in 80 and hit the return key. You should hear and see movement of both Theta (to 40) and 2-Theta (to 80). If that is correct, everything is good. Now click 'Close Dialog'.

6. Load your sample. Sample preparation and sample placement in the holder are two very important things, to get the best results. There are several issues that need to be considered. You want to get maximum X-rays diffracted from your sample, and you want your peaks to be located accurately so that they can be easily indexed.

- a. Slide: you want to use a slide that's has one dimension 29-30 mm (no more than 30 mm wide, so you don't have to cock the slide at an angle, where only 2 are being held; you want to be sure the sample is exactly in the correct plane. See Appendix IV for suggestions.
- b. What form of mount? Smear (dried slurry on slide) or double sticky tape? Clays and fine powders are very amenable to slurries and this provides a robust thickness; some powders however, upon drying and being placed in the vertical position in the machine, fall off the slide (I do not know which will; best to try a smear mount first, as it is superior to the tape mount). Such material must be attached to double sticky tape on a slide. (Make sure you use plain sticky tape, not fancy new 'removable' tape that has a coating with distinct peaks.)
- c. Sample position on slide: while there is only a small area on the slide that "sees" the X-rays, I have found that it is optimal to cover the slide completely from top to bottom with material (though not necessary to cover completely left to right). Why? To achieve maximum accuracy, as there is only one correct sample plane for which the calibration is strictly correct. Therefore, whatever the thickness of the sample, it is held **back** to the proper plane by the top and bottom restraining metal pieces. (See Appendix V)
- d. Sample particle size and specimen thickness: you want a **minimal thickness** because the machine has been calibrated with a standard whose flat surface is exactly in the plane of the glass slide (it was a sample of powdered quartz, pressed into a recess that was then made parallel with the holder surface). This means that if you have (as an absurd example) a 1 mm thickness above the slide, your peaks will be displaced from their correct 2d spacings, creating major interpretation problems for you. The manuals say you want your particles to be very fine, less than 10 microns in diameter. Certainly you want to be no more than 100 microns in size (sieved below 170 mesh). If you are using a coarse particle size (say 200 micron), you will have to understand the accuracy will be reduced.
- e. If you have only a limited amount of material, you will need to think through what you are doing. If you have very fine powder and can make a nice thin smear only in the middle of the slide, you can maybe get away with Rather make an oval or rectangle (extending from the TOP to the BOTTOM across the slide, oriented as it will be in the sample holder) and then position it exactly in line with the black lines on the machine sample holder (See Appendix IV)

7. **Slits.** Check slit sizes on the tube and on the detector. Change sizes to your preference. The normal slits typically used for phase identification are (left to right):

Detector – **0.5** – **1** – Sample – **3** – **2** – Tube

And for particle size determinations:

Detector – **0.3** – **0.5** – Sample – **2** – **1** – Tube

8. **Close the door** to the Scintag. Grasp the handle in the middle, not the right end (that causes twisting of the top hinge).

9. **Set Scan Parameters** in DataScan software.

- a. The second menu button at the top left should say “Routine Qualitative Scans”. Click Scan menu button just to left of it.
- b. The Routine Qualitative Scans dialog box opens. The very first thing you need to do is to set the default file writing to **your own folder**, not the last user’s. Click the Files -> C:\DOCUME~1\GEOUSER\MDIDAT~1\DATA\<thelastuserdir> and select your own folder.
- c. If this is your first experience here with DataScan, you need to create your own folder. Make sure you are in the correct DATA folder and then type in the Subdirectory to Create – probably use your last name and first initial/all if necessary, or something distinctive. Close the window.
- d. You should now see your folder name *.MDI in the top files box.
- e. **SETUP:** You set up your scan parameters. If you will be doing the same thing over and over, you will only need to set up **ONE** time. Click Setup and go to Scan Settings for Routine Scans dialog box. You will be seeing the last user’s settings.
 - i. Verify: Select Axes to Scan = Coupled, and Scan Mode (bottom) must be Step Scan.
 - ii. Settings name box at top, will have the last user’s setup file and default number. Erase all and enter the name of your scan parameters (I recommend: something with your name and/or material). The software will automatically add the number.
 - iii. Normally you will just be entering 4 numbers, in the R01 boxes. A) Starting angle, B) Ending angle, C) Step size (degrees), and D) Dwell time (seconds). Generally you have to experiment to get what works best for you, but some possible values might be step scan size of no more than .01, maybe .005; time of .5 second per channel. Save and go up a level and click on the clock to see how long this will take; if too long or too short, iterate.
 - iv. This box also provides the option of setting up scans where you run at different rates over different (continuous or discontinuous) ranges of angles.
 - v. When you are down here, and made sure you have your settings named, click the Save Setting button, and then the Close button. You return to the Routine Qual Scans dialog box.
- f. Enter a file name in the File ID box, 8 character max. If you want to use the automatic incrementing, you could start as say Fe-ox-00, and click the Auto box, and then it will automatically give new numbers for each subsequent sample. This is obviously cryptic, and you also must enter Scan ID which can be the same thing or a more descriptive phrase. Finally, you can add more details in the comment box, which will be displayed on the scan output.
- g. You should not change any other settings in the box. Assuming the sample is loaded, the goniometer positions verified, the tube tuned up to operating conditions, and the door closed, you are ready to go. Click the Start Scan button on the bottom right.
- h. The scan will be automatically displayed in front of you. It will run until it reaches the end point, and then insert the shutter (click), and then return to the 90-45 resting position. The data is automatically saved.
- i. If you decide to modify the settings, go in to your own setting and modify the one you created. Do not create a new setting file unless absolutely necessary (but don’t forget to save your modifications).

