



# Lab Manual

[Marvell Nanolab](#)[Member login](#)[Lab Manual Index](#)[Mercury Web](#)[Berkeley Microlab](#)

## **Siemens D5000 X-Ray Diffractometer**

(xdif)

### **1.0 Title**

Siemens D5000 X-Ray Diffractometer

### **2.0 Purpose**

An x-ray diffractometer illuminates a sample of material with x-rays of known wavelength, moving the sample and detector in order to measure the intensity of the diffracted radiation as a function of beam and sample orientation.

From the resulting intensity versus angle plot much can be inferred about the structure of the material.

The measurement is generally not straightforward, and the inference required to make useful observations on the sample is quite subtle.

All the diffractometer can tell us is the spacing between atomic layers: For example if the diffracted beam is reflected directly back into the x-ray source (obviously not an experimentally-convenient situation!) we know there are crystal planes exactly two times 1.5418 Angstroms apart, or possibly, some integer multiple of that spacing.

When combined with enough other information, in particular a knowledge of crystal symmetries, remarkably complete understanding of the sample can be achieved. Indeed, in a sufficiently ordered sample (i.e., a good single crystal) molecular structures can be worked out

Conversely, if the structure is known a measurement of the structure's dis-order becomes possible. This has important applications in process control, since epitaxial growth of well known materials is crucial to microelectronic fabrication and x-ray diffraction provides a relatively precise measurement of crystal growth quality.

The D5000 measures atomic spacings in crystals using diffraction of approximately monochromatic x-radiation. It can be used to characterize solid samples ranging in size from about 1 millimeter square up to intact four-inch wafers. Six-inch samples may be measured in this tool if allowing holders to reach maximum extension. It also requires extra attention to secure six-inch samples. The radiation used is copper k-alpha with a wavelength of 1.5418 Angstroms. The instrument has two fundamentally different modes of operation.

#### **2.1 High Resolution Mode**

It works only on samples which are efficient diffractors (usually single crystals) and have relatively simple internal structure, for example repeating layers of alternating composition (common in optoelectronics). It provides detailed information about crystalline quality, composition and layer structure.

High throughput mode will work on essentially any sample; in the extreme it will work on crystalline powders with no grain-to-grain orientation at all. The angle of the diffracted beamlets provides information on the atomic spacings within a crystal, which is a guide to composition.

The software allows any measurement in either mode, but the physical significance of the measurements depends drastically on which mode is used.

The high-resolution mode is the easiest to understand: X-rays from the tube pass through a Bartels four-bounce monochromator. All the off-axis and off-energy (K-beta) photons are filtered out, and the resulting beam encounters the sample. The monochromator has a beam FWHM of 0.0033°; otherwise the beam spread is about 0.2°. Diffracted signal intensity is dominated by the

sample-beam orientation; the direction of the diffracted beam, while measurable, is of limited significance: It is always twice the sample angle and much harder to measure.

Because of the very high losses in the monochromator the sample must be an efficient diffractor to have a usable signal. Typically the sample will be a high-quality single crystal or a well-oriented epitaxial film. In that situation **rocking** the sample under the beam reveals a measure of crystalline quality; better crystals have more sharply-peaked angular intensity dependence. Furthermore, **layer cake** structures can be analyzed for thickness and composition.

To get any signal at all one must have the sample oriented correctly under the beam, and then place the detector in a spot where it can catch the diffracted radiation. Since the relevant orientations are to the sample's crystal lattice, not its surface (though hopefully the two are at least somewhat related) finding peaks can be very much a trial by error in which conviction is assured 8-)

## 2.2 High Throughput Mode

Now consider the problem of a poorly-oriented film; in the extreme it could be a pile of crystalline crumbs. This changes things drastically: The crumbs are apt to be randomly-oriented, so moving the pile under the beam will have no effect: For every crumb that rotates to the **wrong** orientation another will rotate to the **right** one. There will be much absorption, so we need all the intensity we can get. Intensity of the diffracted radiation becomes totally independent of sample-to-beam orientation.

However, diffraction still occurs and still honors Bragg's law: Any photons that strike a crumb with the **right** orientation will be diffracted and can be counted. In this case we have no choice but to use detector position, sample position and x-ray tube position to let us work out the geometry.

In this situation sample angle becomes meaningless and all information is contained in the detector angle. The resolution now does depend on source sizes, illuminated area and slit sizes.

In contrast to the high resolution mode, the relevant measurement is now the detector angle. Diffraction efficiency is necessarily low, since most of the grains in the sample are oriented wrong and angular measurements are less important, so no monochromator is used. However, there is enough k-beta radiation to make confusing double peaks, so typically a nickel filter is used to improve the spectral purity (but not the angular quality) of the x-ray beam. Slits are selected carefully to control noise and find a good compromise between resolution and signal intensity.

## 3.0 Scope

This manual covers elementary physical operation of the instrument and radiation safety compliance requirements. Data interpretation is beyond the scope of this manual.

