

OPERATING PROCEDURES FOR X-RAY DIFFRACTOMETER

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Equipment Description

Scintag PAD V Automated X-ray Diffractometer
Controlled by Scintag's DMSNT proprietary software

Powering On the Diffractometer Generator

- ◆ Be sure the on-board computer is powered up (back left side of the generator – normally this will always be running, but if the following procedures fail, press the square, red button firmly and try again. The light will not illuminate.)
- ◆ Set the thumb-wheel switches on the front of the generator to 10 mA and 10 kV
- ◆ Turn the main power knife switch on the wall to the <On> position (lever up)
- ◆ Press the <Start> button to the Haskris recirculating water cooler. The amber light on the front of the generator should come on.
- ◆ On the front of the generator, turn the key as far as possible in the clock-wise direction, release, and be sure it springs back to its original position. The green light on the front of the generator should come on.
- ◆ Start the DMSNT software on the desk-top computer.
- ◆ Select <Hardware>
- ◆ Select <Manual Commands>
- ◆ Select <Generator>
- ◆ Select <Power On>. You should hear a click at the generator and the meters should advance to 10 mA and 10 kV.
- ◆ Increase the thumb-wheel switch for the voltage step wise to 40 kV
- ◆ Increase the thumb-wheel switch for the current step wise to 30 mA

Initializing the Goniometer

- ◆ Within DMSNT, select <Hardware>, <Manual Commands>, <Goniometer>, and <Initialize Hardware>.
- ◆ Select <Calibrate>. The calibrated dials on top of the goniometer should advance to the next rational position.
- ◆ Visually determine the actual angle position of the 2-theta and omega drives (pencil-shaped metal indicators on the left side of the goniometer face).
- ◆ Highlight the 2-theta line in the computer window, then select <Current Position>, enter the observed position for 2-theta and press <Set>.
- ◆ Repeat this process for the omega position.
- ◆ Select <Next 2-Theta> and enter a value of 10 (or any other number between 2 and 80) in the little window, then select <Move>. The goniometer should move to the 2-theta position entered and reconcile the omega position to half that value.

Scanning a Sample

- ◆ Select <File>, <Open>, then browse for a file with the extension “.evt”
- ◆ When the file is open, select <Data Collection>
- ◆ If you have already established an event file that fits your situation, you skip directly to the next step. If you wish to edit the file, you may change the parameters, such as for the temporary file in which the raw data will be stored, the range of angles to be scanned, the scan rate, and other events that are desired. Save the file under a user-defined name in a user-defined directory. The temporary file that is created with each scan will be overwritten with each scan, so it should be renamed to a descriptive name selected by the user following each scan.
- ◆ Select <Go from top>
- ◆ If you want to observe the pattern as it is collected, in the progress window select <Real-time Display>.
- ◆ When the scan is complete, using Windows Explorer find the temporary file that was saved and rename it to the permanent name.

Analyzing the Data

- ◆ Within the event file window, select <Data Analysis>
- ◆ You may then select from several types of data analysis routines, including background correction and peak finder. You can also print the file, label peaks, and export the data to an ascii file for handling in other software packages.
- ◆ You may also select the raw data file later and analyze the data at another time. It need not be done immediately.

Powering Off the Generator

- ◆ Decrease the thumb-wheel switch for the current step wise to 10 mA
- ◆ Decrease the thumb-wheel switch for the voltage step wise to 10 kV
- ◆ The meters should both be reading 10

At this point, either of the following two procedures may be used:

Procedure 1:

- ◆ In the DMSNT software on the desk-top computer, Select <Hardware>
- ◆ Select <Manual Commands>
- ◆ Select <Generator>
- ◆ Select <Power Off>. You should hear a click at the generator and the meters should drop to 0.

Procedure 2:

- ◆ Press the large red button on the front of the generator. The generator should turn off.

After executing one of the foregoing procedures, do the following:

- ◆ Exit DMSNT

- ◆ Press the <Stop> button on the Haskris water cooler switch
- ◆ Pull the X-ray power knife switch on the wall down to the <Off> position