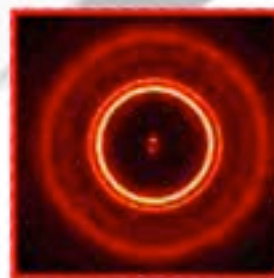
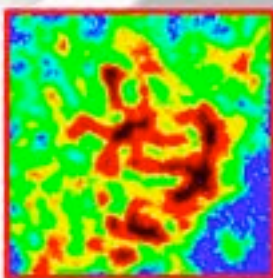
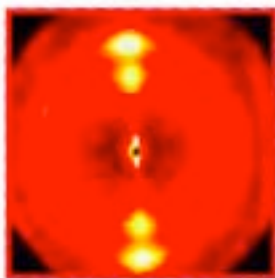


Standard Operating Procedure

Bruker NanoStar SAXS



**all-
in-one-
solution**



Typical SAXS, Nanography, and WAXS pattern

(You must be trained by MSE Staff or Authorized Trainer to use this machine)

Last Revision: 1/30/2009

Location: 2219 HH Dow Building

Hazards: (The following materials and equipment associated with this procedure present exposure or physical health hazards. Safety precautions are prudent and mandatory.)

This unit produces a high intensity X-ray beam. Take all precautions to avoid exposure to the x-rays.

Engineering Controls: [Prior to performing this procedure, the following safety equipment must be accessible and ready for use: (e.g. chemical fume hood, biological safety cabinet, laminar flow hood, chemical spill kits)]

None

Protective Equipment: [Prior to performing this procedure, the following personal protective equipment must be obtained and ready for use: (acid resistant gloves, safety eyewear, lab coat, chemical splash apron)]

You must wear radiation dosimeters when operating this equipment. The dosimeters will be checked every 3 months to determine whether you have received any x-ray exposure.

Waste Disposal: (This procedure will result in the follow regulated waste which must be disposed of in compliance with environmental regulations)

None

Accidental Spill: (In the event that a hazardous material spills during this procedure, be prepared to execute the following emergency procedure)

Not applicable.

Operation Procedures:

Machine Controls and Indicator:



On the left control column:

STOP button: If hit it switches off the control electronics. The X-Ray source is turned off and all moving drives will stop instantly. The stop button should only be used in emergency situations and not for normal shut down of the diffractometer system.



Light button: This button turns ON/OFF the fluorescent tube located at the ceiling of the enclosure.



Fan button: This button turns ON/OFF the fan located on top of the enclosure.

System Instrument-Key Switch: This key will lock/unlock the system. During normal operation the switch must be in position “unlocked”.

Open Door button: This button must be pressed to open the front door. If the Open Door button is activated while the tube window is open the X-ray shutter will close automatically.

Ready light: Status of High Voltage Generator. Illuminated orange display indicates that the high voltage generator is operating. Flashing display indicates the high voltage generator is ramping up.

On light: Illuminated green display indicates the control electronics are ready.

Alarm light: Illuminated red display indicates a pending system alarm, flashing red light indicates a pending warning.

Busy light: Illuminated yellow display indicates that a measurement is in progress.

Flashing yellow display means that the system is not ready.

Right column:

Stop button: Function same as **Stop** button on left column.

 **Power OFF button.** It switches off the control electronics, the high voltage generator and all components connected to the AC outlets.

 **Power ON button.** It switches on the control electronics, the high voltage generator and all components connected to the AC outlets.

High Voltage- Rotary Switch. Used for turning high voltage on or off.

Ready, On, Alarm and **Busy** lights have same functions as those on left column.

1: Sign in Log sheet.

2: Check the machine status on either left or right column before using the machine.

If the “Ready” and “On” lights is on, the “X-Ray ON” lights on top of sample chamber is on, skip the following step and go to step 3. Otherwise follow the step to turn on X-Ray.