## 4.0 Applicable Documents

### [Revision History](#)

- 4.1 Virtually any textbook on x-ray diffraction. Also, there is a wealth of resources on the Web: <http://dept.physics.upenn.edu/~heiney/talks/hires/hires.html>, for example.
- 4.2 Labmembers are invited to peruse the manufacturer's manuals, located on the desk where the dosimetry rings are kept.

## 5.0 Definitions & Process Terminology

- 5.1 **RUA**: Radiation Usage Authorization. A permit issued by the University's Office of Environment, Health & Safety (EH&S) to let a radiation-producing machine be operated on campus. Both the machine and all operators must be explicitly identified.

- 5.2 **Monochrometer:** A device which selects x-rays of a specific energy, traveling along a specific axis. Ours is of a design by Bartels (J.W. Bartels, J. Vac. Sci. Technol. B 1(2), Apr-June 1983, pp. 338-345). Two channel cut germanium crystals are mounted as periscopes in opposition. The radiation diffracts from the first surface to the second, where it diffracts again with direction unchanged but position displaced by the projected width of the channel. The second crystal reverses the displacement, so the beam direction and position are identical to the original beam.
- The crystal lattice spacing in the monochrometer is effectively the **metric standard** to which the sample is compared.
- 5.3 **Goniometer:** The mechanism which supports the sample and detector, allowing precise movement. Goniometer has four different circle of rotation to adjust sample and detector positions.
- 5.4 **Circle:** Any of the separate mechanisms which rotate the sample or detector under the x-ray beam.
- 5.4.1 **Circle 1** rotates the sample about a vertical axis.
- 5.4.2 **Circle 2** turns the detector, also about a vertical axis, and only the vertical axis.
- 5.4.3 **Circle 3** tilts the sample about a horizontal axis and designated chi.
- 5.4.4 **Circle 4** rotates the sample about an axis normal to the sample surface through the center of the holder and called phi.
- Circles 1 and 2 are independent. Circle 3 is mounted on top of circle 1, and circle 4 is mounted on top of circle 3.
- 5.5 **Eucentric:** The point at which all four goniometer axes intersect in space. In the ideal case the sample surface coincides with the eucentric, as does the point illuminated by the x-ray source and the point observed by the detector.
- 5.6 **Counts:** A measure of signal intensity. For a given sample the apparent signal intensity will depend on the scanning parameters with small step sizes giving higher numbers.
- 5.7 **Reflection:** Sometimes used as a synonym for diffraction. There is a big difference, however: in optical reflection the direction of the reflected beam depends on reflector orientation. In diffraction the intensity of the diffracted beam depends on orientation but the direction is fixed in the coordinate system of the incident beam.
- 5.8 **Alignment:** The orientation of sample, beam and detector axes. Ideally all three coincide with the eucentric point.

## 6.0 **Safety & Regulatory Issues**

Radiation producing machines are regulated much more carefully than other common lab equipment. Lapses in procedure can result in major headaches, both for labmembers and staff. Please take care to acquaint yourself with interlocks, rings and logbooks.

### 6.1 **Interlocks**

In normal operation x-ray diffraction is utterly safe. The beam is interlocked to the door, it is impossible to expose oneself to the radiation without opening the door, hence no possibility of getting hurt.

However, x-ray diffractometers have a bad reputation for causing injuries.

The reason is simple. Operators defeat interlocks. After getting frustrated trying to obtain a signal from an ill-understood sample, the operator decides to do a **live adjustment** of sample position, convinced that's the only way to get results. The results tend to be unpretty.

The copper k-alpha radiation used in diffraction is strongly absorbed in most materials, including tissue. The resulting ionization destroys cells very thoroughly to a depth of about one millimeter. Reaction products diffuse somewhat deeper.

In fact, the beam is well confined, making it physically possible to work very close to the beam. Old style diffractometers are open, and people do use them safely. Still, from time to time folks manage to hurt themselves..

The one and only safety rule for xdif is do not defeat the interlocks.

Yes, it would be faster to adjust **hot**. If you know what you're doing, it does not take all that long anyway.

Apart from safety considerations, there are a few regulatory niceties to be observed.

The paper logbook on the desk is a legal requirement; without it the state regulators will shut us down. Always log your use accurately.

Please, do not operate xdif without a ring. If you need a measurement and can't find your ring, ask another labmember for help.

## 6.2 Obtaining a Dosimetric Ring

Please, do not operate xdif without a ring. If you need a measurement and can't find your ring, ask for help.

To obtain a ring, one must be on an **RUA** (Radiation Usage Authorization). It is issued by the Office of Environment, Health & Safety (EH&S), typically to your supervising professor or, more rarely, to a staff member.

Please, remember that there *must* be a valid (signed and current) RUA on file in the lab any time you use xdif. Having your ring is necessary but not sufficient.

## 6.3 Record Keeping Requirements for xdif Users

It is important to distinguish records kept by the accounting system from records required by state and federal government. The records generated by enabling equipment on wand have no legal standing and do not carry any weight in an incident investigation.

