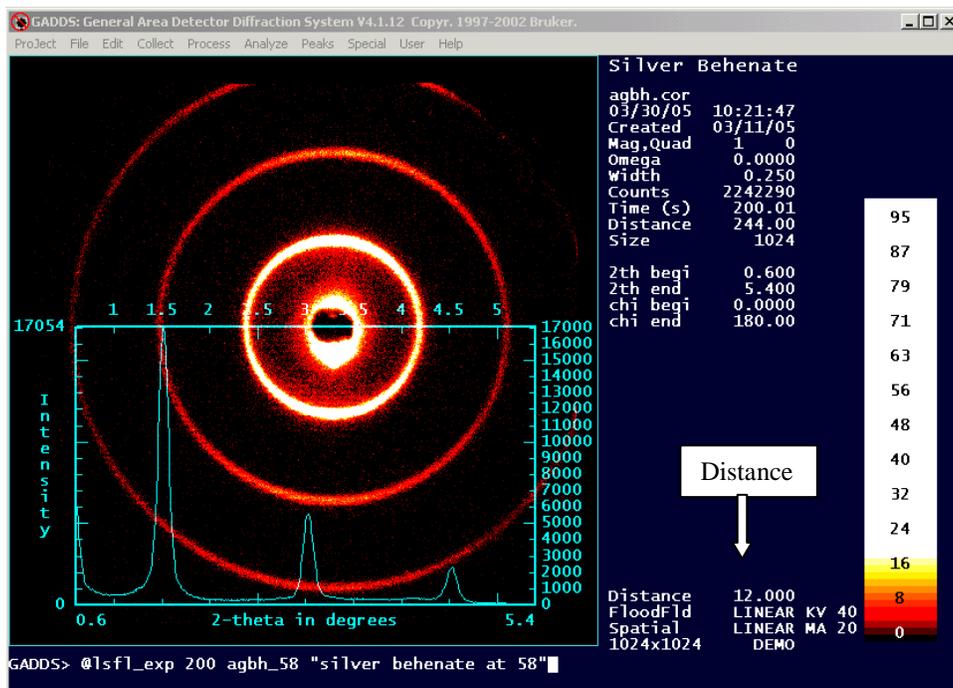


University of Minnesota Characterization Facility X-ray Scattering Facility SAXS—2 meter line

1. Turn on the helium using only the knob at the top if the helium tank. There should be a flow of about 1.0 on the small flow meter near the tube tower.
2. Slowly turn the instrument bias to 45.
3. Log onto the computer: Username: *code number*; Password: *Hrnc\$567* then you will be prompted to change it; Domain: Charfac.
4. Open the detector software: Start, SAXS programs, SAXS nodiff. Compare the distance in the instrument's logbook with the distance in the bottom, right corner of the page.
5. Using the drop-down menu at the top of the software window, choose: Special, Commandmode.



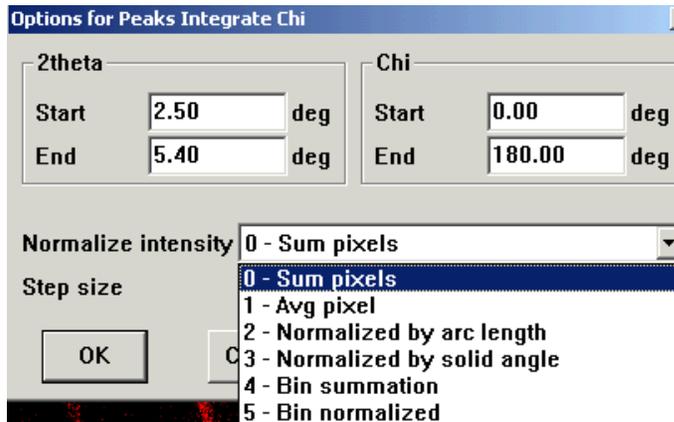
You will get a "SAXS>" prompt at the bottom of the page.

6. Run a quick scan with a sample that scatters well (preferably isotropic) to check the instrument and its calibration.
 - a. @expose 20
 - b. @correct
 - c. cal (the circles of the bullseye calibration should be aligned with the sample)
7. Check the integration settings—type “int”
 - a. Enter the correct step size for the distance. Step size is a function of the sample-to-detector distance. The larger the sample to detector distance, the smaller the step size.

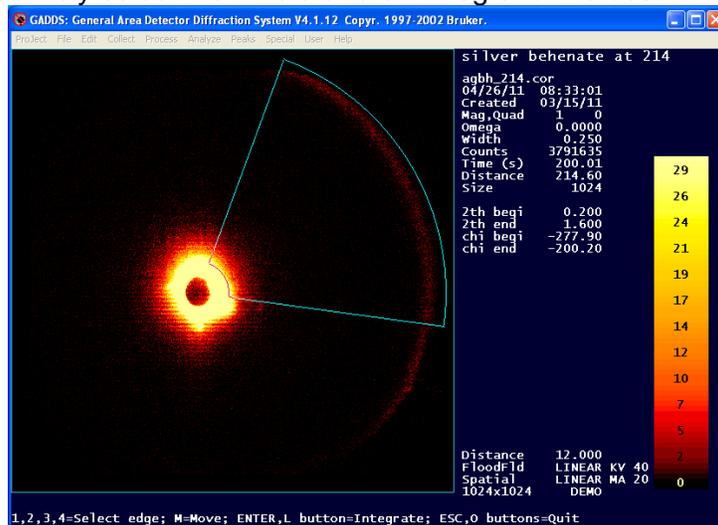
Common step sizes are:

Sample to Detector Dist.	Step Size
58 cm	0.01
104 cm	0.006
150 cm	0.004
200-240 cm	0.002

- b. Enter the correct “normalize intensity” value. The normalization techniques are explained in detail in the “help” menu. Ordinarily, for SAXS “0 – sum pixels” is used—that will magnify the intensity of the higher order peaks.

