Operation Manual for the Scintag θ/θ Diffractometer & DMS-NT Software

By Edward Laitila
Michigan Technological University
May 17, 2012
Version 1.20

REFERENCE GUIDE TO PERFORMING A DIFFRACTION SCAN

Diffractometer Initialization:

1. Sign the logbook and record the hours to the nearest tenth position. The meter is located next to the red safety switch light above the generator.
2. Press the Amber **CONTROL POWER ON** button.
3. Press and hold the green **X-RAY OFF** and observe that the kV is 10 or 15 and the mA is 02. These minimum settings and must be correct before the x-rays are engaged if not turn the respective dials while holding the **X-RAY OFF** button to the proper settings. **Do not proceed until the values are correct.**
4. Press and hold the red **X-RAY ON** button for about 3 seconds or until the second click sound. Wait for the mA's to reach set point before proceeding. **This engages the generator and starts x-ray production, proceed carefully.**
5. Push the locking lever on the kV dial to the left to release the mechanism. Slowly turn up the kV's to the nominal setting of –45. **Make sure the kV's are turned up first and do not exceed this value unless you consult Ed Laitila.** Push the locking lever to the right to lock the mechanism.
6. Push the locking lever on the mA dial to the left to release the mechanism. Slowly turn up the mA's to the nominal setting of 35. **Make sure the mA's are turned up after the kV's and do not exceed this value unless you consult Ed Laitila.** Push the locking lever to the right to lock the mechanism.

    *WARNING: DO NOT EXCEED THE POWER RATING OF THE X-RAY TUBE. EXCEEDING THE MAXIMUM POWER OF 2000 WATTS WILL CAUSE IRREPARABLE DAMAGE TO THE TUBE. (kV * mA) = WATTS*

7. Open cabinet door to permit access to the goniometer and insert sample into sample holder (insert side drifted holder with open end toward the tube). Check that the proper beam slits are in place, standard slits are: 1, 2, 0.5 , and 0.3 mm.

8. Close cabinet door and press the red safety button (which should be illuminated) located on the lower right hand corner just below the cabinet. If this button remains illuminated the shutter will not open.

Diffraction Scan Set-up:

1. Turn on the monitor by pressing the monitor on button.
   If computer is not logged on use local login with username **xrd** and password **xrd (this must be used for Warren data collection computer).**. Note this will change to ISO userid and password once all computers are networked to the campus.
2. Right click the **START** button, then select the **DMSNT** icon to activate the diffraction software.
3. Select **New Event List** to create a new scan or select **Open** and select **Files of type** with the down arrow choose file type **Event Files (*.evt)** to open a previous scan parameter file.
4. When opening an existing event list to run previous scan parameters; select **Browse** then select drive **X:**, then XrayData, **diffraction data**, and finally the directory that you created (typically using your username). Type in the name of the file for your scan. Make sure that the default file information is deleted (typically the default path name is highlighted in blue so if you start typing this will automatically be over written) the only text being your filename. Next type identification information into the **ID** box, its important to be specific about your sample (e.g. composition, heat treatment, mechanical treatment, etc.) to eliminate problems with cryptic naming schemes that need other information. Additionally, more detailed information can be included in the comment box.
5. When selecting **New Event List,** the screen will appear the same as shown below.
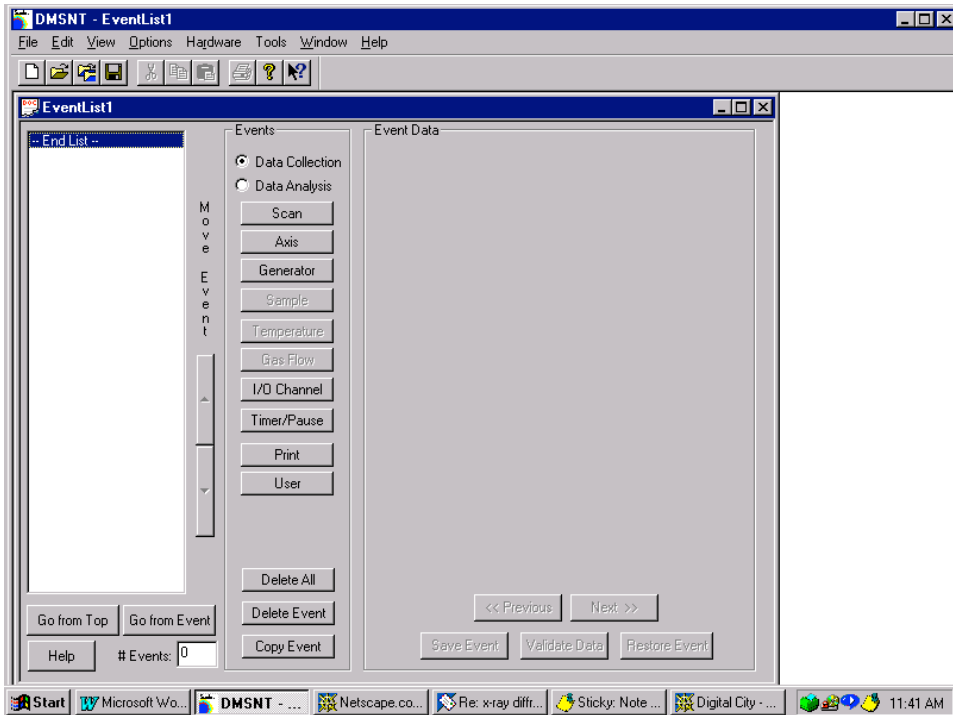
6. Select **Scan** to initiate a new scan. The screen will then change to the following.