Always note the following information in the logbook kept on the operator's desk: Date, labmember login, RUA holder, sample, time, voltage/current and any comments.

In addition, a current copy of the RUA on which your name appears must be present in the holder on the side of the tool.

## 7.0 Statistical & Process Data

Before starting work it is essential to know the characteristic diffraction angles for your samples. They are available in a variety of places notably in PDF's (Powder Diffraction Files). There are digital PDFs for common materials available in room 124 HMMB. Some diffraction reference data can be found in standard handbooks like CRC, but the usual source is a proprietary database.

## 8.0 Available Processes

Our D5000 offers two fundamentally different modes of operation (see above). High-resolution mode is used to examine well-ordered single crystal films. High throughput mode is used to examine textured polycrystalline films. Non-textured or powdered samples can be examined also.

Diffraction measurements commonly take two forms, a **normal coupled scan** and a **rocking curve**. Normal coupled scans provide information about what materials are present and lattice type. Rocking curves allow estimates of crystal quality and in good crystals can also provide quantitative measurements of layer thickness. They can also be helpful in locating a known peak on a sample of uncertain orientation.

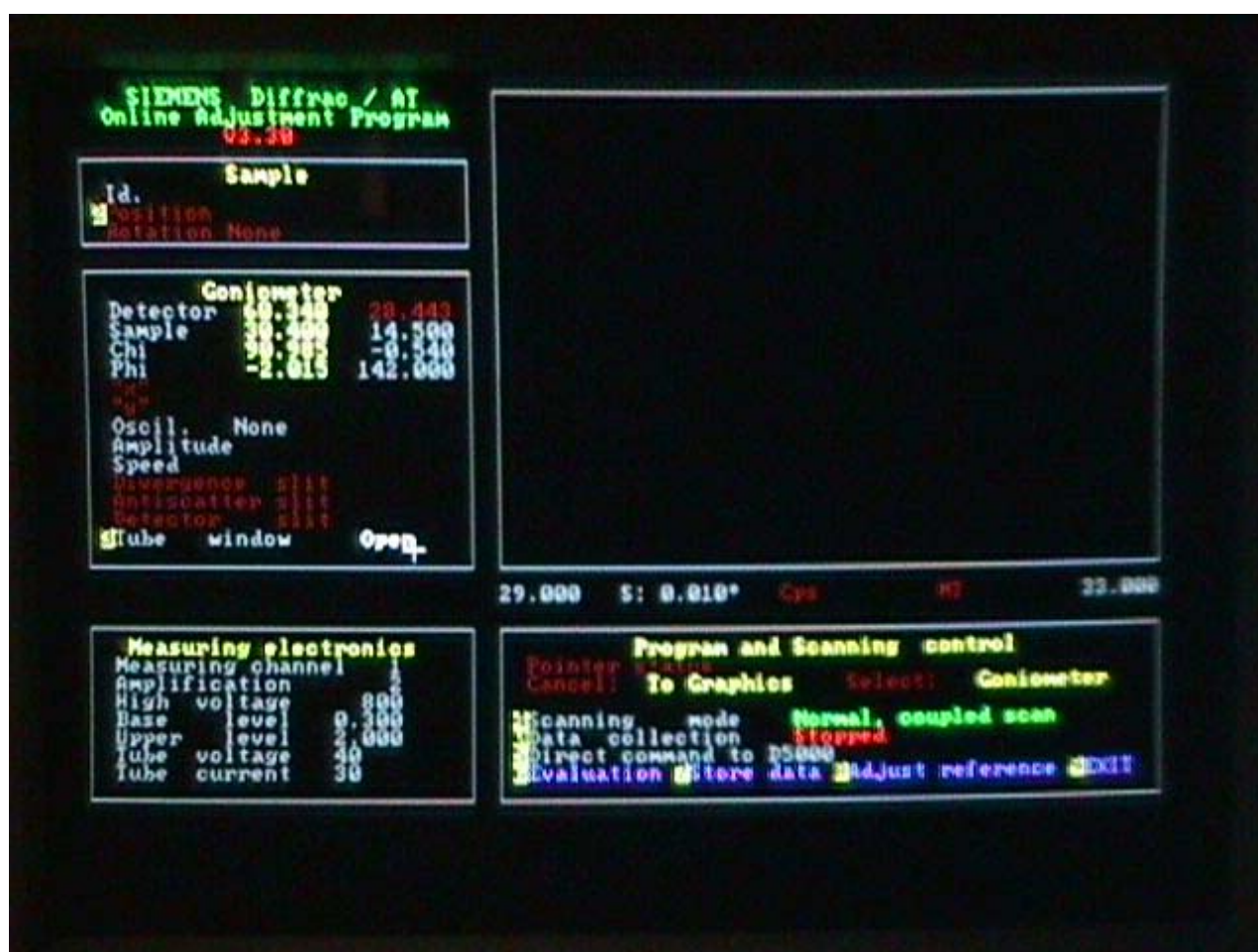
## 9.0 Operation

It is quite possible to make a life's work out of doing x-ray diffraction. This guide tries only to give the barest sketch, sufficient to get started.

