

# User guide for Scintag powder diffractometer

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## • Preparation of your sample

Quartz or glass sample slides and plastic holders are available in the x-ray lab. It is recommended to use the Quartz plate because it gives less background. If your sample is not enough to fill in the area on the quartz plate, use the glass plate instead. Make sure your material is grinded finely using the mortar and pestle. Place your material in the center of the plate (rectangle or oval shape, extending from top to bottom across the slide oriented as it will be on the specimen) to maximum X-ray diffraction from your sample. If you use plastic holder make sure to fill in whole area and create flat surface of the sample material.

## • Before your use

The detector should be cooled down for the measurement at least 1 hour before the operation. Check the cooling status beneath the detector, the red light should be OFF for cooling (red light shown on the right picture means detector is warm). Check this red light before your use every time. Please plan/book your schedule in advance on the online calendar so that the lab manager can keep the detector's dewar filled with LN<sub>2</sub> ready for your use. If you see the red light let the lab manager know to fill LN<sub>2</sub>. Be sure to check the x-ray light is set to OFF too (X-ray power light should be ON). If you observe anything unusual, contact Rie.



## • X-ray tube warm up

The standby condition for the x-ray tube is at 10kV and 2mA. For the measurement the x-ray tube should be warmed up to 45kV and 40mA **slowly** using the knobs. Here's the procedure:

1. X-ray tube at 10kV and 2mA, turn to 15kV then to 4mA (wait 20 seconds).
2. Turn to 20kV then to 6mA (wait 20 seconds).
3. Turn to 25kV then to 8mA (wait 20 seconds).
4. Turn to 30kV then to 10mA (wait 20 seconds).
5. Turn to 35kV then to 12mA (wait 20 seconds).
6. Turn to 40kV then to 14mA (wait 20 seconds).
7. Turn to 45kV then to 16mA (wait 20 seconds).
8. Turn current up to 40mA over 20 seconds.



- **Si standard check**

Before start to measure your own sample, always run the Si standard to check the peak position and intensity. Save your Si standard data under **c:\Document and Settings\geouser\MDI Jade 6\Si\_standard\Si\_standard\_date.MDI**

The run file for this measurement is saved on the control program named “Si standard”.

- **Load your sample**

1. Read the value of the outer detector ring ( $=2\theta$ ) and inner sample holder ring ( $=\theta$ ). These positions should be at  $90^\circ$  and  $45^\circ$ .



2. Check the Chi and the Phi. They should be at 0 (zero).
3. Load your sample slide on the specimen. Place paper under the specimen to keep the machine clean from your material. Close the cover door to the Scintag (make sure you grasp the handle in the middle, not the right end).



3. Open the **MDI DataScan4** control software on the Scintag computer (right screen on the table).

- **Measurement**

1. Set scan parameters ( **$2\theta$  range, steps and exposure time**) for your measurement. (You may have to experiment to find the best parameter for your sample, but some possible values might be: step size: no more than  $0.01^\circ$ , time: 0.5 second. To check the quartz standard use:  $2\theta = 20^\circ$  to  $28^\circ$ , step =  $0.02^\circ$ , exposure time = 0.5 second.)
2. To start the measurement, **Scan>routine qualitative scan**.

- **Data analysis**

1. After the measurement your data file is automatically saved under **c:\Document and Settings\geouser\MDI Jade 6\ your\_name\\*.MDI** on Scintag instrument computer.
2. For the data analysis you can open **JADE** program for the phase identification, unit cell refinement, crystal structure refinement/Rietveld refinement etc.

- **X-ray tube desaturation**

If no one is going to use the XRD Scintag machine after you finished, please desaturate the x-ray tube to the stand-by setting. Here's the procedure:

1. The tube at 45kV and 40mA, turn only the current down to 30 mA over 20 seconds. (wait 10 seconds).
2. Turn down to 16mA, then 40kV (wait 10 seconds).
3. Turn down to 12mA, then 35kV (wait 10 seconds).
4. Turn down to 10mA, then 30kV (wait 10 seconds).
5. Turn down to 8mA, then 25kV (wait 10 seconds).
6. Turn down to 6mA, then 20kV (wait 10 seconds).
7. Turn down to 4mA, then 15kV (wait 10 seconds).
8. Turn down to 2mA, then 10kV.
9. Leave x-ray power **ON**.

- **Write down your record on the log book.**

Date, your name, usage time on the machine should be always recorded on the log book which placed by the Scintag diffractometer.