WARNING: DO NOT SELECT OTHER TYPES OF GONIOMETER OPERATIONS UNLESS YOU FULL UNDERSTAND THEIR OPERATION.
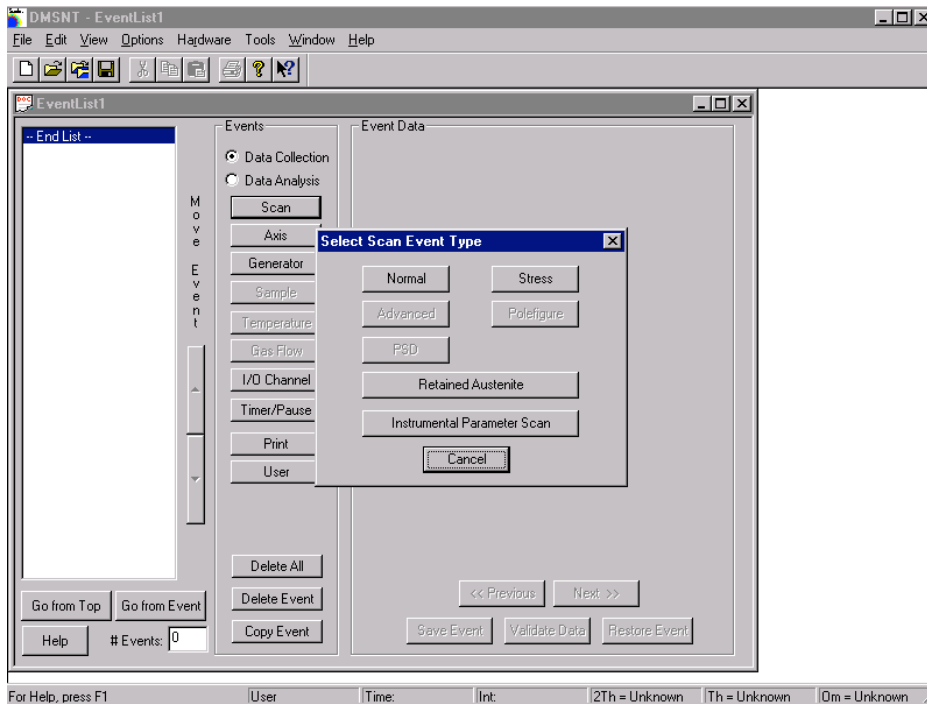
7. Select **Normal** as the scan event type.
8. Event Data dialog box should now be present that contains three folder tabs of scan information as shown below. First, select **Browse** and then select drive **X:**, then **Diffraction Data**, and finally a directory that you create typically using your username. Type in the name of the file for your scan. Make sure that the default file information is deleted (typically the default path name is highlighted in blue so if you start typing this will automatically be over written) the only text being your filename. Next type identification information into the **ID** box, its important to be specific about your sample (e.g. composition, heat treatment, mechanical treatment, etc.) to eliminate problems with cryptic naming schemes that need other information. Additionally, more detailed information can be included in the comment box.