### 9.1 Preliminary Steps

#### 9.1.1 Starting the Diffrac AT Software

- 9.1.1.1 Enable the system. Sign in onto the logbook: include the date, your login name, adviser, start time, end time and tube voltage/current.
- 9.1.1.2 Change directories so that you are in your home directory. The prompt should be: **C:\D5000\DATA\USERS\your\_name>**
- 9.1.1.3 Start up the Diffrac AT software by entering dmenu on the command line.
- 9.1.1.4 Select **Immediate Measurement** from the **Measurement & View** pull-up menu. The screen should look like this:



- 9.1.1.5 Initialize the goniometer by pushing F5, typing in and hitting return.

**Decide whether you need to install the collimator or install the monochromator.**

#### 9.1.2 Installing the Collimator

Occasionally, you will find the monochromator in place when you want to use the collimator. Please don't attempt this procedure without hands-on instruction; it is trickier and more frustrating than it looks.

- 9.1.2.1 First thing is remove the monochromator. Simply loosen the two (very tiny and hard-to-turn) shafts hidden under the large, prominent (and *not-to-be-disturbed* black knobs), lift the monochromator off its platform and put it gently down in the cabinet.
- 9.1.2.2 Next, locate the collimator on the platform and tighten the screws only enough to give slight friction, so you can still move the collimator by hand. Push it all the way to the back of the cabinet and rotate it counterclockwise viewed from above, far as it'll go
- 9.1.2.3 Then turn it clockwise about one degree. Don't tighten the screws yet.
- 9.1.2.4 Slip the phosphor screen onto the sample stage and adjust the stage so that z is 2 mm and the mark centers under the dial indicator ram.
- 9.1.2.5 Remove the indicator and begin a measurement; all you need to do is bring the stage into working position. Stop the measurement and look through the cabinet window at the mirror which gives a view of the bright green spot of fluorescence. It should be uniform and centered on the center mark. If not, close the shutter, open the cabinet and adjust the collimator.
- 9.1.2.6 Repeat until you have a well-centered spot, then snug the screws (two fingers on the driver are enough) and re-check. Now you're ready to measure.

### 9.1.3 Putting the Monochromator in Place

The monochromator is changed by labmembers, after instruction by staff or more experienced users. Please don't attempt a change without any guidance; there are physical nuances difficult to put on the printed page even though the job isn't all that difficult.

- 9.1.3.1 Begin by removing the collimator if installed; just unscrew the two bolts holding it down and lift it out of position. Store it somewhere inside the cabinet.
- 9.1.3.2 You will notice two small steel pins sticking up in the plate which supports the monochromator. They fit into cavities in the monochromator's baseplate where they are engaged by stops and setscrews to fix the location.
- 9.1.3.3 Pick up the monochromator by the handle,



and gently lower it onto the two dowel pins. It should settle down easily. Grasp the two very small and hard-to-touch shafts directly below the large (and not-to-be-disturbed) black plastic knobs, spinning them gently between your fingers, clockwise to tighten. The shafts, which drive setscrews, sometimes jam, wiggle them when they seem reluctant to turn. Tighten them only enough to prevent the monochromator from moving from side to side under the pressure of two fingers.

9.1.3.4 Make sure the slits and filter are out of the sample-to-detector path.



9.1.3.5 You're done.

## 9.2 Mounting the Sample in the Holder

There are two techniques for mounting samples in xdif: Centimeter-sized pieces are usually mounted on clay in a plastic sample holder available in 406 Cory Hall for a nominal charge. Whole wafers and large pieces of wafers are more conveniently held in a three-jawed clamp.

9.2.1 Clay mounting is the simplest: Just place a small ball of clay in the well of the sample holder, center your sample on top and press the sample into the clay using a flat, clean surface.



The goal is to make the top surface of your sample perfectly even with the top surface of the sample holder. Ideally the clay should be completely hidden by the sample. It pays to be fussy about mounting; tilt in the sample that's too small to see can easily shift peaks enough to make them *very* hard to find.



In general, try to avoid doing this:



Clay is after all crystalline and it will give rise to diffraction peaks which can obscure the data you're looking for.

Special thanks to Jeremy Schroeder for the excellent photographs!

The sample holder slides into the fixture normally installed on the goniometer and is supported by its *\_upper\_* surface under spring pressure. This way one has only to measure the z height of the stage; if the sample is correctly mounted in the holder it will come out at the right height automatically.

### 9.3 Loading the Mounted Sample in the Goniometer

- 9.3.1 Once your sample is properly mounted in the clay (flat and flush with the top surface of the holder) it is time to set up the goniometer.
- 9.3.2 Xdif should already be enabled on Wand and the **ON** display on the front panel illuminated. If not enable it now.
- 9.3.3 You must put on your dosimetric ring before opening the cabinet. Also check that the red warning light for the shutter is off, meaning the shutter is closed.