later

10. **Power Down the Generator** when you are finished for the day

- a. Dial current down to 2 mA – slowly
- b. Dial down to 10 kV – slowly
- c. **DO NOT** turn off the Power unless you have been explicitly told to do so by John or Liz. We leave it on to reduce stress on the tube.
- d.

11. **Data processing and analysis** are done with the Jade software on the adjacent See the Jade write-up.

Appendix I: Basic Safety in XRD Lab

X-rays. The X-rays we are concerned with are primarily the tube Cu K and L characteristic lines plus continuum, but also secondary fluorescence from the sample and holder. The highest energy X-ray possible under normal operation is from the 40 keV continuum (Bremsstrahlung) (but there are very few of these 40 keV X-rays produced), and the highest characteristic X-ray is the Cu K α with 8.0 keV energy (there are a lot of these) as well as Cu L α with 0.93 keV energy (also a lot produced, but being weaker many are absorbed by the Be window leaving the tube).

The potential health effects are mainly damage to cells and tissue, i.e., burns, mutations, which under extreme conditions could lead to radiation sickness, cataracts and cancer. The greatest danger is to rapidly growing cells, meaning that pregnant women working in locations of potential X-ray exposure need to pay particular attention to safety precautions.

For the XRD lab, X-rays can only affect users if the shutter between X-ray tube and sample is open AND the metal and leaded glass door is open, or one of the other 5 walls of the enclosure is removed or breached. There are several fail-safe interlocks built in to the equipment: if the HV power bulb is burned out, the HV will not turn on. If the shutter Warning bulb is burned out, the shutter will not open. If the door is open, the shutter cannot be opened. (It is possible to bypass these, particularly the last one, but this is only done by experienced technical personnel and should never be done with users in the room.)

We are licensed by the State of Wisconsin to operate this equipment, and have periodic (yearly) inspections by the University Safety Department, to verify that there is no radiation escaping from the Scintag cabinet during operation. The Safety Department will issue radiation monitoring badges that users can request if so inclined. The Safety Dept also holds frequent 3 hour classes (weekly) on radiation safety at Union South that you are encouraged to attend. They have an excellent 378 page manual "Radiation Safety for Radiation Workers", a copy of which is available in the XRD lab, and you can also get your own personal copy by contacting Arne Jansen, ajansen@fpm.wisc.edu

High Voltage. There are potentially dangerous high voltages present in some of the electronics. Users should never reach inside any of the electronics. In the unlikely case where you were to encounter someone who appeared to be accidental in contact with a high voltage component, first quickly try to alert someone else in the vicinity to the hazardous situation, and then switch off the main breaker on the wall to the right of the machine.

Liquid Nitrogen. If you must, handle LN with care, as you can get burned (frost bite) if any significant quantity remains in contact with your skin (trapped in clothing, gloves, shoes) for more than a couple of seconds. Normally this is the only danger if you have only a dewar in a moderately large room. In an enclosed volume (overturned dewar in a van; frozen open valve on large tank in small room) death by asphyxiation (nitrogen displaces the air) is possible.