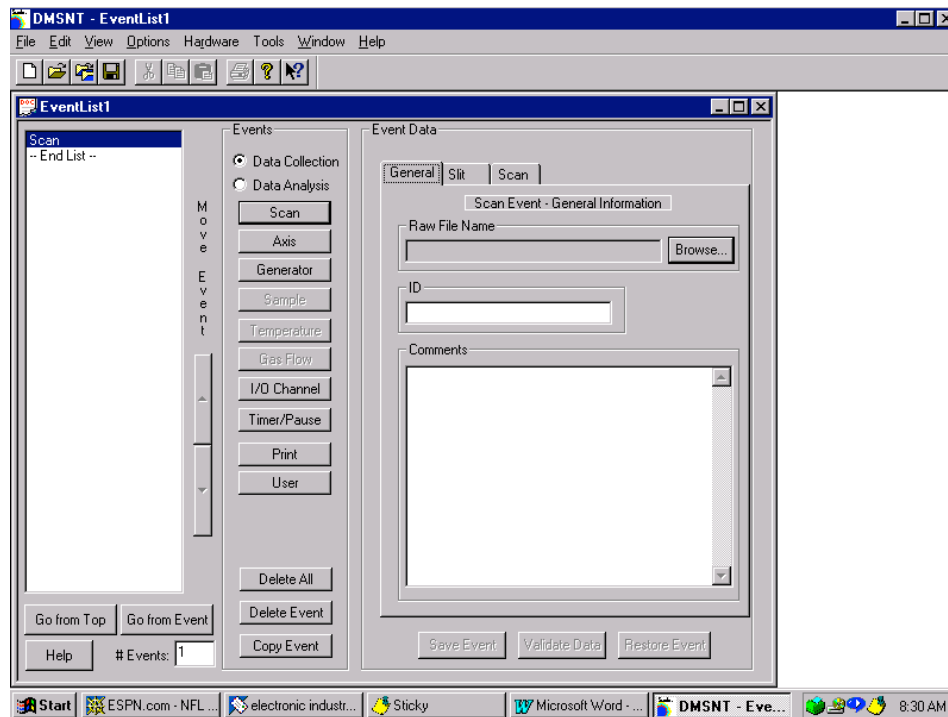
9. Select the **Slit** tab and enter the size of the diffraction optic slits. These are shown in the following figure with the proper values for the nominal settings.
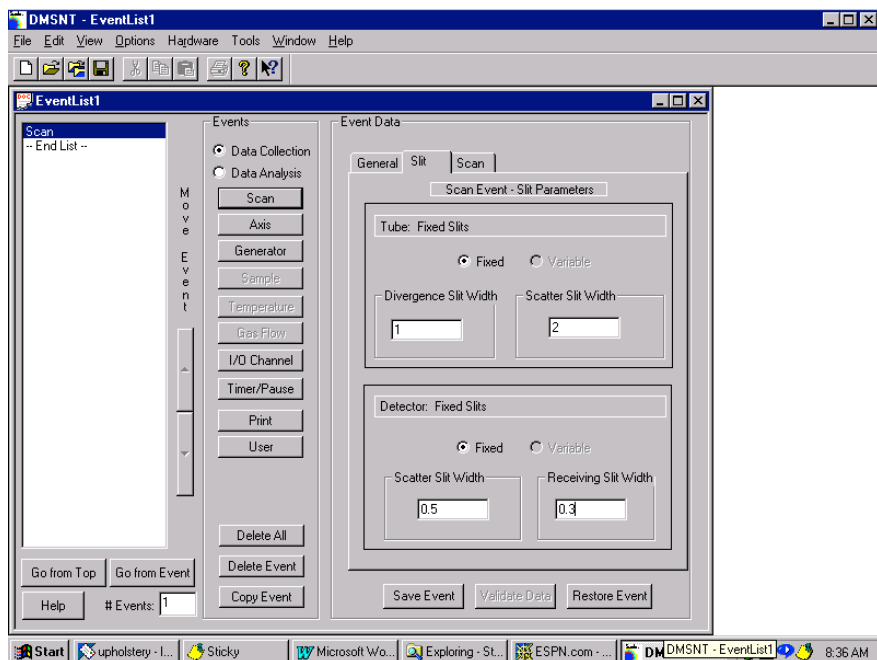
10. Select the **Scan** tab to enter the parameters of the data collection. Enter the desired **Step Size, Start Angle, Stop Angle**. Select the **Scan Mode**. If a **Step** mode is selected enter the **Preset Time** in seconds, or for **Continuous** mode a drop down menu of available scan rates must be selected. Select **Set** to store the inserted parameters in the event file. The following figures show the screen for both options. If a multirange scan is desired it is important to select **Set** for the first range and then input the next range and select **Add** for each subsequent range.