9.3.4 Next, take the dial indicator in your right hand by the mounting arm.



Use your left hand to slide the probe up gently, then transfer the indicator to your left hand, still holding the probe in the raised position. Slip the mounting pin into the socket at the top of the goniometer frame very gently, push it home and slip in the lock cam with your right hand, turning it firmly to lock the indicator in place.

- 9.3.5 Now, gently release the dial indicator probe to it touches the sample stage surface. The indicator should settle to a reading of 2 mm (4 revolutions of the dial from uncompressed state). If it is different, release the lock on the back side of the z slide and turn the lowermost thimble; when finished snug the lock again.
- 9.3.6 Next, lift the probe of the dial indicator with your right hand and use your left hand to slip the sample holder into the stage; just push down on the spring-loaded plate and guide the holder in. Keep the probe of the dial indicator raised and move the sample until centers under the indicator probe. There's no necessity to let the probe touch the sample, but you can if you want. Just be careful to avoid tilting the sample in the clay.
- 9.3.7 Once the sample is centered use your right hand to release the lock cam and take out the dial indicator with your left hand. Be very gentle, the alignment dowels jam easily. The cam can be left in place.
- 9.3.8 Hit the escape key to conclude the loading sequence and prepare to work.

#### 9.4 Locating an Initial Diffraction Peak

The methods vary. This is a simple one for planes parallel to the sample surface

The initial conditions will vary depending on the mode the instrument is in: High Resolution or High Throughput. This example is for High Resolution Mode using the single crystal silicon check sample which is kept in the desk drawer.



The monochromator will be installed between xray tube and sample stage. The slits and filter will be out of the detector mounting.

The xray tube normally operates at 40 kilovolts and 30 milliamperes. Do not increase these figures. The idling values are 20 kV and 5 mA, the actual value starts to ramp up only when a measurement is started. Occasionally, the working values are turned down and forgotten, so it pays to check.

There are a couple of points to remember about the control screen of the diffractometer. Parameters are changed by clicking on the value, for example **Normal, coupled scan** and either dragging the mouse or hitting the arrow keys. Numerical parameters are likewise selected by clicking, but can then be incremented by dragging or by typing in new values.

When a mode is changed, say from **normal coupled scan** to **rocking curve** the *previous parameters from the last time the mode was used* are recalled. That can be confusing if you *were not* the last person to use that mode.

The graph window plots the angle being scanned. In the case of a normal coupled scan the plot displays detector angle, *even though the sample angle is more significant in high resolution mode*. The sample always turns exactly half as much as the detector, but you have the opportunity to correct offsets by **unlocking** the sample via manual entry of an initial value, as we will see shortly.

Another common point of confusion is scanning step. The D5000 operates stepwise, moving the goniometer, accumulating detector counts and then moving again. It does not measure between steps, if the steps are big enough it is possible to completely miss a strong peak: For example: On a good silicon sample the peak width is less than .007 degree! If you scan at .1 degree per step you *may* never find the peak. On the other hand, if you insist on scanning at .001 degree progress will be excruciatingly slow. Typically 100 steps per scan is enough to start with.

In general try to avoid issuing direct rotation commands, as described in the Appendix. When using the **go** command it is very easy to make a mistake and cause a collision between goniometer parts. No harm results, but uncrashing the parts is often non-trivial.

- 9.4.1 Start by setting chi and phi to zero; click on the value, hit zero and then hit enter. Lock the sample to the detector by clicking on the sample angle, hit space to clear the value and hit enter. The value should become half the detector angle with an L next to it.
- 9.4.2 Open the tube window.
- 9.4.3 Perform a normal coupled scan about the expected peak position. This does two things, placing the sample and detector in approximately the correct position and giving you some chance of finding a peak immediately. Usually you will not see a peak, but simply getting the detector and sample into the correct ballpark is worth the time.
- 9.4.4 Try setting the detector to the expected angle and measure a rocking curve about half that value, with a span of say, plus minus five degrees. That will accommodate a large miscut or mounting error. Write down the sample angle giving the highest count rate.
- 9.4.5 Next, switch to a detector scan. Manually enter the sample angle you just measured and sweep the detector a few degrees either side of twice that value. Write down the detector angle giving the highest peak.
- 9.4.6 Once you have your peak, home in on it. Set the detector angle to what looks like the maximum intensity value and repeat a rocking curve. Iterate enough to be sure the process converges.
- 9.4.7 If at any time the peak **disappears** back up immediately to the previous measurement. It is very easy to make a key entry error, the software will recover all the previous values so you don't get lost. In this regard having old values recovered can be very handy.
- 9.4.8 Next, do a chi scan, usually plus/minus 3 degrees is enough, setting chi to the value giving highest signal.
- 9.4.9 Now, try a phi scan over 360 degrees. What happens will depend on the crystal plane you are looking at and how your sample is cut and mounted. It is enough to set phi to the angle giving maximum signal.

What you have done is measure your sample's orientation on the stage. The mounting error is the difference between the expected and observed sample angles: If the published angle is 60 degrees (conventionally detector angles are tabulated) the sample angle should be 30.