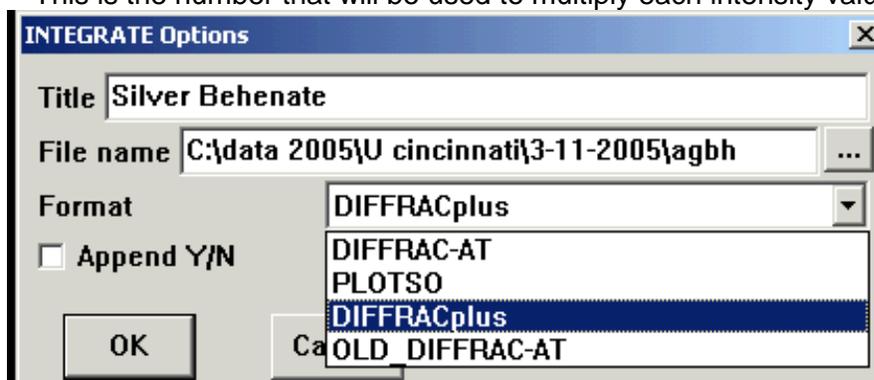


- c. After entering the step size and normalization factors, hit “enter” to interactively select the 2theta and chi integration values.



- d. Make sure the correct format is chosen. PLOTSO will save as an ascii format, DIFFRAC-AT will save in a format that is easily read into the WAXS software program JADE. Make sure the scale factor is also what you want.

This is the number that will be used to multiply each intensity value.



8. Edit your macro with the correct integration values and folder information (create a folder if you'd like) and save as an *.slm file.
9. Begin your sample analyses by using your macro:
 - a. Insert sample, check position with flashlight and mirror.
 - b. Pull vacuum, pressing on door until the vacuum gauge starts moving counter-clockwise.
 - c. Open the yellow mechanical safety switch in front of the x-ray tower.
 - d. Run @expose 20 to check scattering.
 - e. Run data collection macro or 1 second to check that it works properly.
 - f. Begin data collection.

When Done

1. Transfer your files via the charfac server, netfiles, or e-mail.
2. Delete your data files from the hard drive.
3. Turn off the helium.
4. Turn the bias down to zero.
5. Sign out on the computer.

Quick Start

1. Turn on Helium
2. Turn up Bias
3. Load "SAXS No Diff" software and compare distance with value in logbook.
4. Load sample (or standard) and check scatter:
 - a. @expose 20
 - b. @correct
 - c. Cal -- make sure center looks good
5. Edit data collection macro if necessary; begin data collection after checking macro with a 1 second scan.

Macros and Common Commands

Most of the work will be done using macros. Macros must begin with the “@” symbol. The most common macro is: **@expose xx** where xx is the amount of time you’d like to collect data. @expose will open the shutter, collect for XX seconds, close the shutter. You may also open or close the shutter by typing the following: SAXS>system closes shutter or SAXS>system opens shutter. Other commonly used macros and commands are:

- **@correct** (this will unwarp the data)
- **@stemp xx** (this will change the temperature to xx degrees.) The temperature range is -15 to 200C.
- **int** will take you to the integration menu
- **peaks** will plot the integrated peaks
- **cal** will take you to the calibration menu
- **config** will take you to the configuration menu
- **save** will take you to the save menu
- **load** will take you to the file loading menu
- **menu** will return you to the general software, pop-up menu

Macros can be combined to execute a series of commands. For example, you can use a macro to open the shutter, collect the data, save the data, unwarp the data, save the corrected pattern, integrate the data, print the data (either to a printer or to a *.bmp file) and then change the temperature, hold for a few minutes or several hours, and repeat the process. All macros are all saved in the c:\saxs\ directory and have an “slm” extension. You may use other people’s macros as a starting point for creating your own, but make sure you rename them. The following macro is used for general data collection:

```
@expose %1
save e:\sauer\%2' /title=%3'
@correct
save e:\sauer\%2'.cor /title=%3'
integrate /chi 0.5 5 -160 -10 /stepsize=0.01
integrate /write %3' /filename=e:\sauer\%2'.raw
```

The “%#’s” are wildcards and you need to enter the values after the macro’s name. For example, in the above macro, called lsfl_exp, the %1 corresponds to the amount of time the shutter will be open, %2 is the filename (no punctuation marks), and %3 is the title (if you put it in quotation marks, you can type anything you want.) To use the macro to collect a scan for 5 minutes, name it test with a title of “This is a test”, you would type:

```
SAXS>@lsfl_exp 300 test “this is a test”.
```

Editing of macros is done in notepad and all files should be saved as *.slm files (make sure the bottom box says “all files” instead of “txt files”).

For Kapton

Contact Dupont and ask for samples of Kapton 100HN Polyimide Film:

Dupont , K/T Customer Service, P.O. Box 89, Circleville, OH 43113
Product Information: 1-800-237-4357
Ordering Information: 1-800-967-5607