6
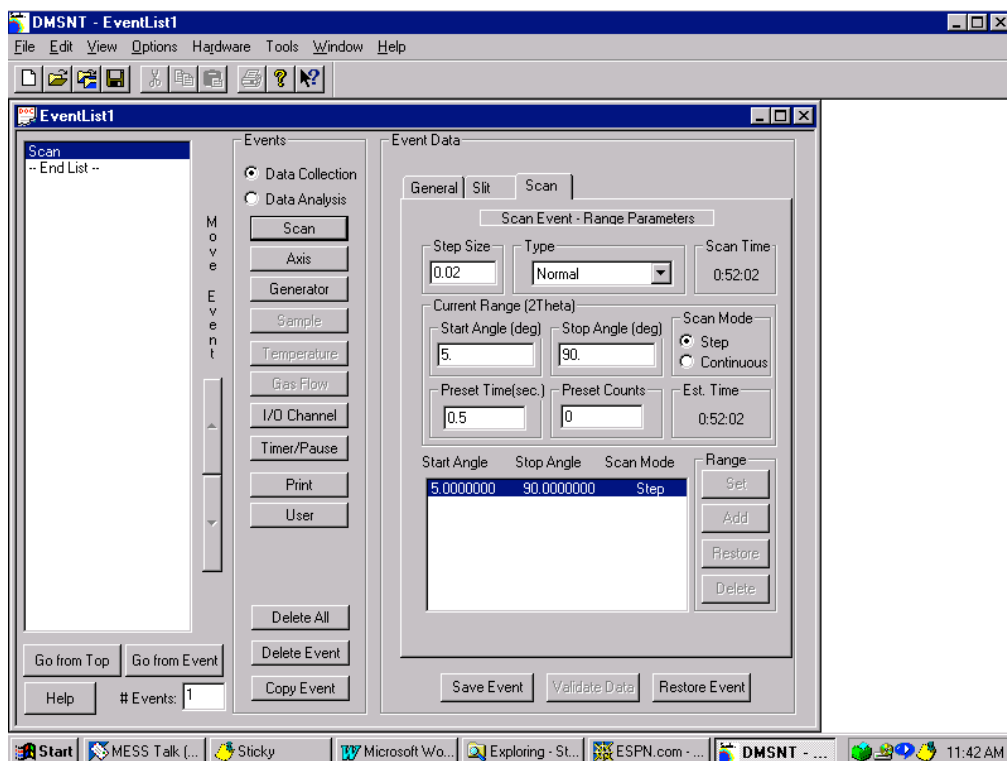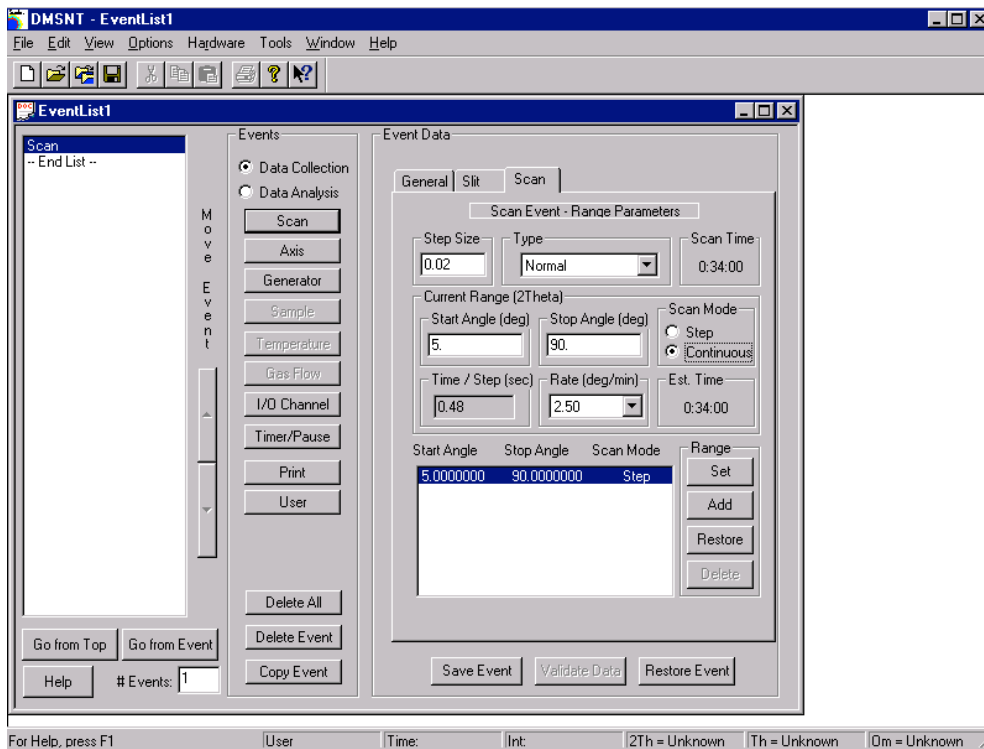
WARNING: FAILURE TO SET THE SCAN PARAMETERS WILL RESULT IN THE SCAN OPERATING WITH THE DEFAULT PARAMETERS NOT THE SPECIFIED PARAMETERS.