Generally it'll be something else, say 29 degrees. In this example the sample is tilted one degree (counterclockwise viewed from above) on the stage. A normal coupled scan will always mis-position the sample and detector, and no peak can be seen with such a large mounting error. We

have to start the scan with the sample axis tilted one degree clockwise, and then the detector will be in the right place when the sample angle is correct for diffraction.

Here's how:

- 9.4.10 Pick a starting angle for the detector.
- 9.4.11 Divide it by two, giving the starting sample angle.
- 9.4.12 Now subtract the error in sample angle.
- 9.4.13 In our example suppose we want to do a normal coupled scan from 55 to 65 degrees. The sample must rotate between 27.5 and 32.5 degrees.
- 9.4.14 The offset is (expected - actual), one degree in this case. We subtract the offset from the nominal starting angle and get 26.5 degrees.
- 9.4.15 Click on the sample angle and type that value into the initial sample angle box, eliminating the L next to the sample angle.

If you later decide to change the starting detector angle, you have to manually re-apply the correction. Many people forget this little detail, and the software will not remind you when you change the scan range in a normal coupled scan.

## 9.5 Saving Your Data

To do anything useful with your data you **must** save it first, even if all you want to do is make a quick hardcopy output. The process begins once you have a graph worth keeping.

- 9.5.1 Click on Evaluation, then File/Plot/system then Write Data File.
- 9.5.2 A box will pop up with the default file name
- 9.5.3 Left-click to pop up a second dialog box, use the arrow keys to position the cursor and edit the name to your liking.
- 9.5.4 Hit enter, select the action you want from the pop up menu.

## 9.6 Printing Out Your Data

The Diffrac AT software uses a two-stage printing scheme. Plot files are first **submitted**, then sent to the printer in a separate operation.

The system is capable of generating color plots. In some cases it is justified, as when overlaying several graphs on a single page, but color printing is extremely slow. For most purposes black and white printing is much more satisfactory.

- 9.6.1 Once you have data worth printing click on Evaluation.
- 9.6.2 A default file name is presented, you may edit it or click the mouse to accept. The evaluation window comes up. Data collection will continue in the background.
- 9.6.3 Click on Files/Plot/system, then on Submit Plot. An announcement box pops up giving you the name of the spooled plot file. You cannot edit it, note the name for future reference.
- 9.6.4 You may return to data collection or employ the other tools to quantify your data further.
- 9.6.5 When finished, click again on Files/Plot/system and then on Quit->DOS/Diffrac.
- 9.6.6 To actually plot the spooled files click on Exit, toggle to **Quit - to DOS/DIFFRAC** so that you're back at the welcome screen.
- 9.6.7 Click on Evaluation, then Plot Monitor. A list of plot files comes up. The arrow keys highlight options, an annunciation bar at the bottom of the screen lists choices. The space bar toggles among options.

- 9.6.8 Once you have selected which files to plot and how to plot them hit Enter. Four buttons appear, Review, Execute, create Batch and Quit. Highlight Execute and hit Enter.

There will ensue much disk activity, after a respectable delay (minutes in the case of color plots, the printer will disgorge your handiwork.

If you are truly unlucky, the plotting will fail; the most common cause is insufficient disk space. It takes about one megabyte of free space for each plot job, which of course nobody checks before they start. The only remedy is to clean house. Any .RAW file outside someone's home directory is fair game for disposal.

## 9.7 Obtaining Plain Text Versions of Your Data Files

The data taken by the D5000's control software is stored internally in a proprietary, binary format. To manipulate the data using anything other than Diffrac/AT it is necessary to convert it to an **interchange** format. Then it can be read into other analysis software. Very often spreadsheet programs are used to prepare graphics for use in publications.

The export format is called **Universal** and is essentially ASCII text. Header information contains plain-text information on machine parameters (tube voltage/current, starting angle, step size, number of steps, etc.) followed by a list of numbers representing count rates at each step.

To export data get into your home directory and

- 9.7.1 Type **xch** and hit **enter**.
- 9.7.2 Highlight **Export/Convert Diffrac / AT** file and hit **enter**. A list of filenames will pop up.
- 9.7.3 Use the arrow keys to select the file you want. Hit **enter**.
- 9.7.4 Highlight **convert to UXD format** and hit **enter**. You will be prompted for a file name.
- 9.7.5 Type the file name: It is conventional to use **.txt** as the extension.
- 9.7.6 The conversion will take place, you will be returned to the file selection page.
- 9.7.7 The escape key has dual functions: Hitting it once allows you to change the selection filter, typically between \*.raw and \*.txt. Hitting the escape key twice without changes exits the xch program.
- 9.7.8 Once back in DOS use the copy command to write the files to a floppy disk: copy filename.txt a:\.
- 9.7.9 Once you know the files have been successfully copied please delete the originals from the hard disk.