Appendix II: Cold Start Up

1. Press orange 'Control Power-On' button. 'Intlk Open' signal will illuminate. This means there is no cooling water flowing into the x-ray tube.
2. Press and hold green 'X-Ray Off' button to see the preselected current and voltage values are. They should be at 10 kV and 2 mA. Release button.
3. Press red 'X-ray On' button and hold until the 'Intlk Open' signal switches to 'Intlk Closed'. Water is now flowing
4. Wait 2 minutes then proceed with the warm up instructions in section 3 above.

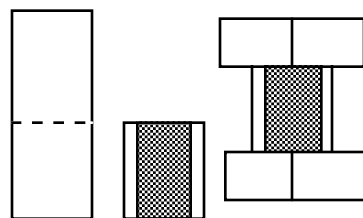
Appendix III: Troubleshooting common problems

1. You decide to abort a scan before completion. The software will realize that an abnormal condition exists, and will ask you to verify the current axes positions — be patient, sometimes it takes a minute or two to get to that step. Then check to see if theta and 2-theta are where it says they are, and if not, key in the values where they are.
2. For some reason (hopefully not operator incompetence), you drive the sample or detector axes past where it should go, and it protects itself by "hitting a limit" switch which freezes operation. To protect itself, the machine goes into a cocoon. You need to power off the axes motors...easier said than done. First try this: in the Controller Window (Scintag PAD-V Dialog), under Goniometer Power, click Power Dn if you can (probably can't). Otherwise you need to totally shut down power to the 4 microprocessor boards, pushing the big red square button over on the lower left center of the machine. The goal of all this is to turn off the motors, so you can MANUALLY back off the offender from the limit it is hitting. It should be very obvious which one it is. You turn the appropriate vernier knob in the front (theta is sample, inner ring; 2 theta is detector, outer ring) in the PROPER direction (do not turn if you feel resistance, reverse direction) until you are several degrees away from limit switch. Now you should turn the vernier to an exact value (00), and you

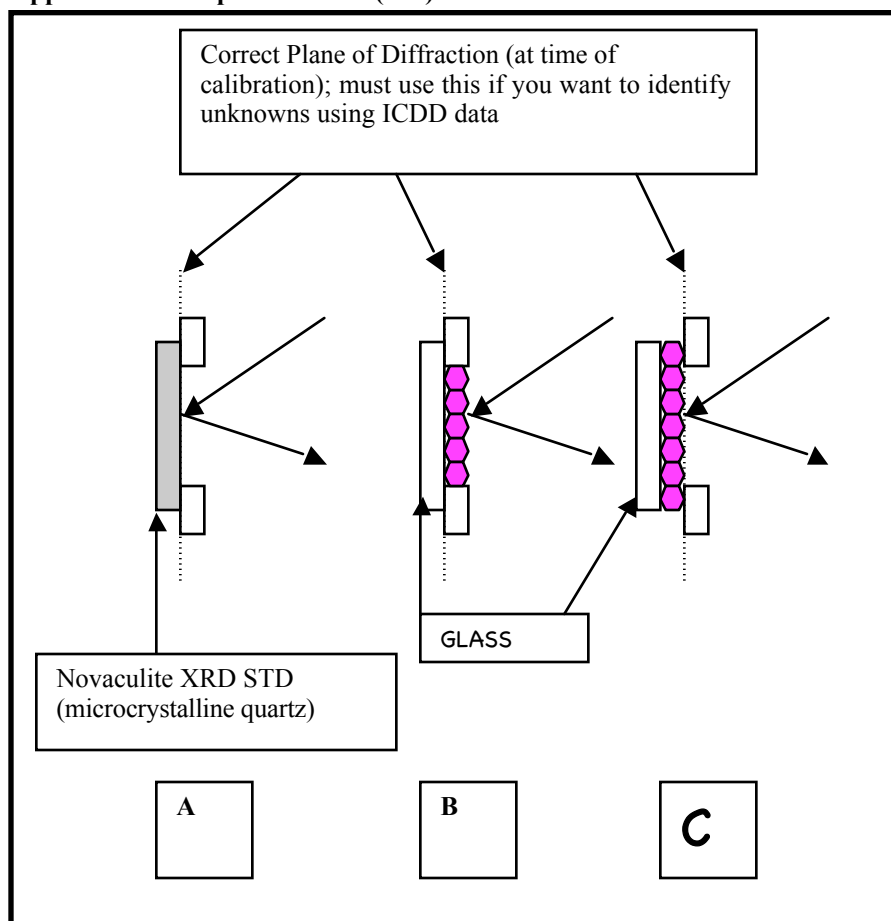
might as well turn the other vernier also to an exact value. You then to turn the power on manually (square red button) or click the “Power Up” in the Controller window. You’ll have to verify the positions.