11. Select **Save Event** to save the input information into the event file.
12. Select **File, Save as**, and enter a filename for the event list just created. A saved event list with particular scan parameters provides a stored copy of the parameters that can be reused with other samples eliminating the need to reenter the parameters.
13. Select **Go from Top** to start the scan procedure. The following screen appears with a dialog box to save changes in the event file. This will save the latest changes made to the event file.

7
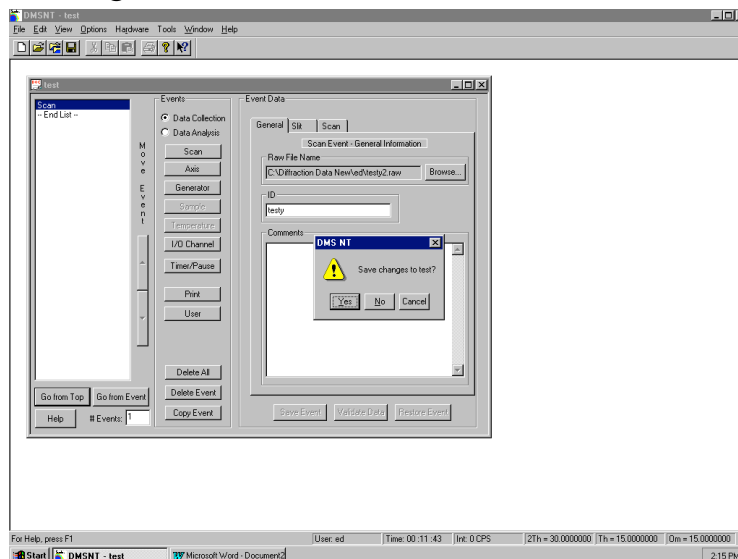
14. The following dialog box will ask if you want to initialize the hardware. You must answer yes otherwise you cannot start a scan. This dialog box will only be present when the first scan is started after logging on to the computer. For successive scans while still logged on to the computer this message will no longer appear. The following screen prompts you to confirm the position of the goniometer. Check the whole degree indicators and then add the vernier dial readings for the decimal places. Note that the vernier dial resolution is 0.01° with interpolation to the nearest 0.0025° between the scale marks. If the angles agree select **Ok.**