## 10.0 Troubleshooting Guidelines

The most common trouble is failure to locate a recognizable diffraction peak. Things to check are:

- 10.1 Is the yellow radiation warning light on top of the cabinet lighted? If not get help, it means the x-ray generator has shut down for some reason.
- 10.2 Is the sample mounted flush with the top surface of the sample holder? Any visible tilt will make it very difficult to find a peak.
- 10.3 Are you *certain* that you know the sample cut and orientation? Occasionally samples get mixed up or mislabeled.

### 10.4 When I use the IN command, the phi axis won't stop nodding back and forth. What should I do?

- 10.4.1 Issue the command **RC0** (zero, not O) to make xdif pay attention to the pull-out keyboard.
- 10.4.2 Go to the keyboard and pres **[shift]**, then **CIRCLE 4:3** to put the phi axis in manual mode.

**Note:** The shift-key is sequential, not like a typewriter. While holding the tuning button down, press the right arrow three times. This will rotate the phi axis 10-15° ccw (as seen from above), past the initialize position.

10.4.3 Go back to the computer and issue **RC1**, then the **IN** command. If this does not do the trick, ask for help.

10.5 Not being able to save data and make a plot. As more and more users make measurement using xdif, the storage space can be very limited if some users do not delete their old measurements. Go under DOS environment and check disk space using command dir. If the free space is less than 3 kB, no new data file can be created. Users can either delete some of their own files or contact other users to delete old files.

## **11.0 Figures & Schematics**

## **12.0 Appendices**

### **12.1 Checking the Monochrometer**

**Q: I really think the monochrometer is not working correctly. How do I check it?**

A: The monochrometer has proven to be exceedingly reliable unless tampered with, so please, do not, however much you may be tempted to, adjust it.

To see if it is working normally:

- 1) Set the generator to 20 kV and 5 mA, take out detector slits, filter, pressure plate and spring from the sample holder.
- 2) Lock the sample to the detector, set chi and phi to zero and do a normal coupled scan about zero, say, -3 to 3 degrees.

The locked sample angle and normal coupled scan is just an easy way to keep the detector from colliding with the goniometer; what you're really doing is a detector scan.

For thoroughness use a resolution of .005 degree; you should get a signal about .6 degree wide, centered near + 0.03 degrees, about 10k CPS full scale.

- 1) If the amplitude is low, make sure the nickel filter is out.
- 2) If the peak is narrow, check that no slit is present in the detector.
- 3) If the amplitude is high, check the generator voltage and current.
- 4) If the angle is off try to gently wiggle the monochrometer on its stand; if it moves at all shift to toward the cabinet door and gently snug the (small and hard to reach) thumbscrews.

**PLEASE, DO NOT TOUCH THE BLACK KNOBS!!**

If the signal is too wide check the middle slit in the monochrometer, should be 2 mm.

If you discover a problem not covered here ask for help. Adjusting the monochrometer is a lot of tedious work. After reading how it is done, you will understand the injunction against tampering.

## 12.2 Monochrometer Adjustment Procedure

### **D5000 Four Bounce Monochrometer Adjustment Procedure**

Prepared with the help of:

**Lutz Bruegemann**, Brucker AXS

**Mike Cich**, Materials Science

**Betty Clark**, Tang Center

**Frederick Hollander**, College of Chemistry

**Rod Warren**, Office of Radiation Safety

#### 1. Introduction

The monochrometer on xdif is of a design by Bartels [J.W. Bartels, J. Vac. Sci. Technol. B 1(2), Apr-June 1983, pp. 338-345]. Two channel cut germanium crystals are mounted as periscopes in opposition. The radiation diffracts from the first surface to the second, where it diffracts again with direction unchanged but position displaced by the projected width of the channel. The second crystal reverses the displacement, so the beam direction and position are identical to the original beam.

The only adjustment attempted so far is to the rotation of the crystals about a vertical axis; all other degrees of freedom can be adjusted and should be left alone insofar as possible. The monochromatized beam is too feeble to be easily visible using scintillator screens and extremely difficult to find if more than one orientation is wrong.

The procedure described takes about 3 relaxed days' work. It is very important to avoid haste; the shutter must be closed and the door opened for **every** adjustment, making for slow progress.

#### 2. Finding The Beam

Tape markers have been put on the crystal turntables and adjusting screws. The markers record the last known working adjustment and will give you a good clue if the monochrometer has been tampered with. Aided by the markers you can probably omit the initial crystal setting and peak identification steps.

To start without any initial information, here is the process:

The crystal nearest the xray source should be set mechanically to about 22.6 degrees counterclockwise (viewed from above) from parallel to the baseplate. The sample side should be set 22.6 degrees clockwise. This selects the 022 reflection planes, the strongest available. The two crystals must now be brought into an exact match: The first is sure to receive radiation it can diffract to the second, but the second must be set to diffract in agreement with the first.

A systematic search is the only way to find the correct angle.

The 6 mm slit should be installed between xray tube and first crystal and 2 mm slit between crystals. The monochrometer cover should be installed; it reduces scatter. Note that the 6 mm slit is covered to limit beam height; that limits the flux of photons coming over the tops of the crystals. Make sure the height limiter is in place, else you will see spurious radiation. Be sure the nickel filter is out of the detector. Slits in the detector should be removed also.