Appendix IV: Preparing Glass slides for optimal usage with Scintag PAD V

With a glass cutter (simple old fashioned kind or dremel tool with carbide tip), scribe a line (with a ruler) 30 mm from one end. Then with slide held inside a paper towel, bend away from the line to break the glass (over a trash can). Now you have a good substrate for your sample; place your material in a line that covers all but the outer 3 mm on each side of the glass slide. Then you position it in the machine sample holder so that the material is aligned with the vertical lines above and below it (see right).



Appendix V: Sample HEIGHT (size) can make a BIG difference



(These are side views of the above plan views)
Fig A shows the position of the quartz standard plate at time of calibration. Note the vertical dotted line that is coplanar with the BACK of the 2 pieces of metal that top reference the sample. ONLY samples whose top surface lie in this plane will yield accurate (true) angles.

Fig B shows a coarse specimen mounted on a glass slide, with the edges of the glass slide pressed tightly to the sample holder. The material sits significantly above the correct plane of diffraction, and therefore data gathered here will NOT be accurate, and determination of unknowns will be extremely difficult.

Fig C shows the correct mounting of the same sample. Here the specimen covers the whole glass slide from top to bottom, so the sample material itself presses against (top references) the holder.

Appendix VI: Computer administrivia

1. We are using two applications from MDI, DataScan (v 3.2) and Jade (v. 6). Both have icons on the desktop.
 2. DataScan operates with a “everyone sees everyone else’s” settings paradigm, whereas Jade allows each user to create their own Jade working environment, which makes life much simpler, as you don’t have to worry about settings the last user has set, or some screwy set of phases they have defined as the default search path.
 3. The user files are to be located only in the following folder/tree structure: C:\Documents and Settings \ Geouser \ MDI DataScan-Jade6 \ Data \ <your folder>
 4. Please exit the programs when you finish for the day. Please do not clutter the desktop with your own stuff (e.g. pdf articles, etc). DO NOT download software onto this computer.
- This computer runs under Win XP. If you somehow end up with a frozen screen, DO NOT turn off power on the computer: simultaneously hold down the Alt + shift + Delete keys, and click on Task Manager, then Kill the last /

offending program, and you should have control back. In general, never turn off power to a computer without going thru the normal shutdown software operations -- left click on the far left bottom corner of screen (menu bar) "Start" and then click the Shut Down option at the bottom. Choose the Restart option if desired.

5. Upon a start up, you will need to login in. We have defined the xrd user environment as user = geouser and the pwd = geology.

Appendix VII: LN

Do not fill the LN unless you have been given explicit OK (or John or Liz are not around to ask, and you must run XRD). Carefully fill the XRD dewar with Liquid Nitrogen (LN). You must then wait until the LED goes out, then wait 30 minutes more (and the first part of the scan may be noisy). If LED is not illuminated, good. Check the LN log sheet, and if it has not been filled yet that day, fill it carefully (use funnel and green spacer underneath), and then record that on the LN log -- if you did not completely fill it, e.g. not enough LN available—make a note below the date saying "not topped off". (If you are running a long day, say 9 AM-9PM, also please fill it at the end of the day if possible.)

⋄ Note: it is very important that you do not write "filled" unless there was an excess of LN put in the XRD dewar and you saw that excess roll over the top and spill out. If the portable dewar is full, and it is poured into the XRD dewar but no excess comes out, the XRD dewar is NOT full.

Appendix VIII: Resources for understanding principles of good XRD practice.

There are 3 excellent resources to provide you with the background knowledge to achieve whatever you are trying to do with XRD.

The first is an online course developed by Jim Connolly of Univ. New Mexico (url and link to it are on the Lab's web page). A hard copy is in a binder in the lab, and there is a link on the lab web page if you want to download the pdf or doc files.

Two other books also present in the lab (also in the Geology Library) are **Modern Powder Diffraction** by Bish and Post (1989), Vol 20 of the Mineralogical Society of America's *Reviews in Mineralogy* (369 pp) (available at modest cost from MSA, with a 20% discount to members), and **X-ray Diffraction and Identification and Analysis of Clay Minerals (2nd Edition)** by Moore and Reynolds (1997), Oxford University Press.