8
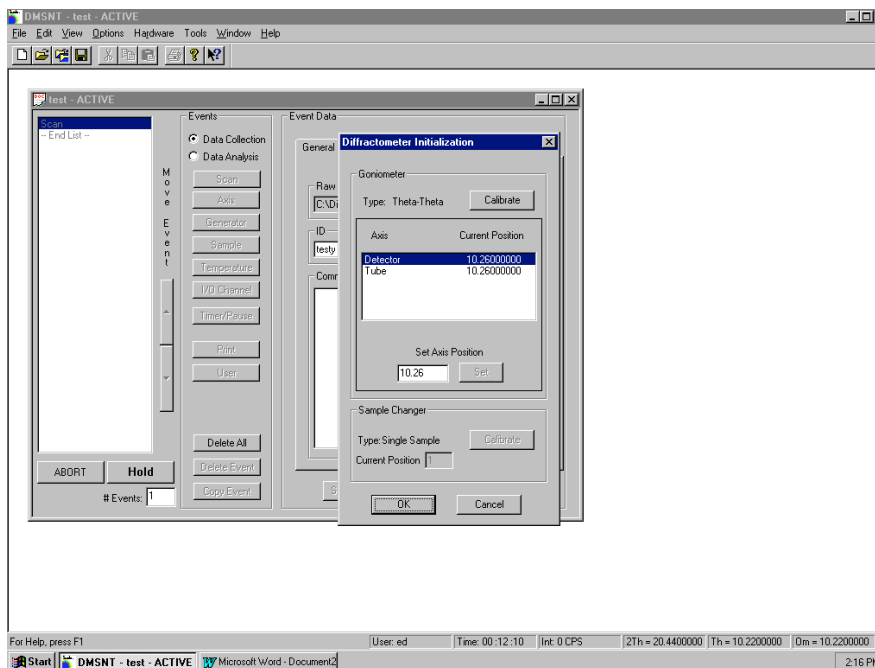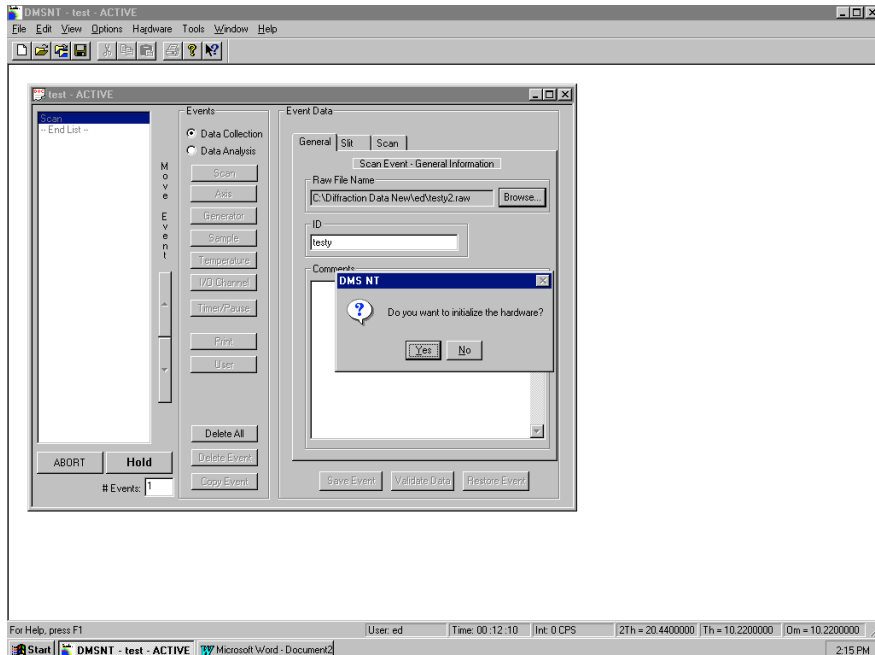
WARNING: MAKE SURE THE GONIOMETER POSITIONS AND THE COMPUTER POSITIONS AGREE EXACTLY. SERIOUS DAMAGE COULD OCCUR IF THESE POSITIONS DO NOT AGREE. IF YOU DO NOT UNDERSTAND HOW TO READ THE POSITION DO NOT PROCEED, SEE ED LAITILA BEFORE YOU CONTINUE. IF THEY DO NOT AGREE PROCEED TO THE CALIBRATION PROCEDURE TO CALIBRATE THE POSITIONS (SEE PAGE 13).

15.   Make sure the goniometer goes to he angle specified in you scan parameters. If it does not then you must abort the scan by pressing the **Abort** button on the lower left-hand corner. Then determine if it is an error on your part in entering your scan parameters or if the instrument is malfunctioning. If a continuous scan is aborted the goniometer must be calibrated, otherwise reenter the proper scan parameters in the event file and restart the scan. Again check to make sure the scan starts at the proper starting angle.

   WARNING: IF THE GONIOMETER FAILS TO START AT THE PROPER ANGLE AGAIN, SELECT ABORT TO STOP THE SCAN, REFER TO THE SHUTDOWN PROCEDURE AND SHUTDOWN THE GENERATOR, STOP ALL ACTIVITY ON THE SYSTEM, THEN CONTACT ED LAITILA.

16.   To observe the scan in real-time select **Real-time Display** button in the **Scan Status** window.
17.   Do not close the DMS-NT software or log off the computer, otherwise the scan will be aborted. Please place a sign on the computer or create a marquee screen saver indicating a scan is in progress.
18.   After completion of your scan time log off the computer and turn the monitor power off by pressing the power button in the lower left hand corner.

Diffractometer Shut Down Procedure:

   Only in an emergency press the green X-RAY OFF button.
1. Push the locking lever on the mA dial to the left to release the mechanism. Slowly turn the mA's down to the minimum value of 02. **Make sure the mA's are turned down first.** Push the locking lever to the right to lock the mechanism.

2. Push the locking lever on the kV dial to the left to release the mechanism. Slowly turn the kV's down to the minimum value of -15. **Make sure the kV's are turned down second.** Push the locking lever to the right to lock the mechanism.
3. Wait approximately 10 minutes for the x-ray tube to cool. Press the green **X-RAY OFF** button.
4. Press the Amber **CONTROL POWER ON** button to complete the power shutdown.

GENERAL COMPUTER AND SOFTWARE INFORMATION

XRD Accounts:

To log on use your ISO userid and password, for local computer acess use **xrd** for the userid and **xrd** for the password. The first time you log on to any of the **X-ray** computers you should create a folder in the Warren computer to store diffraction data collected under **X:\XRayData\diffraction data** typically using your name. This can also be created in network neighborhood.