Set the source to 40 kV and 30 mA. Set theta, phi and chi to zero. Remove the sample pressure plate and spring. Do a detector scan about zero, say -5 to 5 degrees with a step-size of .25 degree for the initial search. You'll get a wide bump of a thousand or so counts. Now increment the sample-side crystal adjustment by no more than one-tenth turn and repeat the detector scan. Keep going for about 2 full knob revolutions. If you don't find a peak, go back to where you started and run the other way. How far you must search is determined by how well you matched the two crystals to start with. Two full knob revolutions are only about 1 degree, so you might need to go further if the initial settings are less than perfect.

After you have located a peak, determine if it is k-alpha1 or k-alpha2. They're about a quarter turn apart, with alpha1 in the more clockwise position **ON THE SAMPLE-SIDE-CRYSTAL**, other way around for the tube-side crystal. At 20 kV/5mA, alpha1 is presently a bit over 10 K cps full scale with a 2 mm slit between the monochromator crystals, alpha2 about half that. With a brand-new tube the intensities should be about doubled those.

Stick to adjusting the sample-side crystal to start with. Later, if necessary, the tube-side crystal will be adjusted.

### 3. Optimizing The Crystal Orientation

Once you find the correct peak, home in on it carefully. Turn down the generator voltage (20 kV and 5 mA gives an adequate signal) once you're in the right ballpark. The smallest adjustment which can be felt makes a big difference close to the peak and backlash in the worm gears is noticeable on this scale. It is best to always adjust in the same direction else life gets confusing. The first few times, you will overshoot, but several passes are needed anyway to make sure you've really found the peak; the peak is so narrow it is easy to miss the top.

### 4. Locating The Beam

Two things have to be measured, the position of the cradle center, and the location of the beam. The cradle center is largely defined by its manufacture; the only thing we need to check is the required value of Z. Several jigs are provided to aid this project. There is an alignment spike, a stage mount for slits and a glass tunnel slit. All are constructed to have the same height from mounting surface to working surface. There is also a telescope which mounts opposite the dial indicator port. Mount the spike, put the stage to nominal center; that's x and y at mid-scale, z at about - 0.4 mm, chi and phi at zero. Do a chi scan. Adjust x and z until the tip remains stationary in the telescope field. Note that it will not necessarily be exactly centered: that's not what counts. It need only be stationary.

To find the beam location it is necessary to scan a slit through the beam, recording intensity as a function of z value. Set z, do a detector scan about zero, record the peak and increment z. Start with large slits, reducing size as you get a better idea of beam position. Remember to center x and y, though neither has much effect at small theta. Chi and Phi should of course be zero.

The final scan works nicely with a 0.2 mm slit between the crystals, a 0.05 mm slit on the stage and a 0.1 mm slit in the detector. The count rate should be around 200-300 if everything else is ok. At this writing the beam is at z = - 0.42 mm, .02 mm below the cradle center.

The slit scan will find if the beam's crossing the center of the Eulerian cradle but not its direction. To do that use a rocking curve with the glass tunnel slit, scanning again in z as before and rocking about theta to see where the intensity is a maximum. Use a 2 mm slit between the crystals and no slit in the detector; extra slits will just make the system sensitive to spurious upsets. You'll probably find the beam does not center on zero in neither theta nor two theta. The factory spec is 1 degree, and mounting errors are usually bigger, so don't worry too much about it. The test is mostly a sanity check (which you will sorely need).

### 5. Aiming the Beam

The xray-tube side crystal orientation influences the aim of the beam across the goniometer, while the sample-side crystal dominates intensity. You will probably find the beam peaks somewhere other than zero degrees. If it is within a degree of zero, count yourself lucky, and the job done factory tolerance is one degree either way. In fact, the angular value is not significant, except as an indicator that maybe something has changed since the last time you checked. Remember, real zero is defined by having the beam pass through the center of the Eulerian cradle.

If the peak intensity is away from the cradle center by, say, 10% of the beam size (2 mm normally) it is worthwhile to re-aim. Clockwise rotation of the xray-source side knob will shift the beam to more positive z values. Progress is best monitored by watching the detector angle change; roughly one turn of the xray-tube-side crystal knob moves the beam about 1 degree more positive and 1 mm more positive in z [**Warning**: Approximate numbers].

The two crystals must be moved together: The movements will be equal and in the same direction, but backlash will make reversal clumsy, so don't be too fussy. Get it close, and leave well enough alone! It is helpful to **walk** the two crystals alternately, that keeps them in sync. Move one until the intensity drops, then move the other to catch up. Slow, but safe.

Shift the beam over slowly by alternately adjusting the crystals. If you lose the beam, immediately take out the stage and detector slits, put the 2 mm slit between the crystals and re-scan; don't risk getting lost.

The displayed angle readings can be re-zeroed if desired; see the D5000 manual for detailed instructions using DiffracAT.