Turn X-Ray on:

- If SAXS is open, close SAXS or go to Manual Control Mode in SAXS, Open D8 tools, Online status, online refresh
- Select "X-ray generator" , then "X-ray software safety circuit capture register"
- Under "Utilities" select "Xray", then "Reset Xray software safety circuit capture" (ignore warning if present)
- Click OK. Password is "password"
- Under "Utilities" select "Xray", then "Reset Xray hardware capture register" (ignore warning if present)
- Select "X-ray generator" again,
- Under "Utilities" select "Xray", first select "Switch-off circuit OFF", then select "Switch-off circuit ON"
- Turn HV switch on machine clockwise and hold it until X-Ray light come on. Then leave switch in vertical position.

After X-Ray is on please check Positioning Drive X, Y and RefSampleWheel. If Actual Positions of any of the axes has “.-”, go to the axis, click double-arrow toolbar icon "adjust" , The axis should move to lowest position and (get position to right of decimal point X: 0.000, Y=0.000, RSW=4.010). Then quit the D8 tools.

3: Open SAXS program, it will prompt “set generator to user setting: 40 KV and 35 mA” Click Yes. The value will actually at “23KV, 14mA” Go to Collect\Goniometer\Generator, change to 40KV, 35mA and check “Wait” and OK.

4: For new user or new project, go to Project\new, input info accordingly. Use working directory C:\frames\users\”unique name”\”project name”

5: Install Sample:



Close valve 1 and valve 2 under the counter. Turn both knobs CCW to vent the chamber. Push “open door” button on control column, you should hear a click sound. then turn the door handle CCW and open the door.

Install your sample on sample holder.

Close the door firmly and turn the door handle until you hear a click.

Turn off both Venting knobs.

Open Valve 1 and 2. Proceed when vacuum is better than 10^{-1} mtorr.

6: Sample alignment and setting targets:

You can use Manual Controller to move your sample.

To use Manual controller, press Ctrl+Shift+M in SAXS program.

The Manual Controller is a general controller and not all the functions will be needed for the SAXS. The axes we used here is:



5- X (range: 24~45)

6- Y (range: 15~95, position of 9th hole is 62)

8- Reference Sample Wheel (0: out of beam, 90: in the beam)

Push “↑” or “↓” button will increase or decrease the position of axis.

Push “+” or “-” will increase or decrease the speed of movement.

Note:

You also can use command to move axis to exactly position:

On controller, press F1/DRVC and it will display Drive, command (axis, command, parameter). For example, if you want to drive RSW to 90, press 8,2,90, then ENTER. The RSW will rotate into beam path. Press Shift+F1/DRVC to leave command mode.

To exit Manual control in SAXS, press Esc.

Use Collect\Scan\Nanograph to image sample and pick the target(s) you would like to analyze:

Check your sample’s approximately X, Y range and input it into the window,

X begin:

Y begin:

Second/frame:

Background:

X:

Y:

7: Taking Data:

After imagine sample with Nanograph, click on the spot(s) of interest, then

In SAXS window, Collect\Scan\, uncheck "Accept Targets"

In Nanograph window, Send target? -> OK

In SAXS window, Collect\Scan\Edit target:

Change the time and move RSW out of beam (RSW 90->0)

Change the run # to same number->1

Then go to Collect\Scan\MultiTargets:

Job Name

Data for each target will be saved in frame. Use Ctrl+> to check each frame collected.

8: Data Process

Analyse\Integrate\Chi. Click OK. Use 1,2,3,4 to change 2θ and Chi range for integration.

Then hit Enter.

Save integrated data in the following two Formats for your further analyses:

Diffraplus: can be opened and processed by EVA.

Plotso: It's Text file and can be opened with notepad.

9: Take your sample out and close SAXS program.

Please refer to step 5 to get your sample out. Leave chamber under vacuum.

To quit SAXS program, go to Project\Exit. Leave generator setting at 10KV, 5mA.

10: If no user booked the NanoStar on the same day after you, turn the X-Ray off by turning the HV switch CCW on the machine and then leave the switch at vertical position.

11: Sign out Log sheet.