Computer Architecture:

Currently there are 4 Windows XP computers all have will have campus network acess. However, only authorized users will have acess to Warren which runs all scans on the θ/θ diffractometer. The computer names are Bragglaw, Averbach, Guinier, and Warren. Please see Ed Laitila before you put any software on these computers **All diffraction scan data is stored on the X: drive on the Warren computer which also controls the θ/θ diffractometer.** The directory used for diffraction data storage on Warren currently is X**:\ XRayData\diffraction data.** In order to access diffraction data from any computer except for Warren go to network neighborhood and select **Warren, XRayData, Diffraction Data.**

WARNING: NEVER CLOSE THE SOFTWARE OR LOG OFF COMPUTER WARREN WHILE THE DIFFRACTOMETER θ/θ IS OPERATING. THIS PROCEDURE WILL ABORT ANY SCAN CURRENTLY OPERATING.

CALIBRATION PROCEDURE

Notice: The conditions under which a calibration is necessary is after a power failure or if a continuous scan is aborted. Despite this fact, one should always be in the habit of checking that the 2θ position indicated by the computer agrees with the 2θ position of the goniometer, since the computer has no feedback mechanism for keeping track of the goniometer position.

Important: The 2θ position is the sum of θ and Ω. The θ axis is the detector axis and the Ω axis is the tube axis.

1. Select the **Calibrate** button when prompted to initialize the instrument when starting a scan.
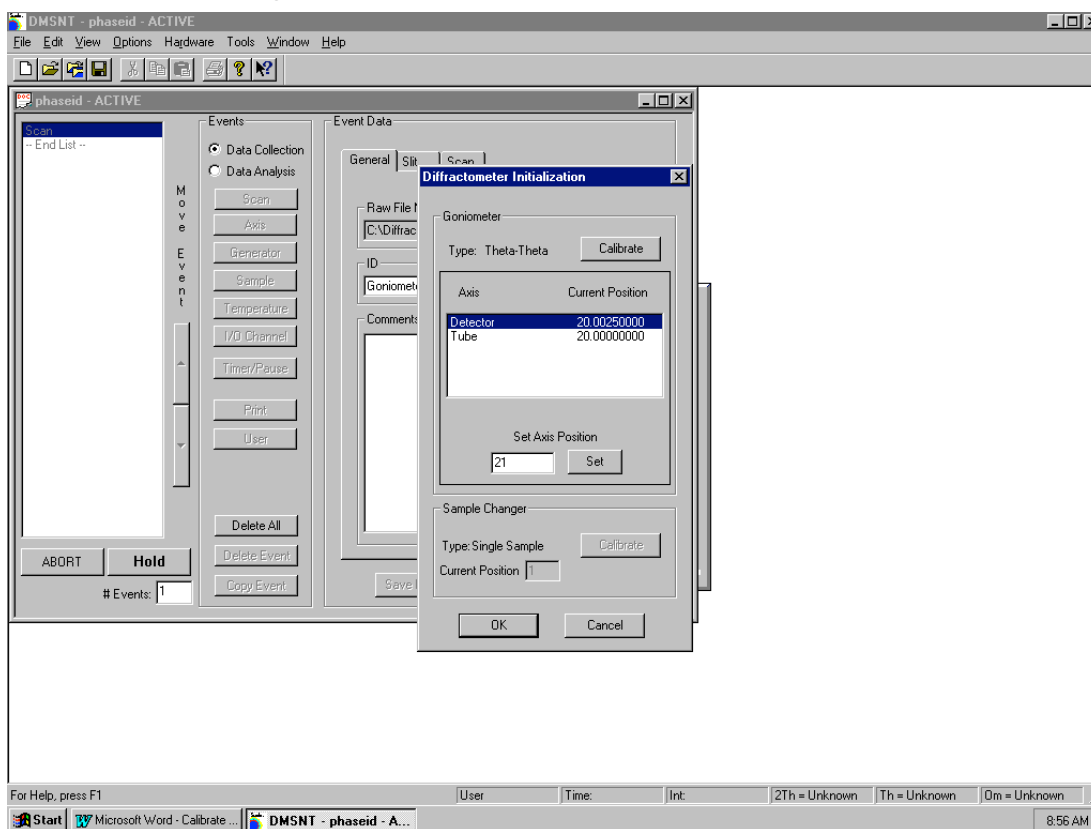
WARNING: DO NOT PROCEED UNLESS YOU FULLY UNDERSTAND HOW TO READ THE GONIOMETER POSITIONS AND THIS PROCEDURE. FAILURE TO PROPERLY CALIBRATE THE INSTRUMENT CAN RESULT IN DAMAGE TO THE EQUIPMENT. PLEASE CONTACT ED LAITILA IF THERE IS ANY QUESTION IN REGARDS TO CALIBRATION.

11

2. Wait until the goniometer stops before proceeding. The computer will drive the θ and Ω axis to the nearest whole degree. However, in some cases the axis may not stop at a whole number degree in that case the position must be interpolated to the nearest 0.0025°. Remember the 2θ position is the sum of θ and Ω.

3. Select the **Detector** axis so that it is highlighted in blue. Determine the position of the θ (detector) axis and in the box under **Set Axis Position** enter the value determined from the goniometer position. Select the **Set** button. The position should now reflect the inserted position if it does not reenter the position.

   WARNING: FAILURE TO HIGHLIGHT A PARTICULAR AXIS WILL NOT ALLOW THE NEW POSITION INSERTED TO CALIBRATE THE AXIS OF INTEREST. THIS WILL RESULT IN A SERIOUS OR DAMAGING POSITION ERROR. IN EFFECT ONLY THE AXIS HIGHLIGHTED WILL BE CHANGED WITH THE SET BUTTON.

4. Select the **Tube** axis so that it is highlighted in blue. Determine the position of the Ω (tube) axis on the goniometer and in the box under **Set Axis Position** enter the value determined from the goniometer position. Select the **Set** button.

   WARNING: MAKE SURE THAT EACH AXIS IS AT THE PROPER POSITION, DO NOT PROCEED UNTIL EACH AXIS IS AT THE PROPER POSITION.

5. Finally select **Ok,** the calibration will be complete and the scan will start.

Reading the Angular Positions of the Goniometer:

To read the angular position you must read both the dial indicator on each axis which gives the whole number of degrees, and add the value of the vernier dials on the top of the goniometer which gives the fractions of a degree. The vernier has 100 divisions so each division is 0.01°. More importantly each 0.01° division can be interpolated to the nearest 0.0025°. The value or 2θ is the sum of Ω and θ angles.

<u>**Sample Size Specifications**</u>

Powder Sample Holders Available:

All specifications are determined for the inside dimension and given in the L X W X H format.

Top Loading Holders:

Large Plexiglas holder 2.6 X 2.6 X 0.1 cm. Volume = 0.675 cm^3.
Small Plexiglas holder 1.6 X 1.6 X 0.05 cm. Volume = 0.128 cm^3.

Side Drifted Holders:

The valid minimum 2θ for quantitative analysis using peak areas is 10° and above for the STD and shallow holder, and 5° and above for the long holder.
Shallow aluminum holder 4.0 X 1.9 X 0.04 cm. Volume (max.)=0.305 cm^3.
Std aluminum holder 4.0 X 1.9 X 0.125 cm. Volume (max.)=0.95 cm^3.
Long aluminum holder 6.58 X 1.9 X 0.110 cm. Volume (max.)=1.375 cm^3.

Zero Background holders:

The larger sample holder mounts directly into the goniometer holder and powder can be sprinkled on top, useful for small amounts of powders. The smaller holder is used in conjuction with the solid holders mounting this on clay with the powder on top and pressed down with a thick glass slide.

Liquid Holder:

Liquid holder 9.35 X 2.275 X 0.428 cm. Volume = 9.1 cm^3.

Solid Sample Holders Available

Low aluminum holder, maximum sample width = 2.525 cm, maximum sample depth = 0.925 cm, and maximum sample length = 20 cm (see Ed Laitila if sample length is greater). Note about clay.

High aluminum holder, maximum sample width = 2.525 cm, maximum sample depth = 1.26 cm, and maximum sample length = 20 cm (see Ed Laitila if sample length is greater).

Goniometer sample holder, sample width must be 3.0 cm, with a maximum depth of 1.3 cm, and a maximum length of 20 cm (see Ed Laitila if sample length is greater).

Sample Preparation Guidelines

- Make sure you place the weight on the clay mount samples for at least 5 minutes.
- In many cases polycrystalline samples should be polished and etched. As a rule of thumb, repeat the last polishing stage and etch 3 times, to be confident that surface deformation from polishing has been removed. Last preparation step is etching.
- When possible, do not mount samples in Bakelite or other crystalline substances. If mounting is required use a cold mount epoxy.
- Ideal grain size or particle size for powders, for quantitative intensity measurements, is less than 10 μm (ideally 1 μm).[1]

REFERENCES

1. JCPDS Data Collection and Analysis Subcommittee, Sample Preparation Methods in X-Ray Powder Diffraction, Methods and Practices in X-Ray Diffraction, 5.2.1.